



*An Analysis of the Effect of Ambient Temperature Aging on Al-Li Airframes*

CUSACK, Aaron Barrie

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# An Analysis of the Effect of Ambient Temperature Aging on Al-Li Airframes

by

Aaron Barrie Cusack

*Submitted in accordance with the requirements for the degree of  
Doctor of Philosophy*

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and

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## **Abstract.**

Aluminium Lithium (Al-Li) alloys exhibit behaviours in mechanical properties which separate them from conventional precipitation hardening aluminium (Al) alloys. One such behaviour is an increase in strength and a reduction in ductility which occurs as a result of exposure to relatively low temperatures (between 70°C to 100°C). This work sets out to establish a representative exposure temperature for internal helicopter main lift frame structure that is subjected to 'hot' environments. This representative temperature, determined to be 53.1°C, was investigated to understand whether exposure at this temperature for times ranging from 0 to 1000 hours had any effect on the mechanical properties of Al-Li 8090 T852 main lift frame forgings.

The material for this project was obtained from in-service military aircraft that had been operating for a minimum of a decade in a number of theatres. This material provided a unique opportunity to compare and contrast the elemental chemical, microstructural and mechanical properties of the in-service material against the original design specification and against material that had been exposed to 53.1°C for between 0 to 1000 hours. A comprehensive mechanical testing regime has shown that, after 1000 hours exposure at 53.1°C, the main lift frame material exhibited a 36% reduction in ductility accompanied by a 7.8% in UTS and a 13.2% increase in 0.2% proof stress. Furthermore, a repeatable pattern of an increase and reduction in strength of the material over the first 72 hours of exposure to 53.1°C has been established and investigated.

A detailed study of the main strengthening precipitate  $\delta'$  ( $\text{Al}_3\text{Li}$ ), using transmission electron microscopy, differential scanning calorimetry and comparison with the current literature was carried out. This has shown that the initial strengthening mechanism (from 0 to 24 hours) is likely to be the result of an increase in volume fraction of Guinier-Preston zones or potentially  $\delta'$  precursors, which form from available solute in the material. This is likely to have occurred due to decomposition of the solid solution, which in turn is a result of low temperature exposure during operational service. However, after 36 hours, a significant decrease in strength indicates that this formation is unstable. A second strengthening mechanism, (48 hours onwards) is likely to be the formation of very finely dispersed  $\delta'$  precipitates (2-4 nm in diameter) and is well documented as being the reason for the increase in strength and loss of ductility in Al-Li alloys that are exposed to these modest in-service temperatures.

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*And to my long-suffering wife Amelia, it was a long journey, thank you for carrying me along the way. I'll get to that long list of DIY now.*

*I dedicate this thesis to my son James (and your future sibling).*

*Look at what can be achieved, if you are willing to put your mind to it – Dad.*

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## **1 Introduction.**

### **1.1 Aluminium-lithium alloys: an overview.**

Lithium (Li), first produced in a metallic form in 1818 (Grady, 1981)<sup>1</sup>, is the lightest metal and the least dense solid element within the periodic table. Li is soluble in aluminium (Al) up to ~ 4 weight percent (wt %) or 16 atomic percent (at %), any further increase in the amount of Li will result in the inability to retain total solubility in the solid state (Williams, 1981)<sup>2</sup>. Conventional aluminium alloys have long been established as a material used in aerospace applications because of their high strength to weight ratio. Research into elements that will form an alloy with Al has yielded a wide range of materials. The American aluminium-copper (Al-Cu) 2xxx series provides alloys with reasonable strength and superior ductility, the aluminium-zinc (Al-Zn) 7xxx series can provide alloys with strength comparable to steels and the aluminium-magnesium-silicon (Al-Mg-Si) 6xxx series alloys provides superior welding properties compared with the other aluminium alloy series.

When alloyed with Al, Li is one of only two elements (the other being beryllium) which alters the density and the modulus of an alloy (Edgar, Starke & Quist, 1988)<sup>3</sup>. The high solubility of Li in Al yields material that for every 1 weight percent (wt%) addition of Li, the density is reduced by 3% and the Young's modulus increases by approximately 5% whilst retaining mechanical properties that are comparable with similar conventional heat treated Al alloys. The effect of alloying elements on the Young's modulus of Al alloys can be

seen in Figure 1. The key material property attribute in Al-Li alloys, used in transport, is the high strength to weight ratio associated with the reduction in density. Combined with a number of additional factors (including the cost of manufacturing carbon fibre reinforced composites and the well-established worldwide aluminium production industry during the 1980's), Al-Li alloys were and still are seen as a cost-effective material for use in aviation (*aluminium insider editorial*, 2014)<sup>4</sup>.

The potential benefits for the use of Al-Li alloys in an industry which is always demanding stronger and lighter materials, is evident. Furthermore, using Li as an alloying element provides the capability to produce the metastable precipitate delta prime ( $\delta'$ ) (Polmear, *Light Alloys*)<sup>5</sup> which is considered to be the main strengthening precipitate in Al-Li alloys (Whitman, 1990)<sup>6</sup>.

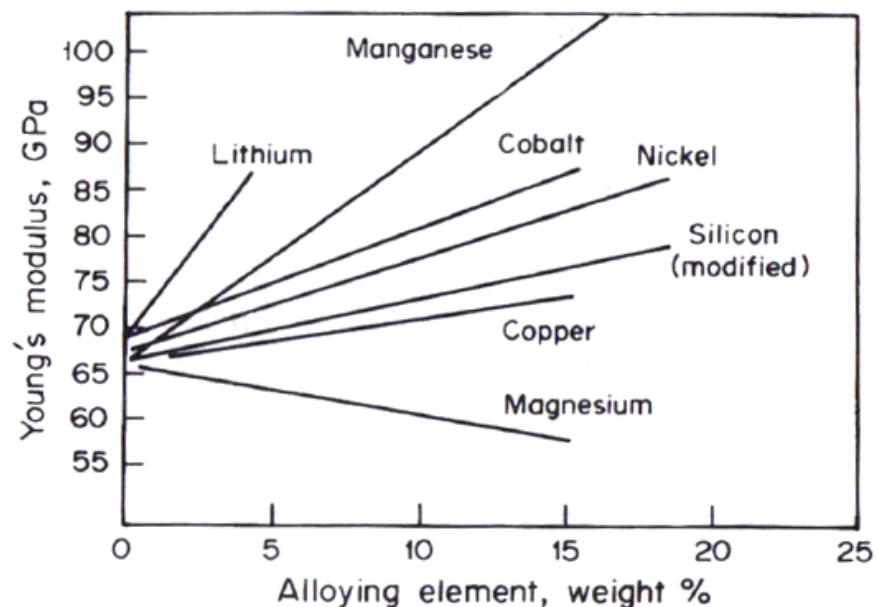


**Figure 1:** Showing the effect of alloying elements on the Young's modulus of Al alloys (Polmear, *Light Alloys*, 1995)<sup>7</sup>.

## 1.2 Development of the Al-Li alloy 8090.

Commercial interest in Al-Li alloys has historically “waxed and waned”. However, a substantial amount of research was conducted during the 1980’s with the first three Al-Li specific international conferences held between 1980 and 1985. During this period in the United Kingdom, research into Al-Li alloys was conducted by Alcan International and the Defence Research Agency (DRA)<sup>i</sup> Farnborough. In 1985 DRA published a paper detailing the status of Al-Li alloy development in the UK (Peel, Evans & McDarmaid, 1985)<sup>8</sup>. The main focus of the research in the UK centred around three alloy designations: Lital (Al-Li-Cu-Mg-Zr) ‘A’ ‘B’ and ‘C’ being used to ‘substitute’ conventional Al alloys’.

- Lital A (medium strength) was intended as a substitute for the 2014-T6 alloy in sheet, extrusion, plate and forgings.
- Lital B (high strength) was intended as a substitute for the 7075-T6 alloy in sheet, extrusion, plate and forgings.
- Lital C (damage tolerant) was intended as a substitute for the 2024-T3 sheet, extrusion and plate.

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<sup>i</sup> DRA was an amalgamation of five Defence research establishments; *Admiralty Research Establishment (ARE)*, *Royal Aircraft Establishment (RAE)*, *Aeroplane and Armament Experimental Establishment (A&AEE)* *Royal Armament Research and Development Establishment (RARDE)* and the *Royal Signals and Radar Establishment (RSRE)*. DRA existed between 1991 and 1995 until it became the *Defence Evaluation and Research Agency (DERA)*.

The objective was to manufacture existing aircraft components with one of the three Lital alloys. In the context of the Peel, Evans & McDarmaid (1985)<sup>8</sup>, the material 'substitution' consisted of two different philosophies: 'literal substitution' - direct replacement with little to no redesign of parts and 'substitute by type' - a partial or complete redesign of components combined with a suitable Al-Li alloy that would allow for superior weight savings due to exploiting the density reduction and stiffness increase.

Reference is made to the fact that Lital in the 'A' and 'C' conditions had both been registered in the designation of '8090'. However, further investigation revealed that Lital 'A' became 8090 and Lital 'C' became 8091 (Gregson et al., 1987)<sup>9</sup>. Although it was not known at the time, Al-Li 8090 would become one of the primary alloys used in the construction of a multi-purpose aircraft that would see frontline service with UK armed forces.

A history of common Al-Li alloys has recently been published by Riojia and Liu (2012)<sup>10</sup>. The early, Aloca and Soviet Al-Li alloys (1970's) are classed as the '1st generation'. Alloys produced in the 1980's including Al-Li 8090 are found within the 2nd generation and all further production after the 1990's are designated under the 3rd generation. This is shown in Table 1.

Furthermore, evidence of current continued interest in Al-Li alloys can be seen in Table 1, with alloys 2060 and 2055 being recently added and 2050 (in the T84 condition) being used on the Airbus A380-00011.

**Table 1: Showing a reproduction of Rioja and Liu's table of the three generations of Al-Li alloys (2012)<sup>10</sup>.**

|                                  | Li   | Cu   | Mg       | Ag   | Zr   | Sc  | Mn       | Zr       | Approximate Date     |
|----------------------------------|------|------|----------|------|------|-----|----------|----------|----------------------|
| 1st generation                   |      |      |          |      |      |     |          |          |                      |
| 2020                             | 1.2  | 4.5  |          |      |      |     | 0.5      |          | Alcoa 1958           |
| 01420                            | 2.1  |      | 5.2      |      | 0.1  |     |          |          | Soviet 1965          |
| 01421                            | 2.1  |      | 5.2      |      | 0.1  | 0.2 |          |          | Soviet 1965          |
| 2nd generation (Li $\geq$ 2 pct) |      |      |          |      |      |     |          |          |                      |
| 2090                             | 2.1  | 2.7  |          |      | 0.11 |     |          |          | Alcoa 1984           |
| 2091                             | 2.0  | 2.0  | 1.3      |      | 0.11 |     |          |          | Pechiney 1985        |
| 8090                             | 2.4  | 1.2  | 0.8      |      | 0.11 | 0.2 |          |          | EAA 1984             |
| 01430                            | 1.7  | 1.6  | 2.7      |      | 0.11 |     |          |          | Soviet 1980s         |
| 01440                            | 2.4  | 1.5  | 0.8      |      | 0.11 |     |          |          | Soviet 1980s         |
| 01450                            | 2.1  | 2.9  |          |      | 0.11 |     |          |          | Soviet 1980s         |
| 01460                            | 2.25 | 2.9  |          |      | 0.11 | 0.1 |          |          | Soviet 1980s         |
| 3rd generation (Li < 2pct)       |      |      |          |      |      |     |          |          |                      |
| 2195                             | 1.0  | 4.0  | 0.4      | 0.4  | 0.11 |     |          |          | LM/Reynolds 1992     |
| 2196                             | 1.8  | 2.9  | 0.5      | 0.4  | 0.11 |     | 0.35 max | 0.35 max | LM/Reynolds 2000     |
| 2297                             | 1.4  | 2.8  | 0.25 max |      | 0.11 |     | 0.3      | 0.5 max  | LM/Reynolds 1997     |
| 2397                             | 1.4  | 2.8  | 0.25 max |      | 0.11 |     | 0.3      | 0.10     | Alcoa 1993           |
| 2198                             | 1.0  | 3.2  | 0.5      | 0.4  | 0.11 |     | 0.5 max  | 0.35 max | Reynolds/McCook 2005 |
| 2099                             | 1.8  | 2.7  | 0.3      |      | 0.09 |     | 0.3      | 0.7      | Alcoa 2003           |
| 2199                             | 1.6  | 2.6  | 0.2      |      | 0.09 |     | 0.3      | 0.6      | Alcoa 2005           |
| 2050                             | 1.0  | 3.6  | 0.4      | 0.4  | 0.11 |     | 0.35     | 0.25 max | Pechiney 2004        |
| 2060                             | 0.8  | 3.95 | 0.85     | 0.25 | 0.11 |     | 0.3      | 0.4      | Alcoa 2011           |
| 2055                             | 1.15 | 3.7  | 0.4      | 0.4  | 0.11 |     | 0.3      | 0.5      | Alcoa 2012           |

Al-Li alloys had been heralded as the 'future of light metals' and a 'natural progression' of the use of Al alloys in aerospace engineering applications. The justifications for this varied, but range from: a perceived 'threat' from composite materials superseding metallic materials (where in reality aircraft are a blend of composite and metallic materials), to the understanding that reducing the density of a material (whilst retaining mechanical properties) is one of the most effective strategies when designing aerospace components.

### 1.3 History of Al-Li alloys.

The first Al-Li alloy (Scleron) was developed in Germany in the 1920s and the first (limited) application of an Al-Li alloy was in the use of the alloy 2020 in the

wing tips of the Vigilant aircraft in the 1950's. A well-publicised application of Al-Li is its use in the super lightweight external tank designed by NASA, to carry the fuel required to propel space shuttles beyond the earth's atmosphere (NASA, 2005)<sup>12</sup>. The reduction in weight provided by Al-Li alloys enables a heavier payload to be carried. Whilst this application has been very successful, the external tank is not a reusable component and is discarded once the operation is complete.

#### **1.4 Development of the EH101 helicopter.**

The AW101 helicopter formerly known as the EH101 (Figure 2), is the result of a joint venture between the Italian company Agusta and British company Westland Helicopters (WH) now collectively known as Leonardo Helicopters (LH)<sup>ii</sup>. In 1988, WH presented a paper on the current development of the aircraft and supporting data in the use of Al-Li alloys (Smith, 1988)<sup>13</sup>.

Smith (1988)<sup>13</sup> states that the EH101 programme began in 1984 and resulted in 9 prototype (PP) aircraft. PP1 (developed by LH) first flew on 9<sup>th</sup> October 1987. The two aircraft variants were defined as 'maritime' and 'utility'. The roles of the 'maritime' aircraft were defined as follows:

- Anti-Submarine Warfare (ASW),
- Anti-Ship surveillance and tracking,

---

<sup>ii</sup> Note that for the purposes of this project, AW101 and EW101 are synonymous. However, EH101 is the more widely known designation (outside of the UK MoD).



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- Anti-Surface Vessel,
- Amphibious Operations,
- Search and Rescue,
- Airborne Early Warning,
- Vertical Replenishment,
- Electronic Countermeasures.

The 'utility' variant was noted to have a rear loading ramp with typical roles stated to be:

- Offshore Oil Support,
- Civil Defence,
- Vehicle/troop transportation,
- Air ambulance,
- Exploration,
- Logging,
- Search and Rescue.

The UK Ministry of Defence (MoD) adopted the EH101 platform (designated Merlin), to replace the aging Sea King fleet, which primarily undertook maritime operations such as Anti-Submarine Warfare (ASW). The Royal Navy (RN) received the 'maritime' variant which was designated 'Merlin Mk1'. The Royal Air Force (RAF) received the 'utility' variant which was designated the 'Merlin Mk3'. The Merlin aircraft first flew in 1999 and entered into service in 2000.



**Figure 2: Showing an example of a Royal Navy Merlin helicopter.**

### **1.5 Helicopter main lift frames.**

Main lift frames provide the foundation on which the structural integrity of rotary-wing aircraft are built. The main lift frame structure supports; the cockpit, main rotor head, tail structure, the tail rotor, gearboxes and other ancillary items relating to the aircraft role and specific mission. Depending on whether the aircraft is stationary or in flight, the forces on the main lift frame can vary between compression, tension and torsion. Often these forces occur at multiple locations at the same time.

Due to their construction, helicopter main lift frames are not easily removed for repair without rendering the aircraft unserviceable. Therefore, in most instances it is uneconomical to remove main lift frames unless absolutely necessary. In the case of the EH101, the torsion box structure which houses the main undercarriage also provides two sections of main lift frames on the

port and starboard sides of the aircraft. These frames are incorporated in the 'hockey stick' shaped forgings (blue and yellow sections, Figure 3) located at stations 9875 and 10375<sup>iii</sup>, Figure 3. Whilst still not easy to remove, these sections are technically replaceable if the torsion box becomes damaged. If required the entire 'hockey stick' is removed under a specialist repair procedure. It is these sections which provided the unique material for this current research project.

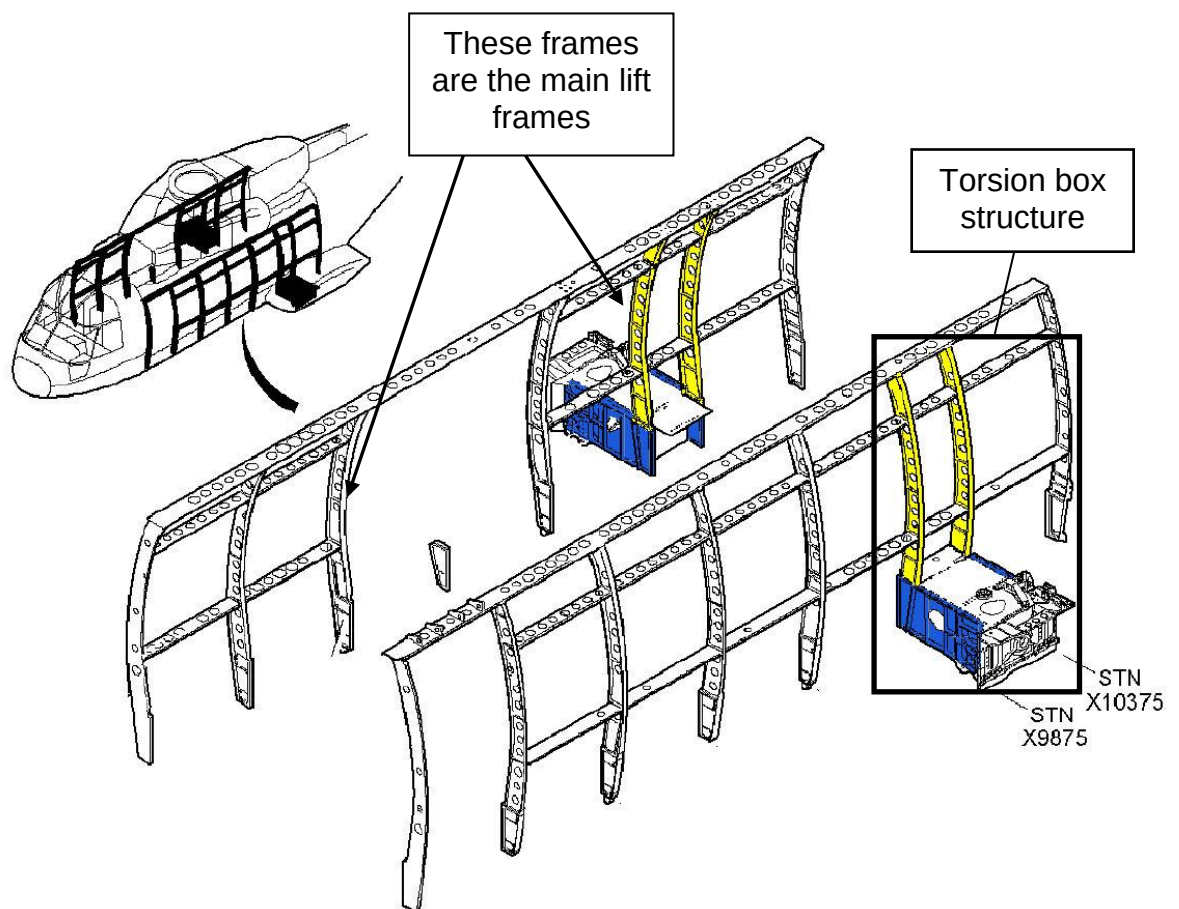


**Figure 3: Showing the location of the main lift frames and the torsion box structures in the EH101 helicopter.**

<sup>iii</sup> Stations are numbered by the distance (in mm) from a 0,0 point on the x and y axes at the front of the aircraft.

### **1.6 Justification for use of Al-Li 8090 in the EH101 development programme.**

It is apparent that there were two philosophies adopted in the selection of materials for the EH101:

- Firstly, the use of stronger and stiffer materials to reduce the cross-sectional thickness of the main lift frame structure,
- Secondly, the use of lighter materials which provided equivalent strength and toughness as conventionally denser material.

Smith (1988)<sup>13</sup> stated that the first philosophy had already been fully exploited in the development of the EH101. In the pursuit of lighter materials, Al-Li was not the only consideration; composite materials were also investigated. However, composite materials were rejected as not having been sufficiently researched and developed at the time. In addition, there were two further considerations:

1. Al-Li alloys were cheaper to produce than composites (but were still 3-5 times more expensive than conventional Al).
2. The aviation industry had already been using conventional aluminium. Therefore, processes and operations were in place that could accommodate the production of Al-Li components with minimal adjustment in the manufacturing environment. The manufacturers

were more comfortable with an Al alloy that they understood rather than the relatively unfamiliar concept of composite materials.

The required weight saving for the EH101 main lift frame was stated to be 55 kg (Smith, 1988)<sup>13</sup> and (Abdel-Latif et al., 1988)<sup>14</sup>. The WH report noted that complete substitution of all of the conventional Al with Al-Li would result in a saving of 82 kg. The decision was made for the main lift frames of the aircraft to be forged from Al-Li.

Smith (1988)<sup>13</sup> makes reference to the statement that the 0.2% proof stress of a material should not be less than 75% of the ultimate tensile strength (UTS) of the same material (75% for military and 67% for civilian applications) (Peel et al., 1985)<sup>8</sup>. The materials used in the testing regime for the main lift frame forgings were Al-Li 8090, Al-Li 8091 and conventional Al-Zn alloy 7010. The tensile test results of the mechanical testing regime undertaken by Smith are detailed further in Section 2.4 of the Literature Review. However, the relevant point for this introduction is that the mechanical properties of the conventional aluminium alloy 7010 were found to consistently meet the minimum mechanical property requirements, whereas the two Al-Li alloys did not always meet the criteria. The results are quoted as: “...they nevertheless generally achieve the minimum allowable EH101 main lift frame requirements (S-values) as determined by WHL (Westland Helicopter Laboratory) stress office for longitudinal, long transverse and short transverse directions” (Smith, 1988)<sup>13</sup>.

A report by the Quality Engineering Test Establishment (QETE - Abdel-Latif et al., 1988)<sup>14</sup> states that the minimum requirements for the 0.2% proof stress and elongation could not always be achieved by Al-Li 8090. The report also mentioned that it was anticipated that an increase in the level of magnesium would improve the 0.2% proof stress and elongation properties by ~10%.

However, the QETE report notes that at that time, no experimentation had been undertaken to establish that this ~10% increase in elongation was achievable. Smith references Gregson, Peel and Evans, (1986)<sup>15</sup> who identified that increases in Mg (and/or Cu) resulted in limited increases in UTS and 0.2% proof stress. Work undertaken by Dinsdale, Harris and Noble, (1981)<sup>16</sup> noted an increase in mechanical properties resulting from additions of Mg, but also noted that the presence of Zr as a grain refiner severely limited the ductility of the alloys.

Microstructural examination of the two Al-Li alloys revealed that the 8091 material exhibited a recrystallised structure with varying grain sizes. Scanning Electron Microscopy (SEM) examination of tensile fracture surfaces revealed a reduction of features consistent with ductility (i.e. the material became more brittle) in comparison with Al-Li 8090. Therefore, Al-Li 8091 was dropped from the programme and all of the Al-Li material on the EH101 was manufactured from Al-Li 8090.

### 1.7 EH101 main lift frame manufacturing process.

The main lift frame and torsion box 'hockey sticks' are a single piece forging.

The manufacturing process is detailed below:

- 1) Hand forging from a billet, at a temperature between 470-490°C to form the 'hockey stick' or 'L' shape.
- 2) Solution heat treatment (T4) between 525-535°C for two hours with a subsequent water quench.
- 3) Cold working by way of cold compression of 2-5%.
- 4) Artificial aging at a temperature between 170-180°C for 20-50 hours, resulting in a T852 temper state.

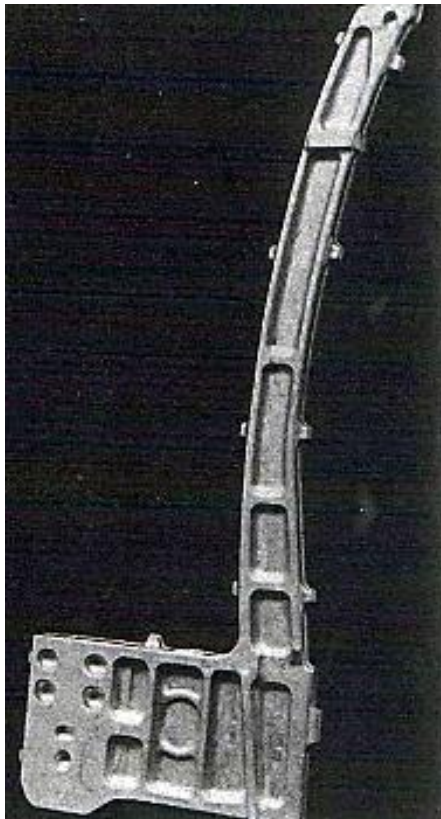


**Figure 4:** Showing a forged 'hockey stick' section, prior to final machining (Kaufman *Introduction to Al Alloys and Tempers*)<sup>17</sup>.

### **1.8 Initial project aim(s), objectives and subsequent development.**

The initial aim of the project was an attempt to characterise the damage inflicted by ballistic impacts upon Al-Li 8090 T852 main lift frames, with a view to understand how damage at the area of impact propagates to final failure under static loading. The justification for this work was that by understanding the failure mode at the damaged regions and subsequent propagation cracking, a greater level of knowledge could be called upon in the consideration of the repair process.

To achieve this aim, the following objectives were set out:

1. Characterise the as-received main lift frame material and determine whether the material was comparable with existing literature.
2. Undertake a feasibility study of ballistic testing to enable the examination of the fracture surface, the impact area and of the surrounding microstructure.
3. Carry out an in-depth examination of the damaged material using Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM).



4. Replication of the ballistic damage on test coupons and tensile testing of samples to investigate the propagation to failure of damaged material.
5. Tensile testing of repair techniques on repaired specimens to determine which technique restored the greatest amount of static strength to the main lift frame.

Following the first ballistic testing regime for this investigation, microstructural analysis of main lift frame material revealed a localised grain structure that was denuded of precipitates. This feature was deemed to be directly attributable to the damage on the Al-Li main lift frame. It became evident that before any repair techniques could be investigated, greater clarification of these features was required. This resulted in the proposal of a more rigorous investigation method. It was decided (with agreement between all invested parties) to alter objective 4 & 5 to the following:

- 4.1 Detailed examination of the identified microstructural features in the damaged main lift frame material, primarily using Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) with the potential use of Focussed Ion Beam thinning.
- 5.1. Undertake a feasibility study to determine whether a suitable model that replicates the mechanical and microstructural

behaviour of Al-Li during ballistic damage either exists, or can be created.

- 6.1 Development and testing of said model to determine whether the characteristics of Al-Li can be suitably replicated in the instance of a ballistic impact on a simulated section of an Al-Li main lift frame and ultimately comparison of the simulated results to the physical evidence.

Two further ballistic trials incorporating video capture and thermography were undertaken, with a commensurate amount of metallurgical examination of the impacted regions. A summary of the work and findings is written in another document<sup>iv</sup>. In short however, the microstructural feature of interest was limited to a single lift frame and the feature (whilst the evidence suggests the causality is directly linked to the ballistic damage) was not found to be present on another lift frame. Therefore, this line of investigation was deemed to not be repeatable, nor suitable for further work.

A third approach originating from a number of considered factors by the author attempted to determine whether certain atmospheric conditions (primarily temperature) had an effect on the mechanical properties of the main lift frames. The justification for this was the operation of Merlin aircraft in Afghanistan. A number of 1710 NAS military personnel (in discussion with the

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<sup>iv</sup> *The report is not relevant to this work.*

author) had suggested that aircraft were exposed to temperatures that were not equal to the recorded air temperature (as a result of the combination of a number of factors, including concrete pads and shelters and more<sup>v</sup>).

In addition, the author surmised that it was likely that the external skin of the aircraft would be subjected to a different temperature than the internal structure – and recent work by the Defence Science and Technology Laboratory (DSTL) had the potential to provide empirical data to support work in this area.

Therefore, a final set of objectives with a single aim was agreed as follows:

Aim: To determine whether the effect of exposure to “hot” operating environments on the mechanical properties and microstructure of the main lift frames is comparable with known “low” temperature exposure work on the mechanical properties and microstructure of Al-Li 8090.

To achieve this, the following objectives were set out:

1. Characterise the as-received main lift frame material and determine whether the material was comparable with existing literature.

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<sup>v</sup> *Some of which cannot be discussed in the current work.*

2. Using available data, determine a maximum (realistic) exposure temperature for in-service Al-Li 8090 lift frames and compare that temperature with temperatures employed in the literature.
3. Undertake a feasibility study to determine whether the exposure temperature has any effect on the mechanical properties of the Al-Li 8090 main lift frames.
4. Show that any effect is repeatable (through a programme of heat treatment and subsequent mechanical testing).
5. Investigate any effect of heat treatment on the microstructure using advanced analytical techniques, such as Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and Differential Scanning Calorimetry (DSC).

Prior to undertaking any work, a literature review of Al-Li alloys and in particular Al-Li 8090 was undertaken and is detailed next in Section 2.

## 2 Literature review.

### 2.1 Microstructure of Al-Li alloys.

The quinary alloying system of Al-Li 8090 is composed of Al, Li, Cu, Mg and Zr. Of note is the formation of the precipitates:  $\delta$  (AlLi),  $\delta'$  ( $\text{Al}_3\text{Li}$ ), S ( $\text{Al}_2\text{CuMg}$ ),  $T_1$  ( $\text{Al}_2\text{CuLi}$ ),  $T_2$  ( $\text{Al}_6\text{CuLi}_3$ ) and  $\beta'$  ( $\text{Al}_3\text{Zr}$ ), (Figure 5). The main strengthening precipitate is  $\delta'$  with secondary strengthening provided by the precipitation of S phase.

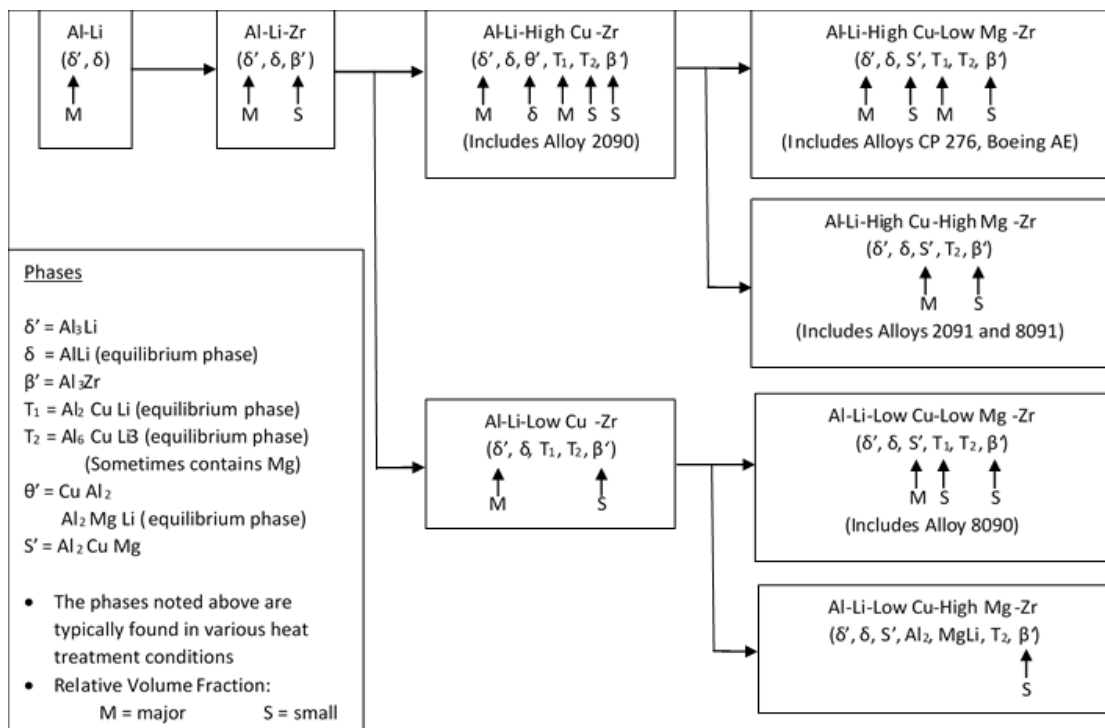


**Figure 5:** Showing an illustration of Narayanan and Quist's diagram of a selection of Al-Li alloys and their precipitates, including Al-Li 8090. Note  $\delta'$  is present in all of the systems (as shown by Edgar, Starke & Quist, 1988)<sup>3</sup>.

#### 2.1.1 Al-Li binary system – the $\text{Al}_3\text{Li}$ ( $\delta'$ ) and AlLi ( $\delta$ ) precipitates.

The Al-Li binary equilibrium diagram is often subject to review, the version shown in Figure 6 was published by Butts and Gale (2004)<sup>18</sup>. At the Al

dominant end of the equilibrium diagram: on slow cooling from 600°C results in the formation of an Al rich  $\alpha$  solid solution (Al) + the  $\delta$  precipitate (AlLi).

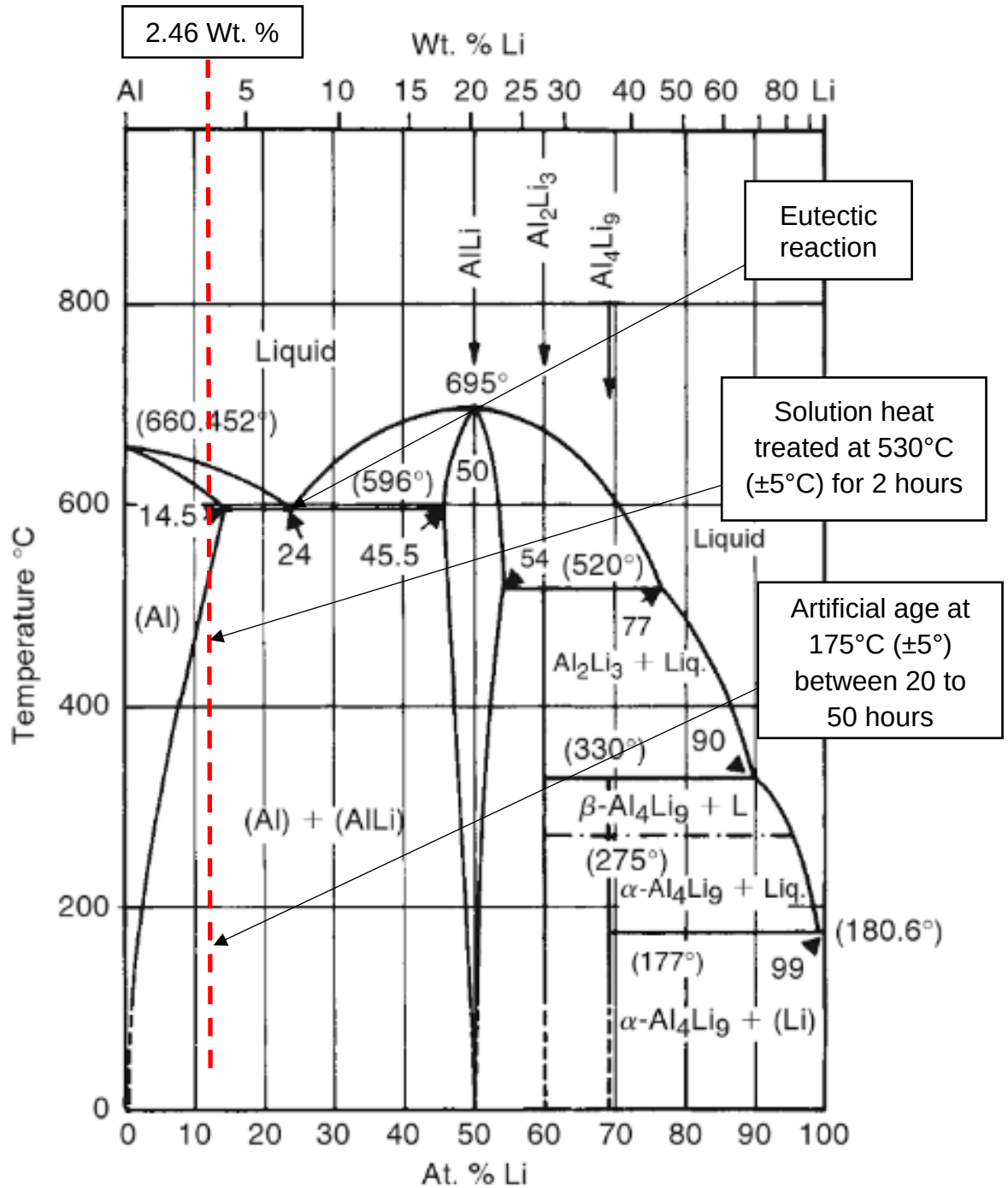


Figure 6: Al-Li binary precipitates diagram (Butts and Gale 2004)<sup>18</sup>.

However, prior to the formation of the equilibrium  $\delta$  precipitate if the same alloy is rapidly cooled and aged, there exists the metastable  $\delta'$  precipitate ( $\text{Al}_3\text{Li}$ ). First reported by Silcock (1960)<sup>19</sup>,  $\delta'$  has an ordered face centre cubic (FCC)  $\text{L1}_2$  lattice structure. The eight corner points of the structure are occupied by Li atoms and at the centre of the six faces are Al atoms (Sanders, 1980)<sup>20</sup>, Figure 7. Precursors to the formation of  $\delta'$ , either GP zones and/or a  $\delta''$  precipitate have been predicted by a number of researchers, but have not been directly observed (Prasad, Gokhale and Wanhill, 2014)<sup>21</sup>. Work undertaken to determine the  $\alpha / \delta'$  solvus boundary (from data gathered from a number of researchers) can be seen in Figure 8, (Nobel and Bray, 1998)<sup>26</sup>.

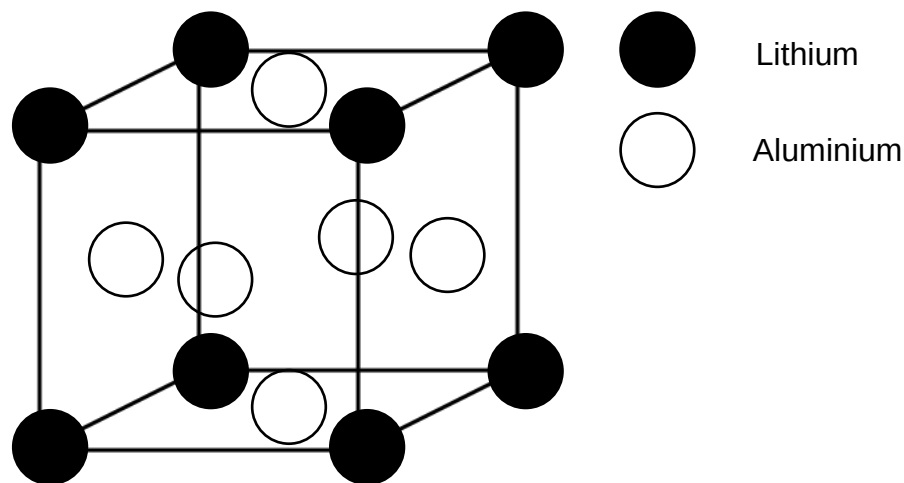


**Figure 7: Showing a representation of a  $\delta'$   $\text{L1}_2$  cubic structure.**

Notably, the  $\delta'$  and the Al matrix share similar structure (cube-cube) and lattice parameters (0.401 nm and 0.405 nm for  $\delta'$  and the matrix, respectively). This results in a relatively low misfit strain between the  $\delta'$  and the matrix (Williams and Edington, 1975)<sup>22</sup>. The  $\delta'$  precipitate forms as spheres that are

homogenously distributed in the matrix. It is the presence of the  $\delta'$  within the matrix that is accredited with impeding the motion of dislocations within the crystal structure and hence providing the main strengthening element to Al-Li alloys, (Whitman, 1990)<sup>23</sup>. However, typical  $\delta'$  precipitate diameters in Al-Li 8090 have been reported to be  $\geq 45$  nm, Edgar and Starke (1988)<sup>24</sup> and Nobel, Harris and Dinsdale (1996)<sup>28</sup>. This is below the stated critical diameter for Orowan/dislocation looping which is  $\sim 50$  nm, (Edgar, Starke and Quist, 1988)<sup>3</sup> and hence the majority of the  $\delta'$  precipitates in Al-Li alloys are susceptible to being sheared by dislocations, thereby promoting localised planar slip (Hornbogen and Gahr, 1975)<sup>25</sup>.

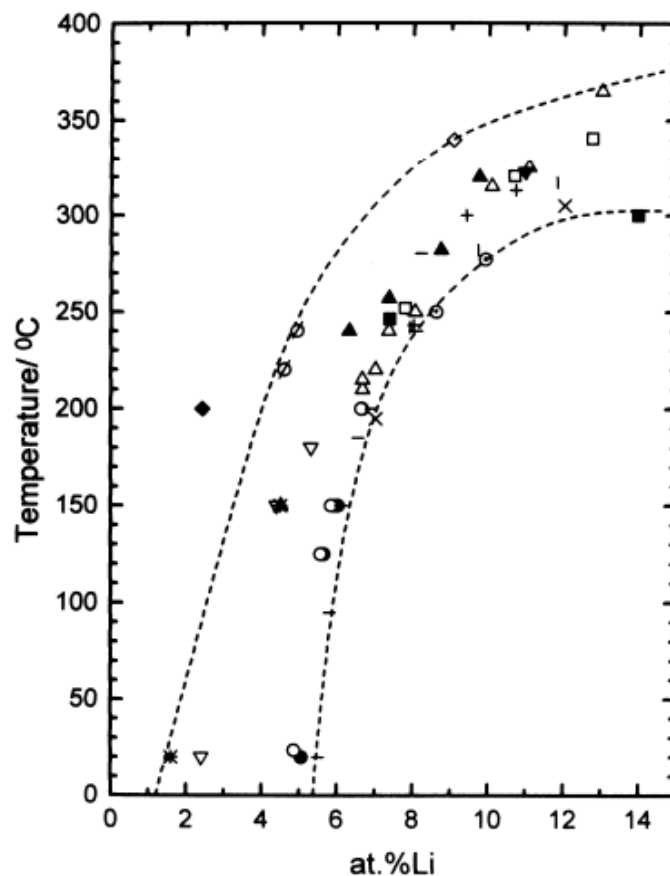


**Figure 8:** Showing collected  $\alpha/\delta'$  solvus data from a number of researchers (Nobel and Bray, 1998)<sup>26</sup>.



Artificial aging of the  $\delta'$  precipitates results in coarsening which will increase the diameter of the precipitates (potentially up to 300 nm) (Williams, 1981)<sup>2</sup>. During coarsening,  $\delta'$  precipitates retain their spherical shape and remain coherent with the matrix. However, Williams and Edington (1975)<sup>22</sup> noted that within 20°C of the solvus (for a 7.8 at% Li alloy with a 250°C solvus) preferential coarsening occurred at matrix dislocations. Although the diameter of  $\delta'$  is deemed to be relatively small, enabling shear and dislocation movement, it is the sheer volume fraction of  $\delta'$  which is attributed to the primary strengthening effect in Al-Li alloys (El-Aty, Xu Guo, [et al.], 2017)<sup>27</sup>.

The volume fraction of  $\delta'$  is subject to the amount of Li available in the solid solution.  $\delta'$  precipitates are noted to be larger and less numerous in alloys with lower amounts of Li available in the solid solution. A greater volume fraction of smaller precipitates form in alloys with a greater availability of Li (Noble, Harris and Dinsdale, 1996)<sup>28</sup>. One observation made by Nobel and Bray, (1998)<sup>26</sup> was that true diffusion controlled coarsening kinetics of  $\delta'$  did not apply at temperatures < 130°C (over a 2000 h period of observation).

The plate-shaped equilibrium precipitate  $\delta$  (Al-Li) is not present during the quench and its precipitation results from: slow equilibrium cooling from above the solvus; high aging temperatures or prolonged aging times at normal aging temperatures (Venables, Christodoulou and Pickens, 1983)<sup>29</sup>.  $\delta$  primarily nucleates heterogeneously on grain boundaries (Jensrud and Ryum, 1983)<sup>30</sup> but can also precipitate within the grains during the latter stages of aging. It

was reported by Angerman (1963)<sup>31</sup> that it is possible to precipitate  $\delta$  in both the grains and grain boundaries in Al-Li foils by using the heat from the electron beam of a TEM.

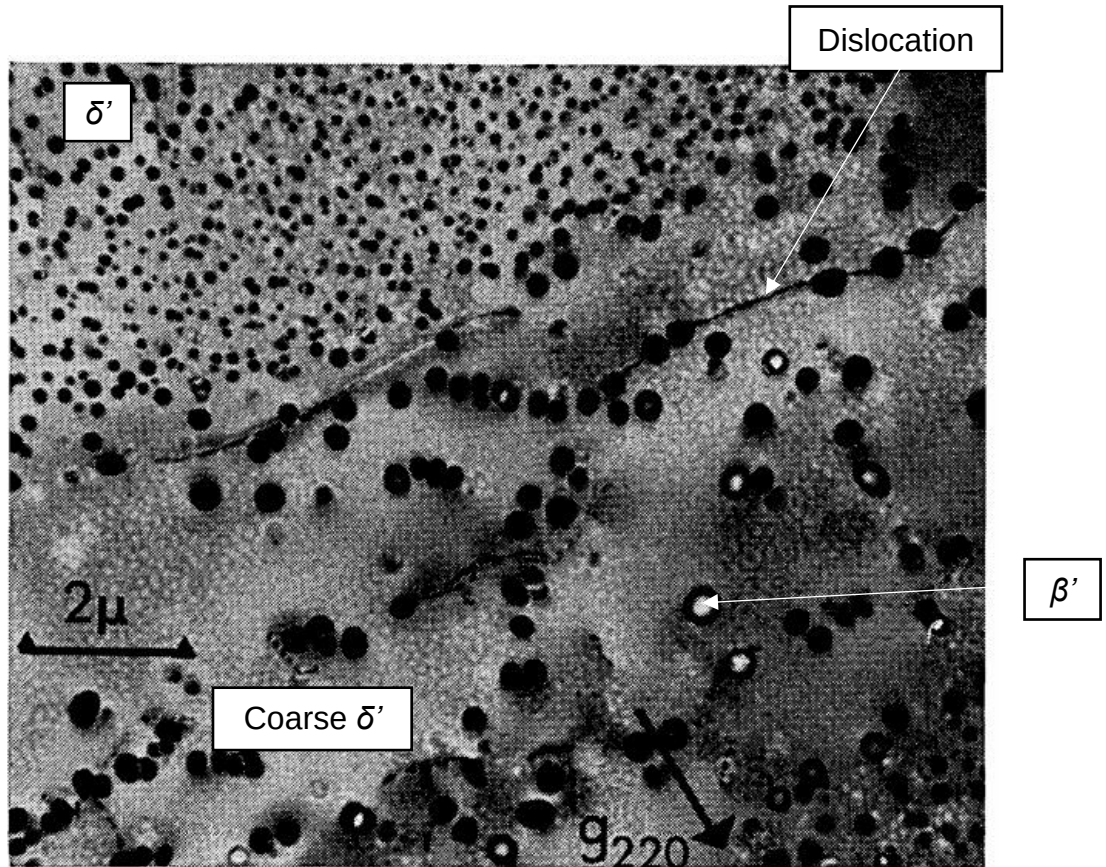


**Figure 9:** TEM micrograph showing  $\delta'$  precipitates (dark circles) with preferential coarsening (larger dark particles) along dislocations (dark lines). (The white particles are suspected to be  $\beta'$  ( $\text{Al}_3\text{Zr}$ ) (Williams and Edington, 1975)<sup>22</sup>.

An early proposal for the precipitation of the  $\delta$  precipitates put forward by Shchegoleva and Rybalko in 1976 (as cited by Williams, 1981)<sup>2</sup> suggested that the formation of  $\delta$  was due to the transformation of the  $\delta'$  precipitates from an F.C.C to a B.C.C lattice structure. However, it was noted by Williams that the theory contained certain assumptions about the  $\delta'$  precipitates and argued that

evidence existed in support of independent, heterogeneous nucleation of  $\delta$ . The independent formation of  $\delta$  was also confirmed by Venables, Christodoulou and Pickens (1983)<sup>32</sup>, who concluded that  $\delta$  nucleates at high energy sites such as grain boundaries and dislocations, which correlates with the heterogeneous nature of the nucleation of  $\delta$ .

Although the nucleation of  $\delta$  has been shown to be independent of  $\delta'$ , further coarsening and growth of  $\delta$  results in dissolution of the surrounding  $\delta'$  precipitates and gives rise to Precipitate Free Zones (PFZs). As the  $\delta$  particles grow, Li is removed from the solid solution which results in the dissolution of the smaller surrounding  $\delta'$  particles. resulting in the aforementioned PFZs at grain boundaries and the formation of coarse  $\delta$  precipitates (Williams, 1981)<sup>2</sup> and (Venables, Christodoulou and Pickens, 1983)<sup>32</sup>.

### **2.1.2 $\text{Al}_2\text{CuMg}$ ( $S'$ ) and ( $S$ ) precipitates.**

$S'$  and  $S$  ( $\text{Al}_2\text{CuMg}$ ) precipitation is initiated by Mg additions to Al-Li alloys which contain Cu. Al-Li alloys with higher Li and lower Cu additions (typically with a ratio of 3:1 Mg:Cu), result in a lower concentration of the  $T_1$  precipitates (see Section 2.1.4) and the  $S$  precipitates becomes the secondary Cu-based precipitates. Increasing the ratio of Mg to Cu will completely suppress  $T_1$  growth. Initial precipitation of the  $S$  precipitates is reportedly slow (Gregson et al., 1987)<sup>9</sup>. Work undertaken by Gregson and Flower (1984)<sup>33</sup> noted that the heterogeneous nucleation of the  $S$  precipitate is predominantly located at quenched-in dislocations in the matrix – and for Al-Li alloys (which reportedly

exhibit a lack of such defects) this was a limiting factor in promoting S precipitate formation throughout the matrix. Whilst homogenous nucleation of the S precipitate is theoretically possible (acknowledged and cited by Gregson and Flower, 1984)<sup>33</sup>, it was concluded that the necessary required lengthy heat treatment was not deemed to be commercially viable.

Although no specific times are given by Gregson and Flower (1984)<sup>33</sup>, this requirement is assumed to be in excess of a 31 hour overaged condition (170°C/3 h + 190°C/28 h). Therefore, the alternative option was to increase the number of dislocation sites on which the S precipitates could nucleate heterogeneously by cold working the material by stretching (which is detailed further in Section 2.2.4).

One notable feature about the Al<sub>2</sub>CuMg precipitate is that as it lacks any Li content during formation and therefore does not affect the precipitation of the  $\delta'$  precipitates. Gomiero et al. (1991)<sup>34</sup> state that *"The  $\delta'$  precipitates grows and coarsens independently at a rate that is hardly affected by the presence of Cu or Mg"*. Furthermore, precipitation of the S precipitate does not contribute to the formation of PFZs unlike Al<sub>2</sub>CuLi (T<sub>1</sub>) precipitates (Prasad, Gokhale and Wanhill, 2014)<sup>35</sup>.

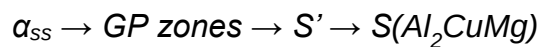
A predominant reasoning for increasing the amount of S precipitates in the microstructure of an Al-Li alloy originates from the observations that the primary strengthening precipitate ( $\delta'$ ) is susceptible to planar slip (as detailed

in Section 2.1.1). This was (at the time) considered to be one of the primary reasons for poor fracture toughness properties in Al-Li alloys (which is further detailed in Section 2.3.2). Gregson and Flower (1984)<sup>33</sup> surmised that the open-packed structure of the S precipitates would not be “penetrated by dislocations” resulting in dislocation bowing (by Orowan looping) and therefore by increasing the volume fraction of S precipitates, there would be a reduction in susceptibility of Al-Li alloys to planar shear resulting in an overall improvement in toughness properties.

However, it should be noted that Khireddine, Rahouadj and Clavel (1987)<sup>36</sup> did show that under low cyclic fatigue conditions, the cyclic movement of dislocations at persistent slip bands is sufficient to result in shearing of S’ precipitates.

#### 2.1.2.1 Clarification of the S’ and S precipitate designations.

The precipitation sequence for the S precipitate that was originally summarised by Silcock (1961)<sup>37</sup> is as follows:



Furthermore (Prasad, Gokhale and Wanhill, 2014)<sup>35</sup>, cite crystal lattice parameters for ‘S’ and S crystals:

$$S' = a \ 0.405 \text{ nm}, b \ 0.916 \text{ nm and } c \ 0.720 \text{ nm}.$$

$$S = a \ 0.400 \text{ nm}, b \ 0.923 \text{ nm and } c \ 0.714 \text{ nm}.$$

Noting that 'S' and S are very similar in orientation and morphology, Prasad et al. refer to Gomiero et al. (1991)<sup>34</sup> 'S' as an epitaxial version of S. However, in the same section of literature Prasad et al. also consider the views of Radmilovic et al. (1989)<sup>38</sup> (and consequently Flower and Gregson, (1987))<sup>39</sup> that during their observations of alloys (with and without Li additions) there was no clear evidence to suggest the existence of separate S' and S precipitates and that the S' notation should cease to be recognised. For ease of clarity the author is in agreement with this statement and the S precipitate will singularly be referred to from now on.

### 2.1.3 $\beta'$ ( $\text{Al}_3\text{Zr}$ ).

$\beta'$  is the only precipitate in Al-Li 8090 which contains Zr. Primarily used as a grain refiner, Zr inhibits recrystallisation (see Section 2.2.3) and therefore the amount of Zr in an Al-Li alloy can have substantial effects on the mechanical properties in the longitudinal and short-transverse orientations.  $\beta'$  precipitates are isostructural with  $\delta'$  and can result in a bimodal distribution of  $\beta'$  surrounded by  $\delta'$  precipitates, Figure 9 (Williams and Edington, 1975)<sup>22</sup>.

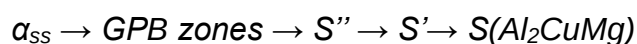
### 2.1.4 $T_1$ ( $\text{Al}_2\text{CuLi}$ ) and $T_2$ ( $\text{Al}_6\text{CuLi}_3$ ).

The  $T_1$  and  $T_2$  precipitates are both Cu containing precipitates.  $T_1$  precipitates are hexagonal lath shaped precipitates (akin to the morphology of the S precipitates, although S precipitates are orthorhombic lath shaped) but where the S precipitate is present in ratios equal to or greater than Mg 3:1 Cu,  $T_1$  is predominant in lower Mg containing environments. The  $T_2$  precipitates at high angle grain boundaries and is similar to the  $\delta$  precipitates. Both the  $T_2$  and the

$\delta$  precipitate remove Li from the solute, resulting in PFZs at the grain boundaries.

#### 2.1.4.1 Guinier-Preston and Guinier-Preston-Bagaryatsky zones.

It should be noted that further to Silcock's (1961)<sup>37</sup> S precipitation sequence, Bagaryatsky (1952) proposed an additional sequence<sup>40</sup>:



The term Guinier-Preston-Bagaryatsky (GPB) zone is first mentioned by Silcock (1961)<sup>37</sup>, however the reference that cites Silcock's work (Prasad, Gokhale and Wanhill, 2014)<sup>35</sup> only refers to GP zones in the S precipitation sequence. The use of GPB zone originated from the suggestion by Silcock (1961)<sup>37</sup> that GPB zones may differ from Al-Cu Guinier-Preston (GP) zones (Wang and Starink, 2005)<sup>41</sup>. However, there is significant contradictory data on the validity of GPB zones and their contribution in the formation of S precipitates. A comprehensive review of the literature is given by Wang and Starink (2005)<sup>41</sup>. The term 'GPB zone' is not employed hereon, instead 'GP zone' (see Section 2.1.4.2) is used to define both terms, apart from when the literature directly makes reference to GPB zones.

#### 2.1.4.2 Guinier-Preston zones.

Clustering of solute atoms is a well-known and understood strengthening mechanism in aluminium alloys (Jacobs, 1999)<sup>42</sup> especially in Al-Cu-Mg alloys which naturally begin to age harden immediately after quenching (Wilm, 1911)<sup>43</sup>. In Al alloys, Guinier-Preston (GP) zones are ordered, copper-rich

clusters of atoms that are only a few atomic layers thick. These clusters are coherent with the matrix result in elastic coherency strains (Raghavan, 2006)<sup>44</sup>, Figure 10. GP zones provide two essential elements to the strengthening of Al alloys; firstly, it is the coherency strain that initially impedes dislocation movement, increasing the strength of the material. Secondly, during hardening transition precipitates ( $\theta''$  in Al-Cu alloys) nucleate at GP zones.

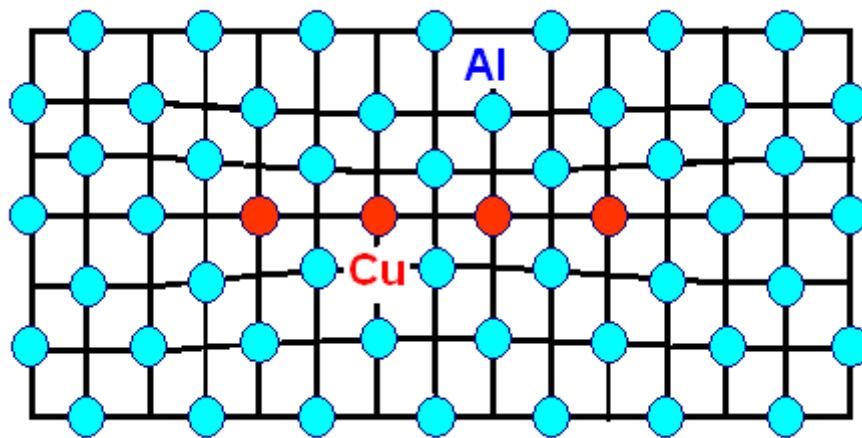


**Figure 10: Illustration of a cluster of Cu atoms in an Al matrix and the resultant coherency strain<sup>45</sup>.**

## 2.2 Notable characteristics of Al-Li alloys.

Whilst the perceived benefits of alloying Al with Li are widely acknowledged (and were covered in Section 1.1) as with all alloying systems, there are inherent advantages and disadvantages. Al-Li alloys are no exception to this and a number of characteristics of Al-Li alloys have been investigated over the past 30 years. Some notable advantages and disadvantages are detailed in this section and all are attributable to 2<sup>nd</sup> generation Al-Li 8090. Although some



of the disadvantages are reported to have been overcome by the newer 3<sup>rd</sup> generation Al-Li alloys, mainly as a result of a reduction of Li content (< 2 wt%). Although a separate question posed is; are these 'truly' to be classed as Al-Li alloys or conventional Al alloys containing Li.

### **2.2.1 The modulus of elasticity of Al-Li alloys.**

The elastic (Young's) modulus of (pure) Al is 69 GPa<sup>46</sup>. This value only varies slightly across conventional Al alloy families with a range of 69-73 GPa. Commercial 2<sup>nd</sup> generation Al-Li alloys (2090, 2091 and 8090) exhibit a ~7% increase in Young's modulus (ASM Handbook)<sup>47</sup> (which is around 74 GPa).

Work undertaken by Müller, Bubeck and Gerold (1985)<sup>48</sup> showed that the Young's modulus for binary Al-Li alloys at room temperature increased with additions of Li (in 8.5 and 9.6 at% Li alloys respectively). Müller, Bubeck and Gerold identified that the elastic shear constants (for some crystal orientations in the alloys), were considerably higher in the  $\delta'$  precipitates than in the Li depleted matrix. Broussaud and Thomas (1985)<sup>49</sup> determined that the increase in Young's modulus is directly attributable to the precipitation of  $\delta'$  within the matrix (in comparison with as-quenched and aged alloys). However, they stated that variations in volume fraction and coarsening of  $\delta'$  precipitates had no noticeable effect.

The  $\delta'$  precipitates are not the only precipitates that have been found to have an effect on the Young's modulus of Al-Li alloys: Agyekum et al., (1985)<sup>50</sup> calculated that in Al-Li-Cu-Mg alloys, there is an insufficient volume fraction of

the  $\delta'$  precipitates to support the theory that  $\delta'$  is responsible for the increase in Young's modulus. In an alloy containing 8.37 at% Li, which was aged for 36 hours at 150°C, the recorded Young's modulus of 80.9 GPa was attributed to the precipitation of the  $T_1$  precipitates.

Further aging of numerous alloys from 50 hours at 163°C to 150 hours at 163°C and subsequent testing, was found to support Broussaud and Thomas's (1985)<sup>49</sup> observations that the increase in Young's modulus occurs at the early stages of the age hardening process. In addition, Agyekum et al., (1985)<sup>50</sup> also observed that exceeding the solubility limits for Li results in large equilibrium particles (which are likely to be  $\delta$  precipitates) which are shown to have a negative effect on the elastic properties. (This may also be a contributory factor as to why 2090 and 2091 alloys are classed as Al-Li alloys and not 2xxx series Al-Cu alloys which contain Li).

A more recent publication regarding modern Al-Li alloys by Elagin and Zakharov (2013)<sup>51</sup> states that the increase of Young's modulus in Al alloys (when directly attributable to alloying) is a result of when the modulus of elasticity of a material composed of a pure alloying element is greater than the modulus of elasticity of the matrix. However, Li is an exception to this rule. Elagin and Zakharov (2013)<sup>51</sup> describe Li as having "*virtually no elasticity*" whilst having a substantial effect on modulus of elasticity of Al-Li alloys. The authors further suggest that this effect is a result of the solid solution of Li in

Al – in addition to the standard metallic interatomic bonding that would be expected from a metallic material.

The solid solution is likely to contain stronger bonds with ionic and covalent characteristics, which is consistent with the reduced electrical and thermal conductivities of Al-Li alloys (when compared to conventional Al alloys). It is still unclear to what the increase in the modulus of elasticity can be attributed to. In any case, the increase in Young's modulus that is one of the major reasons factors for the interest in this alloy system by the aerospace industry.

### **2.2.2 Precipitate free zones.**

The following paragraphs appertain to all Al-Li alloys which are strengthened through the homogenous nucleation and growth of  $\delta'$  precipitates from a supersaturated solid solution. Accompanying these precipitates will be the heterogeneous nucleation of  $\delta$  and S precipitates at the grain boundaries within the microstructure. The mechanical properties of these alloys are affected by the formation of precipitate free zones (PFZs) adjacent to the grain boundaries, (Figure 11). The primary reasons as stated by Polmear (1995)<sup>52</sup> for the growth of PFZs are:

- The diffusion of atoms from the solute into a grain boundary assists in the formation of the precipitates within the boundary. These precipitates are typically larger than the matrix precipitates ( $\delta$  and  $T_2$ ) resulting in a solute depleted region either side of the grain boundary.

- In addition, grain boundaries acting as vacancy 'sinks' and reduce the available levels of vacancies for the nucleation of the matrix precipitates which inhibits their formation in the region adjacent to the boundaries.

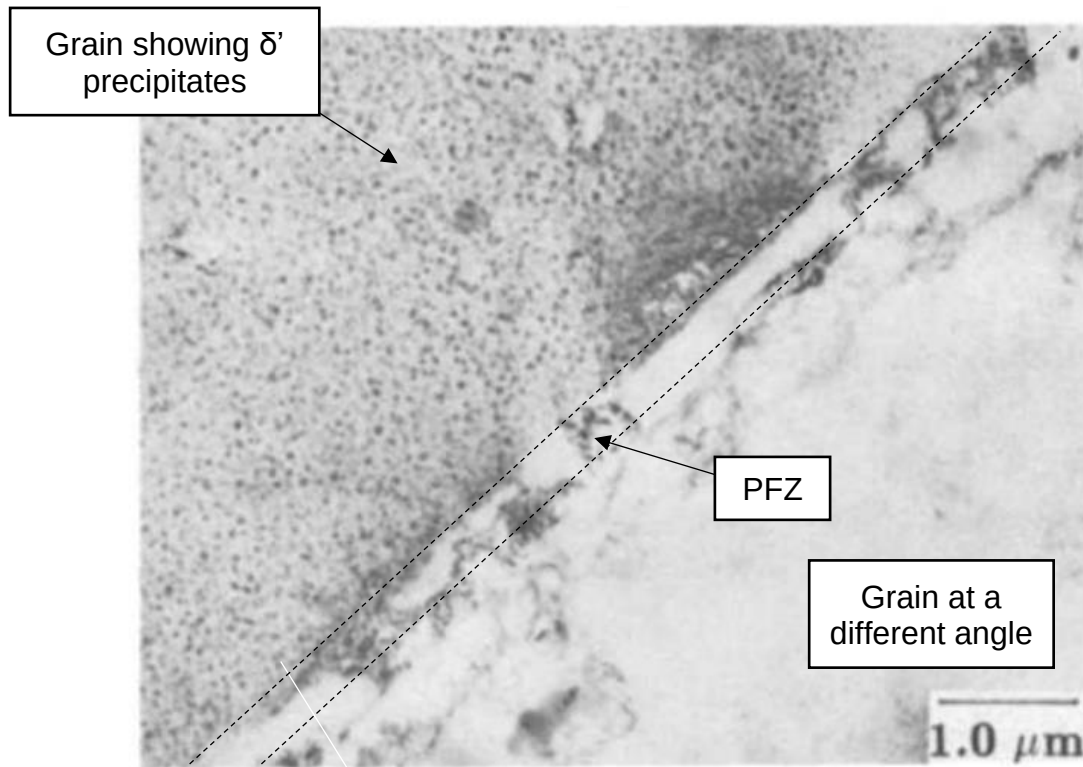


**Figure 11: TEM micrograph showing the precipitate free zone in an Al-Li alloy (Jha, Sanders and Dayananda, 1986)<sup>53</sup>.**

In the opinion of Edgar, Starke and Quist (1988)<sup>3</sup>, the PFZs are weaker than the matrix. Coupled with heterogeneous precipitation of grain boundary precipitates, these weak PFZs result in strain localisation at grain boundaries and this has a detrimental effect on the fracture toughness and ductility of the material. However, an argument against this theory is discussed in Section 2.3 “Mechanical failure of Al-Li alloys”.

### **2.2.3 Anisotropy and recrystallisation of Al-Li alloys.**

Al-Li alloys are known to exhibit a high degree of anisotropy in the direction of primary grain flow. Work undertaken by Palmer et al. (1985)<sup>54</sup> identified that the relationship between the grain size and microstructural texture of a material can have substantial effects on the mechanical properties of a material. One such relationship is due to the anisotropic microstructures that exist in the three orthogonal directions in rolled products. This results in a tendency for enhanced strength when tensile loading is applied in the longitudinal microstructure direction, and inferior mechanical properties when tensile loading is applied in the transverse orientations of microstructures. This is commonly given as one of the reasons why un-recrystallised Al-Li alloys exhibit poor ductility and fracture toughness.

There are number of factors that are known to affect the recrystallisation capabilities of Al-Li alloys. Work undertaken by Lin (1982)<sup>55</sup> identified a relationship between yield strength and the amount of recrystallisation within the microstructure (Lin had been studying the effects of Cd additions to Al-Li-Cu alloys). Microstructural examination by Palmer et al. (1985)<sup>54</sup> identified a variation in the recrystallisation of Al-Li alloys containing Zr. As can be seen in Figure 12, by increasing the Zr content in the alloy, recrystallisation in the microstructure is increasingly inhibited. Comparison of binary Al-Li, Al-Zr and a ternary Al-Li-Zr alloys by Makin and Stobbs (1985)<sup>56</sup> revealed that in the binary alloys, the Zr and Li additions were insufficient to inhibit recrystallisation of the grain structure. However, in the ternary alloy the recrystallisation had

been inhibited. Makin and Stobbs proposed multiple theories for the recrystallisation inhibition, two of which follow. Firstly, Li loss from solid solution results in an acceleration of recrystallisation. Secondly, an inhomogeneous distribution of the Al-Zr particles ultimately produces a heterogeneous microstructure which inhibits the recrystallisation.

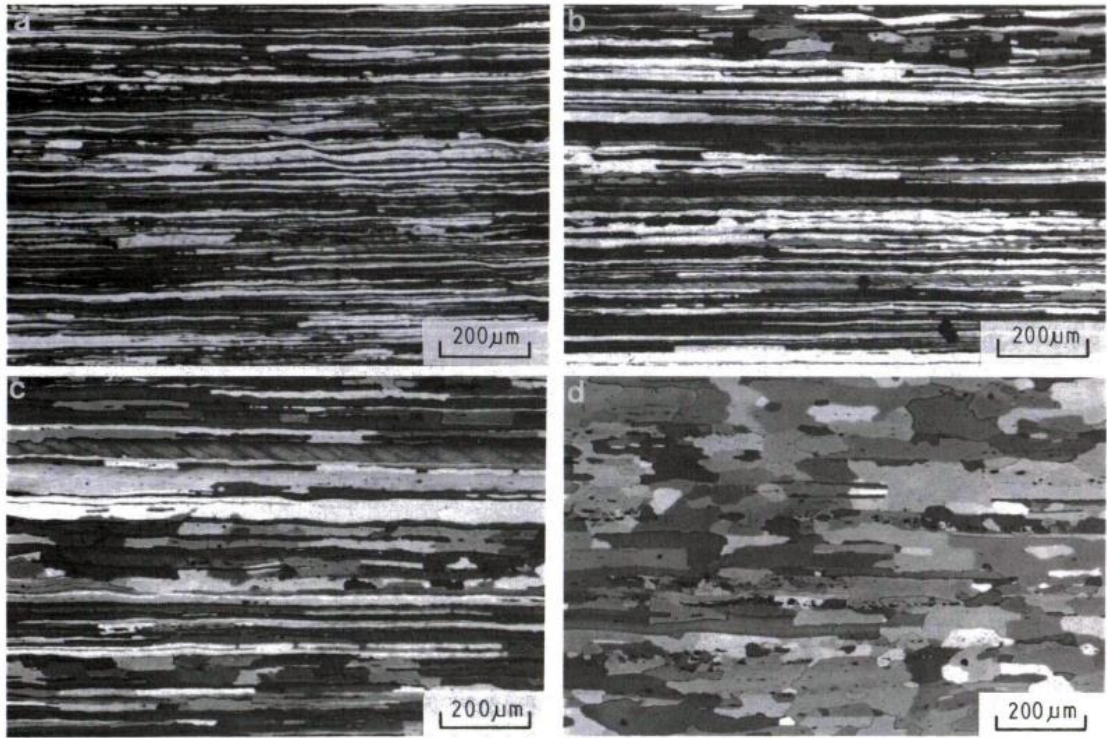


**Figure 12:** Palmer et al.'s images showing the effect of Zr on the recrystallisation of Al-Li grain structure. (a) 0.16% Zr (b) 0.13% Zr (c) 0.09% Zr and (d) 0.05% Zr<sup>54</sup>.

In either case, Makin and Stobbs concluded that the combination of Li *and* Zr within an Al alloy were sufficient to inhibit recrystallisation. In addition to the variations in Zr content, Palmer et al. (1985)<sup>54</sup> discovered that variations in the amount of cold work applied, over-ageing temperature and heat-up rate to the over-ageing temperature all had effects on the recrystallisation of the grain structure.

By applying 87.5% cold work (the samples were hot rolled to a thickness of 4mm, solution heat treated and cold rolled to 37.5%, 62%, 75% and 87.5% CW) the grain structure was almost equiaxed following heat treatment. Lower values of cold work displayed a 'zig-zag' microstructure following heat treatment, which was attributed to intense amounts of planar slip from the cold work, Figure 13. Over-ageing of the grain structure prior to cold working was found to produce sporadic recrystallisation without a uniform grain size. An increased heat up rate to the recrystallisation temperature of 3000°C/min produced a uniform recrystallised grain structure.

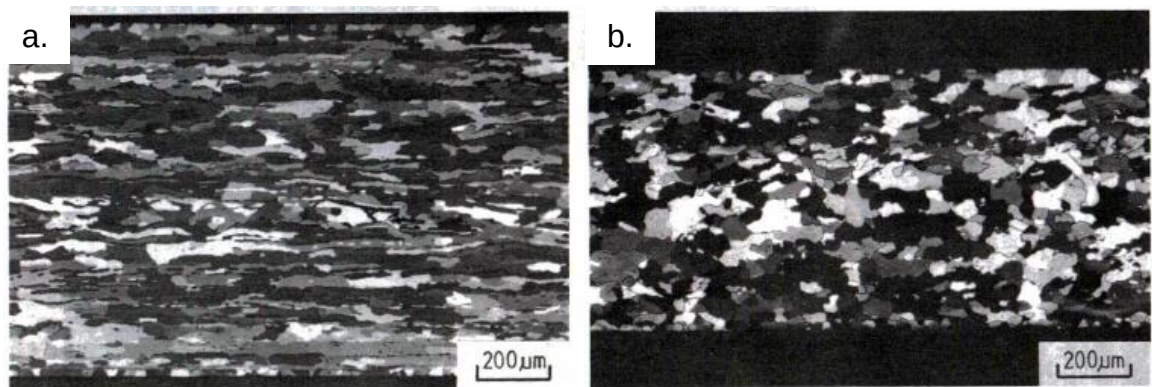


**Figure 13: Showing the effect of cold work on recrystallisation (a) 75% cold work (b) 87.5% cold work (Palmer et al., 1986)<sup>54</sup>.**

The mechanical properties of the recrystallised material were found to be inferior to the as-received anisotropic material (both materials were 1 mm thick sheet). The ultimate tensile strength and 0.2% proof stress strength results were lower than their anisotropic counterparts. The % elongation results were shown to be mixed as can be seen in Figure 14. Although the mechanical testing results were lower than the original specification material, the findings

revealed that recrystallised microstructures can be produced in Al-Li alloys. Ultimately however, the mechanical properties of the isotropic recrystallised material were inferior to the traditionally processed anisotropic material tested in the longitudinal grain orientation. This is arguably an important reason in support of the use of anisotropic material in high strength applications with a primary loading orientation, such as aircraft main lift frames. The longitudinal grain orientation would be aligned along the axis of the lift frame to give the maximum strength (and toughness) in the desired direction.

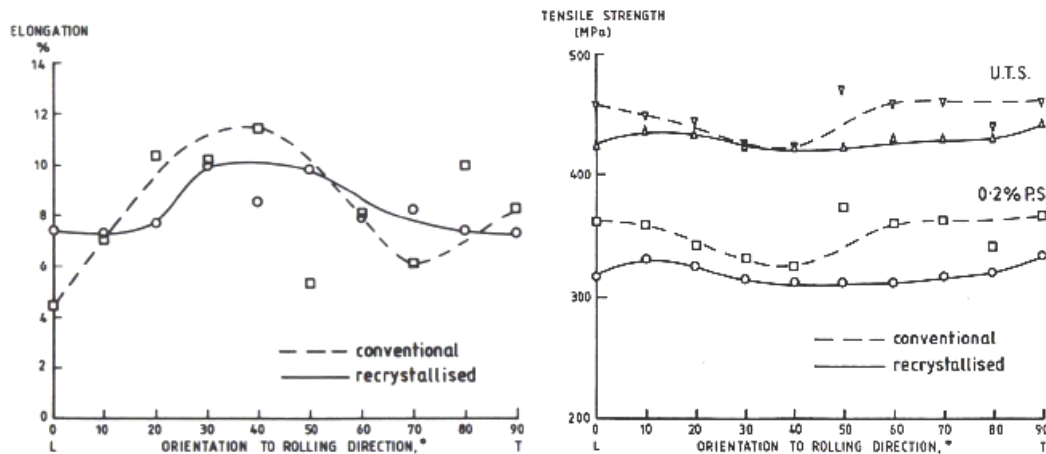


**Figure 14: Showing the mechanical properties of 1mm recrystallised sheet against traditionally anisotropic material (Palmer et al., 1986)<sup>54</sup>.**

#### 2.2.4 Mechanical stretching of Al-Li alloys by tensile loading.

Many alloy systems are cold worked, in tension (by stretching) or compression to achieve desired mechanical properties. Xiaoxin and Martin (1989)<sup>57</sup> conducted research on how the varying percentage of stretching and heat



treatments by tensile loading had an effect on the distribution of the  $\delta'$  and S precipitates in 8090 Al-Li alloy. Xiaoxin and Martin found that by increasing the degree of stretch in a material between the dual age heat treatment, (following solution heat treatment but prior to artificial aging), the volume fraction and average length of the S precipitates also increased. These findings are shown in Table 2.

**Table 2: Showing the average length and volume fraction of S precipitates particles in relation to heat treatment and % cold work. (Reproduced from Xiaoxin and Martin, 1989)<sup>57</sup>.**

| <i>Treatment</i> | <i>Average length<br/>(nm)</i> | <i>Volume fraction<br/>(%)</i> |
|------------------|--------------------------------|--------------------------------|
| Unstretched      | 403 $\pm$ 12                   | 0.15                           |
| Duplex Aged      | 319 $\pm$ 17                   | 0.23                           |
| 4% stretched     | 412 $\pm$ 12                   | 0.63                           |
| 7% stretched     | 467 $\pm$ 18                   | 1.12                           |

The reason for the increase in length and volume fraction of the S precipitates was attributed to the increase in dislocations present in the microstructure as a result of the stretching process, therefore providing more nucleation sites for S precipitates. The 0.2% proof stress of the 7% stretched material was found to be inferior to the 4% stretched material. This was attributed to the S precipitates in the 7% material being coarser and more widely dispersed than the 4% material, resulting in a decrease in mechanical properties, indicating that there is an optimal S precipitate particle size and distribution.

Xiaoxin and Martin (1989)<sup>57</sup> also reported that the distribution and volume fraction of  $\delta'$  particles was however, not affected by stretching process. These observations were further reinforced by the observations of Pitcher, Stewart and Gupta (1991)<sup>58</sup>.

### **2.2.5 Thermal instability of Al-Li alloys.**

In the mid 1990's the Defence Research Agency published findings of work undertaken regarding the effect of the exposure of multiple Al-Li alloys to an environmental temperature of 70°C. Initial (unpublished) findings by McDarmaid and Starr (1990)<sup>59</sup> raised concerns that 1.6 mm thick 8090 and 2091 Al-Li sheet in what was considered to be a damage tolerant condition (T81 – solution heat treated, cold worked and artificially aged) was found to exhibit an increase in strength and decrease in fracture toughness following exposure to 70°C for a period of 12 weeks<sup>vi</sup>.

McDarmaid further investigated the effect of ambient (21°C) and 70°C temperatures in two complementary publications that were published within a few days of each other (McDarmaid, 1995<sup>60,61</sup>). The summary of the ambient temperature testing determined that the mechanical properties of 1.6 mm thick Al-Li 8090 T81 sheet had not changed over a period of 4.6 years. However, the 70°C exposure and subsequent tensile testing reported that after 12

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<sup>vi</sup> *It must be noted that the zirconium content of the 8090 and 2091 alloys was altered from 0.12% to 0.06% around this time period. Therefore, the reduction in zirconium may have had an effect on the recrystallisation of the alloy, which could have changed the results of the mechanical performance when compared with a current Al-Li 8090 which specifies 0.12% zirconium.*

weeks, there was a minor variation in UTS and 0.2% proof stress but a 23% reduction in ductility (although it should be noted that the report focussed on the fracture toughness properties). From this work McDarmaid concluded that both 8090 and 2091 Al-Li alloys are “*less thermally stable*” than their conventional alloy counterparts at the same strength level. McDarmaid (1995)<sup>60</sup> does not indicate which specific alloys are considered to the conventional counterparts. However, as has already been mentioned ‘Lital A’ (8090) was intended to replace 2014-T6 (Peel, Evans & McDarmaid, 1985)<sup>8</sup>.

#### 2.2.5.1 Work into the causes of thermal instability of Al-Li alloys.

Defined by Zakharov (2003)<sup>62</sup>, in the context of Al alloys thermal stability is:

*“... understood as the capacity of heat-treated semiproducts to preserve the mechanical and chemical properties in long-term aging at enhanced temperatures (70 – 100°C) corresponding to solar or operational heating.”<sup>vii</sup>*

Earlier work by Nobel, Harris and Dinsdale (1996)<sup>28,viii</sup> determined that all Al-Li alloys regardless of composition show evidence of ‘embrittlement’ when exposed to temperatures  $\geq 100^\circ\text{C}$ . This ‘embrittlement’ was attributed to the “*formation of significant amounts of  $\delta'$  ( $\text{Al}_3\text{Li}$ ) during exposure.*” Noble Harris and Dinsdale (1996)<sup>28</sup>, attempted to identify the scientific reason for the thermal instability of Al-Li 8090 at 70°C, 100°C and 130°C after 1000hr

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<sup>vii</sup> This is an expansion on his earlier definition in 1999.<sup>64</sup>

<sup>viii</sup> Note 1996 is the latest date on the document – there is no official publication date on the document.

exposures. However, they encountered complex issues which were attributed to interactions between the precipitates present in the binary, ternary and quaternary alloys in Al-Li 8090 material. Therefore, they centred their work on binary Al-Li alloys that would allow for interpretation of the behaviour of ternary and quaternary Al-Li alloys (Zakharov, 2003)<sup>63</sup>. The binary alloys that were investigated contained 1.7 and 2.6 wt% Li, respectively.

TEM examination was carried out on the two Al-Li alloys by Nobel, Harris and Dinsdale (1996)<sup>28</sup>. Following solution heat treatment these alloys were subjected to a 'pre-age' heat treatment (24 h at 150°C) and were held for 1000 hours at 70°C, 100°C and 130°C, respectively. Following the pre-age heat treatment, the typical  $\delta'$  precipitate diameters were measured as 14.5 and 11.0 nm, for the 1.7 and 2.6 % Li alloys, respectively. The 1000 hr exposure at 70°C had no significant effect on the precipitate size of the existing  $\delta'$  precipitates in either binary Al-Li alloys, however a fine dispersion of new  $\delta'$  precipitates with a diameter of 2 nm was observed between the established  $\delta'$  in the 1.7% Li alloy. A similar feature was noted within the alloy containing 2.6 % Li – however measurement was noted to be difficult due to the high volume fraction of  $\delta'$  resulting from the previous 150°C heat treatment.

Following the pre-age heat treatment, exposure of the binary Al-Li alloys to 100°C for 1000 hours resulted in the growth of  $\delta'$  in both alloys. The coarse (already established)  $\delta'$  precipitates increased in diameter from 14.5 to 18.5 and 11.0 to 13.2 nm, respectively. Further to this the fine dispersion of  $\delta'$  had

grown to a diameter of 5nm – resulting in a bimodal distribution of the larger coarse  $\delta'$  precipitates (classed as dislocation distributed) and a fine uniform dispersion of  $\delta'$  in the matrix. Further growth of the pre-existing coarse and fine  $\delta'$  precipitates was achieved by the 130°C heat treatment for 1000 hr. A summary of the increase in precipitate diameters is shown in Table 3. It should be noted that all of these heat treatments were conducted in parallel.

**Table 3: Showing the % increase in  $\delta'$  precipitate diameters for 1.7% and 2.6% Li binary Al-Li alloys.**

|                           | 1.7%               |              | 2.6%               |              |
|---------------------------|--------------------|--------------|--------------------|--------------|
|                           | $\varnothing$ (nm) | % $\uparrow$ | $\varnothing$ (nm) | % $\uparrow$ |
| Pre age HT (24h at 150°C) | 14.5               | N/A          | 11.0               | N/A          |
| 1000h at 70°C             | 14.5               | 0.0          | 11.0               | 0.0          |
| 1000h at 100°C            | 18.5               | 27.6         | 13.2               | 20.0         |
| 1000h at 130°C            | 28.7               | 55.1         | 19.2               | 45.5         |

From these findings, Nobel, Harris and Dinsdale (1996)<sup>28</sup> determined the change in mechanical properties were a direct result of  $\delta'$  precipitation. For the 70°C testing, the mechanism was primarily determined to be the formation of fine  $\delta'$  precipitates within the matrix and for the  $\geq 100^\circ\text{C}$  testing both fine dispersion and coarsening of established  $\delta'$  precipitates were attributed with causing the change in mechanical properties – with the coarsening effect being the primary factor in the increase in strength and reduction of fracture toughness.

As these findings primarily indicate, the resultant change in mechanical properties is due to growth and coarsening of  $\delta'$  particles. In order for this to occur there must be available Li and Al within the solid solution to allow for the nucleation and growth of  $\delta'$ . In support of this, Zakharov (2003)<sup>62</sup> states:

*“...the solid solution in Al-Li alloys is not stable and easily decomposes even after the low supersaturation that always accompanies hardening and artificial aging.”*

Zakharov (1999)<sup>64</sup> argues that all Al alloys including non-heat treatable Al alloys are susceptible to thermal instability to some extent<sup>ix</sup>. For example, annealed AMg2 (a Russian aluminium alloy, which is similar to the American designation alloy 5251) alloys exhibit a reduction of manufacturing properties at ambient temperature over several years.

The loss of ductility in Al-Mg alloys (specifically AMg2, AMg5 and AMg6, Zakharov, 1999<sup>64</sup>) is primarily attributed to the partial retention of a solid solution of Mg within the Al matrix. This retention occurs as a result of aging or low-temperature heating and subsequent decomposition of  $\beta$  precipitates ( $\text{Al}_3\text{Mg}_2$ ) which causes Mg to segregate to the grain boundaries, which impacts on the ductility of the alloy. Considering Al-Li alloys, Zakharov (1999)<sup>64</sup> suggests that a retained residual supersaturation of Li after quenching and

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<sup>ix</sup> *Naturally all metals are subject to thermal instability at a certain temperature - however in this context the temperature range is still 70 – 100°C.*

aging, coupled with the low thermal stability of Al-Li, allows for the decomposition of the solid solution and the formation of  $\delta'$  – which results in an *‘undesirable change in mechanical properties’*.

Work undertaken by Zakharov (1999)<sup>64</sup> using the quaternary alloy 1420 (Al - 5% Mg - 2% Li - 0.1%Zr) identified a notable change in mechanical properties. This followed solution heat treatment, quenching and subsequent natural aging from ambient temperature to 70°C for up to 10,000 hours (Table 4). As anticipated, with an increase in strength these results were accompanied with a reduction in elongation.

Furthermore, this effect is not limited to a single alloy group as Zakharov (2003)<sup>62</sup> reported similar results with alloy 1464 (Al – 3% Cu – 1.7% Li – 0.5 % Mg – Zr – Sc) which was aged at 85°C over a period of up to 1000 hours – the resultant effect on the mechanical properties is shown in Figure 15.

**Table 4: Showing the effect of aging of alloy 1420 at 70°C for a number of time periods. Reproduced from Zakharov (1999)<sup>64</sup>.**

| $\tau$ , h | $\sigma_r$ , Mpa | $\sigma_{0.2}$ , Mpa | $\delta$ , % |
|------------|------------------|----------------------|--------------|
| -          | 450              | 360                  | 12           |
| 100        | 500              | 360                  | 13           |
| 500        | 530              | 390                  | 11           |
| 2000       | 550              | 400                  | 9            |
| 5000       | 570              | 420                  | 8            |
| 10,000     | 560              | 460                  | 6            |

As can be seen in Figure 15, the mechanical properties follow a similar pattern to alloy 1420 (Table 4), with an increase in strength and reduction in ductility, albeit with a reduced timescale and additional age hardening heat treatment (120°C for 20 hours + 170°C for 12 hours). This is likely to have had an impact on the rate of increase of strength. However, the elongation is noted to have reduced by half (from 10 to 5%) by the 1000 hour mark, Figure 15. Whereas alloy 1420 (extrapolating from a trend created from the data) exhibited an approximate 10% elongation at the 1000 hr mark.

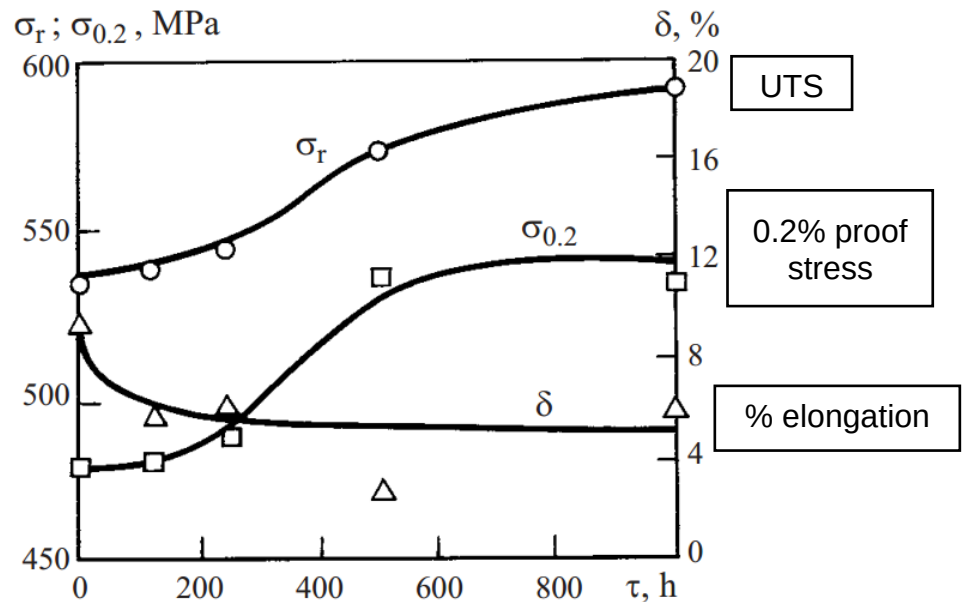


**Figure 15:** Showing the effect of aging at 85°C of alloy 1464 (Zakharov 2003, Fig 1)<sup>62</sup>.

Therefore, from the above it can be determined that Al-Li alloys exhibit a change in mechanical properties at relatively low temperatures (70-130°C). Whilst Zakharov notes a change in properties following exposure at 70°C, Nobel, Harris and Dinsdale (1996)<sup>28</sup> determined that there was no coarsening



of the established  $\delta'$  at 70°C (although this did occur at higher temperatures) they did identify the presence of far smaller  $\delta'$  precipitates.

Sections 2.1 and 2.2 are primarily based on the early or original available research data that was undertaken on Al-Li alloys (focusing on 8090) and the subsequent issues up to a period around 1995. It must be noted that post 2000 there are multiple recorded incidents of crashed EH 101 aircraft, a number of which 1710 NAS was involved in investigating<sup>x,xi</sup>. The subsequent failure investigations on these aircraft notably raised questions on the fracture behaviour of Al-Li alloys and how they contrasted with conventional Al alloys. The following Section (2.3) considers work that was primarily conducted post 1995, or is work based on pre-existing research which has gained more prominence<sup>xii</sup> post crashing of Al-Li structured aircraft. The result is a truly international collaborative effort towards unlocking the more intriguing aspects of Al-Li.

### 2.3 Mechanical failure of Al-Li alloys.

Al-Li alloys are often stereotyped for exhibiting brittle intergranular fracture surfaces post overload failure conditions. Overload failure in this instance being classed as a single dynamic event resulting in the failure of a structure or component. These structures or components may or may not have already

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<sup>x</sup> Royal Navy Merlin, RNAS Culdrose 30 Mar 04.

<sup>xi</sup> Canadian Armed Forces Search and Rescue Helicopter, Nova Scotia 13 Jul 06.

<sup>xii</sup> This is personal opinion arising from both the literature review and my work environment.

been weakened by other mechanisms such as cyclic loading from high cycle fatigue and stress corrosion cracking. Notable examples of dynamic failure of structural Al-Li alloy components include multiple reported instances of heavy landings and crashes of EH101 aircraft (Symonds, 2006)<sup>65</sup>. Following overload failure of 8090-T852 forgings, the fracture surface exhibits a brittle intergranular morphology, with additional features consistent with localised ductility found to be present on the surface of the exposed grains. An example of this can be seen in Figure 16, which shows the fracture surface of a tensile test specimen manufactured from an in-service Al-Li main lift frame. This in contrast to conventional Al alloys, which as can be seen in Figure 17, exhibit features consistent with ductile microvoid coalescence.

Al-Li alloys can fail with evidence of ductility on the fracture surface depending on: whether they are in sheet or plate form; heat treatment history and direction of applied stress. Work undertaken by a number of authors (referenced later in Section 2.3) note that failure of Al-Li 8090-T81 sheet (aged at 150°C for 24 hours) primarily exhibited a transgranular shear failure mechanism, with only sporadic areas of brittle intergranular fracture. This gave rise to the argument that Al-Li sheet material varies in many aspects from that of Al-Li plate and therefore, in terms of failure analysis comparisons cannot be drawn between the two. Furthermore, Al-Li 8090 can be heat-treated to a condition that when subjected to tensile overload failure, the fracture surface exhibits features consistent with ductile microvoid coalescence. This surface is without any intergranular indications and therefore exhibits a true ductile fracture surface.

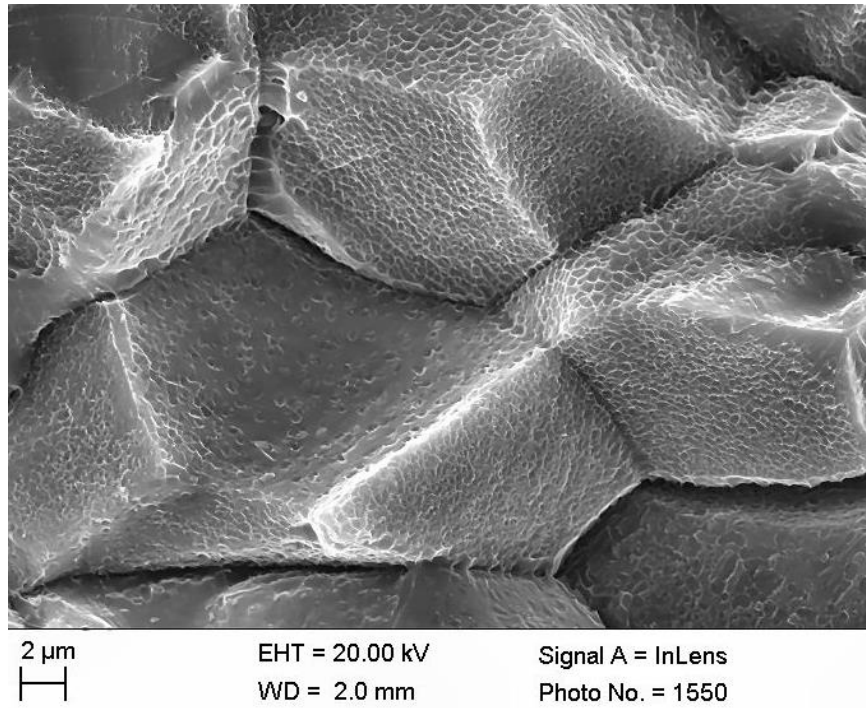


**Figure 16:** Showing the intergranular fracture surface of a failed Al-Li 8090-T852 tensile test piece. (Cusack, 2014)<sup>66</sup>.

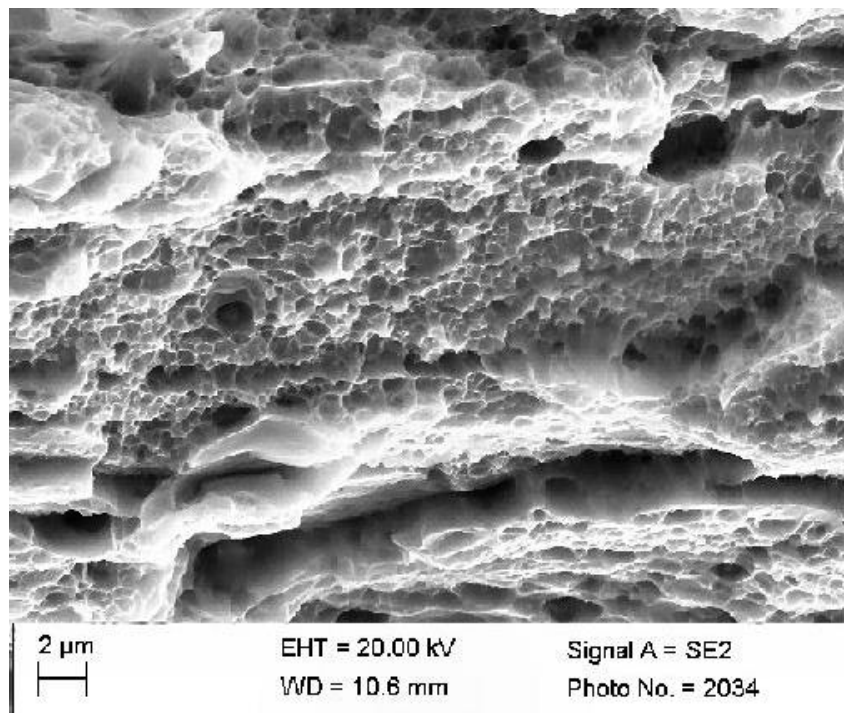


**Figure 17:** Showing features consistent with microvoid coalescence indicating ductile failure of a conventional Al 2024-T4 tensile test piece. (Cusack, 2014)<sup>66</sup>.

It is perhaps more appropriate then to state that all *Al-Li alloys of structural interest* (normally plate or forgings that have been subjected to structurally beneficial heat treatments – such as 8090-T852) are notable for failure in a brittle intergranular manner with localised ductility at the grain boundaries. Therefore, there are limitations to the proposition that Al-Li alloys fail in a brittle intergranular manner.

Pasang et al. (2012)<sup>67</sup> considered a selection of proposed explanations for the primary reason behind the brittle intergranular mechanism (which were originally proposed by two of the authors of Lynch, Muddle and Pasang (2001)<sup>68</sup>. A number of these are detailed in the following section.

### **2.3.1 Grain boundary precipitates and precipitate free zones.**

Pasang et al. (2012)<sup>67</sup> make reference to a theory that a combination of grain boundary precipitates and precipitate free zones (PFZs) can result in intergranular fracture. This is where the comparatively soft PFZ allows for strain localisation at grain boundaries and encourages an intergranular failure mechanism, with grain boundary precipitates facilitating microvoid growth - as stated by Vasudevan and Doherty (1986)<sup>69</sup>:

*“In the Al alloys, where gb (grain boundary) precipitates are formed from the same solute additions as the matrix precipitates, the existence of a pfz (precipitate free zone) is an inevitable consequence of the formation of gb precipitates”.*

The following discusses how PFZs can form and observations of how grain boundary precipitates (and also their absence) can affect the observed tensile failure mode. Although solute depletion is not the only way PFZs can form; grain boundaries act as sinks for excess vacancies and depletion of these vacancies reduces the rate of solute diffusion in a localised region, which in turn affects clustering and GP zone growth.

Further to this, the paper states that in Al alloys (assumed to be conventional Al alloys at this point, however Al-Li alloys are also assessed in this paper), observations of microvoid formation in the grain boundaries of aged alloys post fracture, resulted in dimples that were attributable to grain boundary precipitates. This correlates with work originally reputed to be undertaken by Thomas and Nutting (1957-8)<sup>70</sup>. In addition, Suresh et al., (1986)<sup>71</sup> implied that the ductile microvoids in Al-Li alloys are formed around either  $T_2$  or  $\delta$  precipitates. The latter are known to be large plate shaped precipitates that nucleate at the grain boundaries of Al-Li alloys.

Work undertaken by Lynch, Muddle and Pasang (2001)<sup>68</sup> and Lynch, Wilson and Byrnes (1993)<sup>72</sup> on 8090-T8771 plate revealed that the fracture behaviour of notched cantilever test pieces in the as-quenched Very Under-Aged (VUA) conditions (re-solution heat treated at 545°C for 20 mins and quenched) resulted in a ductile fracture surface. Although ductile microvoid coalescence was found to be present on the fracture surface of the material post re-solution heat treatment, after a very short aging time (1 day at 43°C / 10 days at 21°C

/ 100 days at 3.5°C) the specimens exhibited 100% brittle intergranular surface morphology.

The argument presented by Lynch, Muddle and Pasang (2001)<sup>68</sup> is that the relatively low aging times and temperatures were not sufficient to enable the formation of grain boundary precipitates, or PFZs in the VUA material. SEM analysis of the fracture surfaces revealed smooth intergranular facets, with no indication of the presence of grain boundary precipitates (findings which correlated with TEM foils). Therefore, Lynch et al. (2001)<sup>68</sup> concluded that grain boundary precipitates and precipitate free zones could not be the primary source of intergranular failure in Al-Li alloys.

A similar experiment was undertaken with Al-Li main lift frame material. Two tensile test pieces were solution heat treated at 530°C for 30 minutes, quenched in water and subsequently immersed in liquid nitrogen to halt any microstructural formation at ambient temperature. The test pieces were tensile tested at 0 mins and 5 mins aging time at room temperature (Figure 18 and Figure 19). As can be seen in Figure 18, at 0 mins the fracture surface of the test piece was a ductile fracture surface, showing ductile microvoid coalescence. However, in the +5 mins sample (Figure 19), the ductility was greatly reduced and brittle facets were beginning to appear, showing the rapid change towards a brittle failure mode.

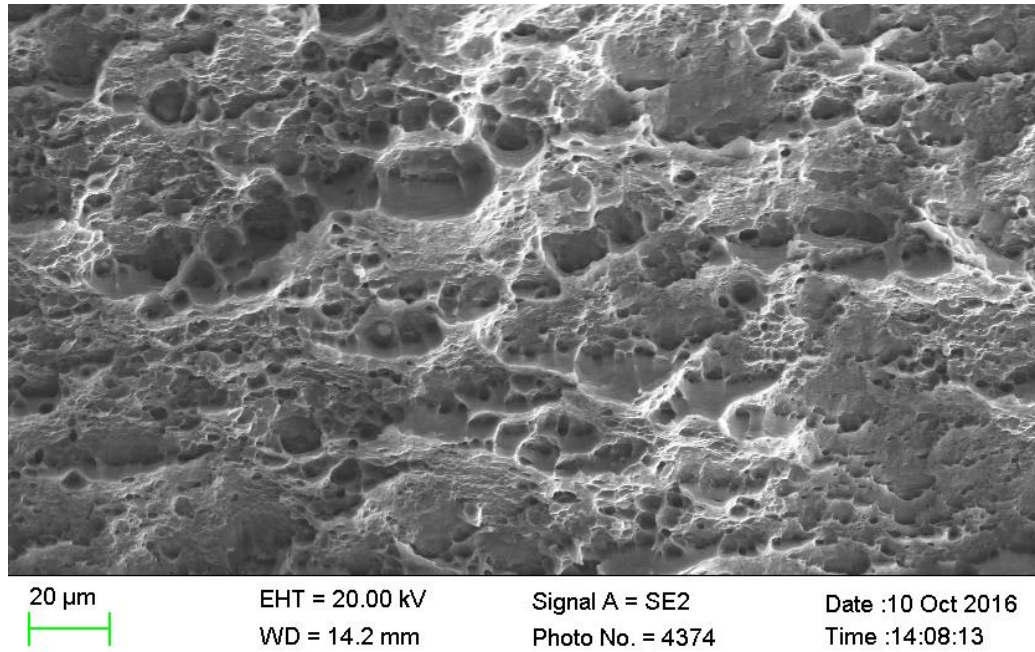


**Figure 18:** Showing the ductile fracture surface of an Al-Li 8090 tensile test piece, solution heat treated (530°C for 30 mins), quenched and cooled in liquid nitrogen (0 mins aging time). (Cusack, 2014)<sup>66</sup>.

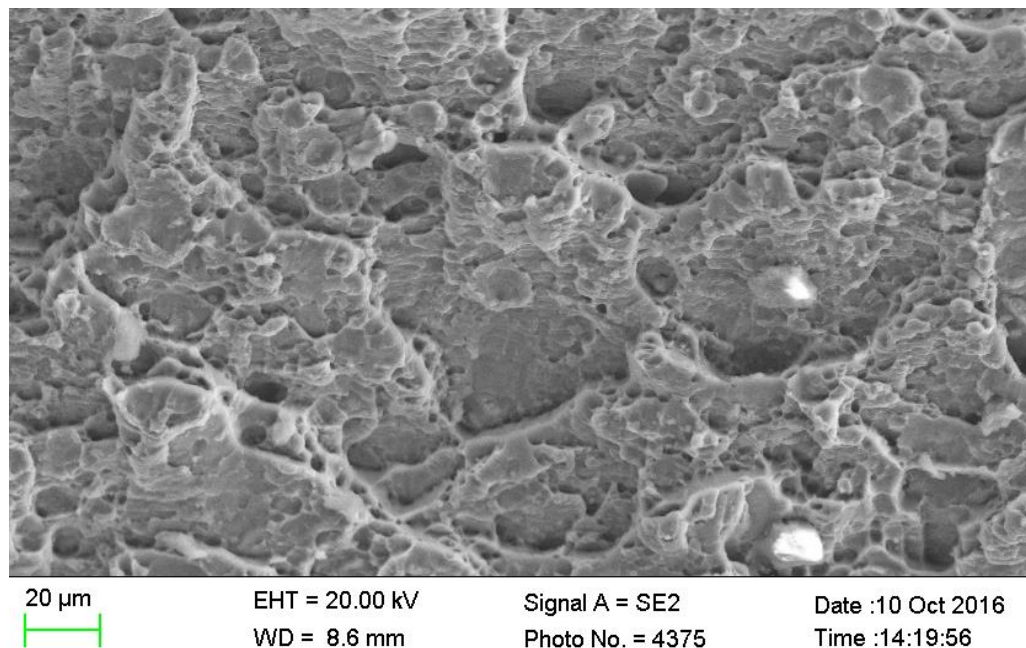


**Figure 19:** Showing the fracture surface of an Al-Li 8090 tensile test piece, solution heat treated (530°C for 20 mins), quenched and cooled in liquid nitrogen (+5 mins aging time). (Cusack, 2014)<sup>66</sup>.

### 2.3.2 Planar slip of Al-Li alloys (due to precipitate shearing).

As has been described previously for Al-Li alloy systems, it is the  $\delta'$  precipitates which are considered to be the primary strengthening mechanism. This strengthening is achieved by precipitation hardening and occurs due to the impedance of dislocation movement. This mechanism is dependent on the interaction between the dislocations and precipitates. Depending on the size, spacing and coherency of the precipitates, the interaction between moving dislocations and precipitates results in; shearing, looping or bypassing (Totten and Mackenzie, 2003)<sup>73</sup>. Ordered  $\delta'$  precipitates are coherent with the matrix, with a slight misfit strain and it is this elastic coherency strain field (Witt and Gerold, 1969)<sup>74</sup> which determines the subsequent interaction between the precipitate particle and moving dislocations. This includes the Critical Resolved Shear Stress (CRSS) (i.e. the resistance to the shearing), which itself is determined by a minimum particle diameter (Figure 20).

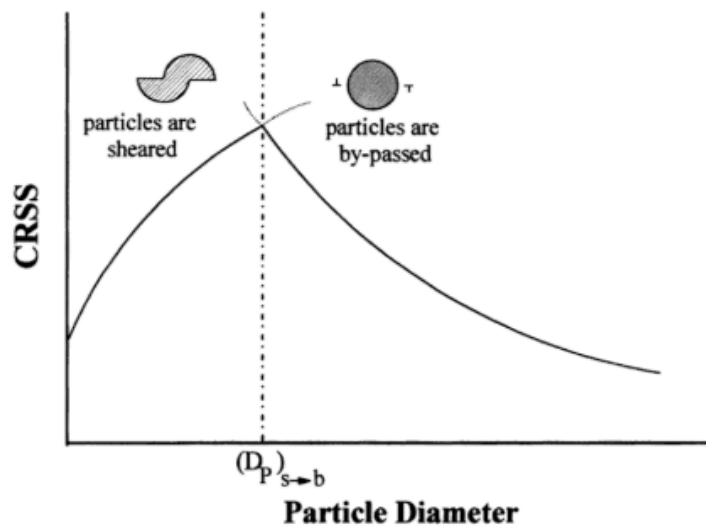


**Figure 20:** Illustrating the critical particle diameter for Orowan looping (Totten and Mackenzie, 2003)<sup>75</sup>  $(D_p)_{s \rightarrow b}$  = particle diameter for the change from shearing to by-passing.



As also previously mentioned, the average diameter of the spherical  $\delta'$  precipitates has been reported by a number of authors including (Noble et al. (post 1996)<sup>28</sup> , Edgar and Starke (1988)<sup>76</sup>) to be less than the critical 50 nm diameter to enable the occurrence of Orowan looping (or Orowan Strengthening), Edgar et al. (1988)<sup>3</sup>. Once the crucial particle diameter size has been met, if there is sufficient space between the precipitates, then the dislocations can bypass the precipitates entirely, with no strengthening occurring. Therefore, the greatest strengthening combination for an alloy is a combination of precipitates that are large enough to resist dislocation shearing and are sufficiently closely spaced to not be bypassed ( $\sim 10$  nm), (Totten and Mackenzie, 2003)<sup>73</sup>.

If shearing occurs in an ordered  $L1_2$  FCC crystal, such as in the case of  $\delta'$ , the following occurs; the passage of the first dislocation through the precipitate results in the formation of an Anti-Phase Boundary (APB) on the slip plane of the precipitate, Figure 21. Gerold and Karnthaler (1988)<sup>77</sup> report that this shearing mechanism occurs with pairs of dislocations, referred to as super dislocations by Thompson and Noble (1971)<sup>78</sup>. The passage of the first dislocation reduces the net cross-sectional area of the sheared precipitate by a value proportional to the Burgers vector (Reed-Hill and Abbaschian, 1991)<sup>79</sup>. Passing of the second dislocation enables the order of the particle to be re-established. Cutting and subsequent restoration of the precipitate's order shears the precipitate with no strengthening effect. This effect reduces the diameter of the precipitate and enables further movement of dislocations

allowing for further shearing to occur, Figure 22. This ultimately results in pile-ups of dislocations at grain boundaries which causes strain localisation.

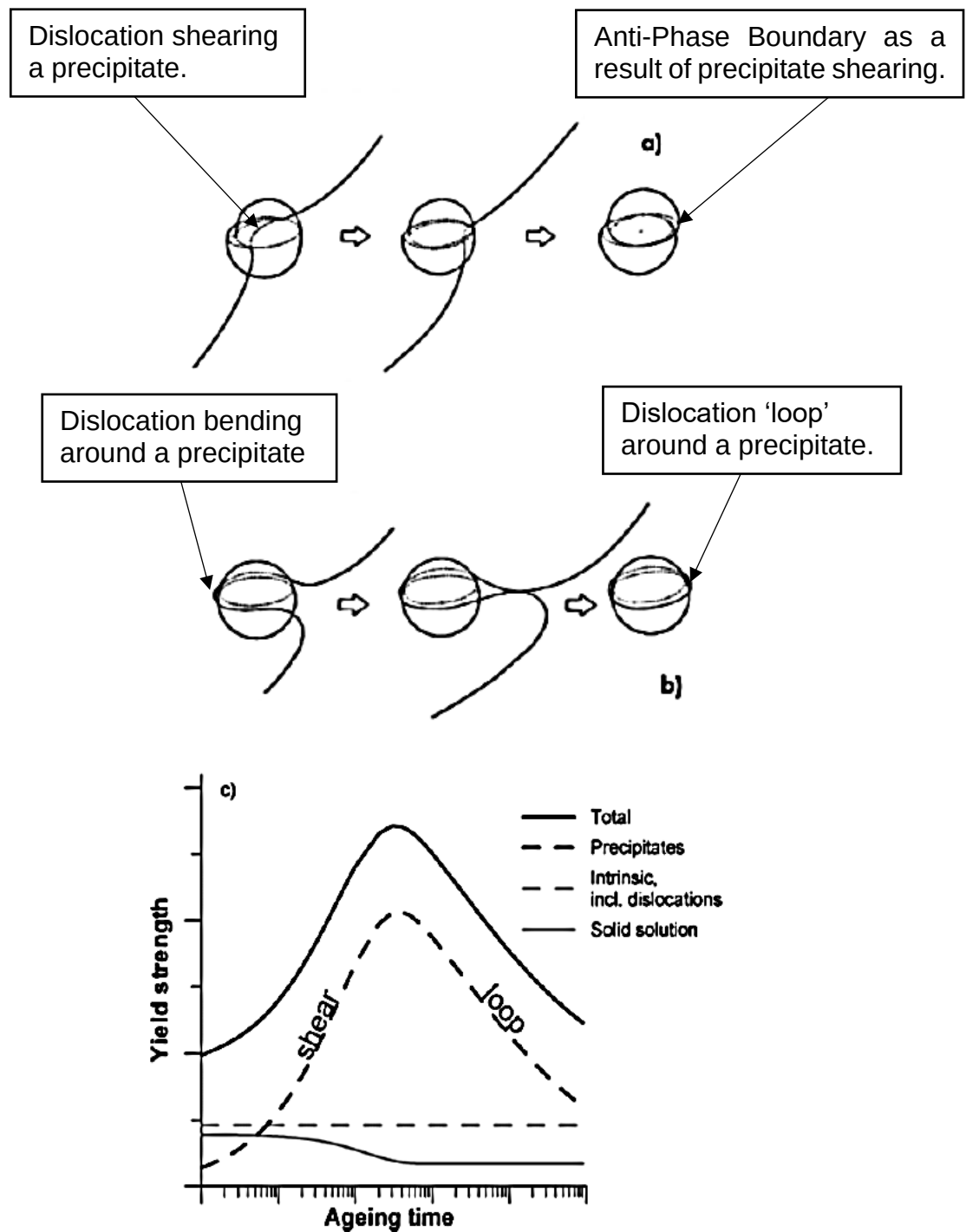


**Figure 21:** Illustration showing a) precipitate shearing and a resultant Anti-Phase Boundary, b) dislocation looping and c) effect of shearing and looping on yield strength (Mohamed and Fawzy, 2012)<sup>80</sup>.

Many researchers (including Gregson and Flower (1984)<sup>33</sup> have proposed that it is strain localisation at grain boundaries in Al-Li alloys resulting in high stress concentrations that initiate brittle intergranular failure. This occurs as a result of pile ups of dislocations at grain boundaries and high stress grain boundary triple points (or triple junctions). This exacerbates localised stress concentrations, which in the case of Al-Li alloys also contain PFZs at the grain boundaries, resulting in subsequent intergranular failure.

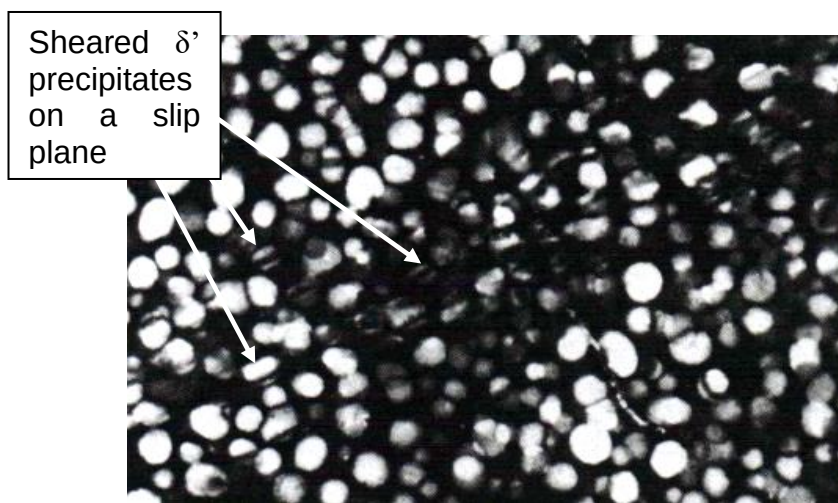


**Figure 22:** Showing a TEM dark field image of  $\delta'$  precipitates having been sheared by dislocations whilst the surrounding precipitates remain intact. (Pitcher, 1993)<sup>81</sup>.

### 2.3.3 The effect of lithium segregation on the brittle intergranular fracture of Al-Li alloys.

Lynch (1991)<sup>82</sup> concluded that the brittle intergranular fracture of Al-Li 8090 was due to segregation of Li to the grain boundaries during the single aging heat treatment. His conclusions resulted from investigations into the effect of single (170°C for 32 hours) and double re-aging heat treatments (170°C for 32 hours + between 180-230°C for 5 minutes) on the fracture toughness of 8090

T8771 plate. Li segregation was postulated as the reason for low toughness behaviour identified in the Al-Li 8090 T8771 material. Furthermore, dual age heat treatments (with 230°C for 5 mins appearing to triple the toughness results) reportedly decreased the extent of the lithium segregation, therefore improving the toughness behaviour. However, 're-aging' of the dual aged material between 60-160°C, caused the embrittlement to reoccur.

Lynch (1991)<sup>82</sup> determined that the toughness vs. the square root of time at a particular temperature gave approximately linear results (Figure 23) indicating that it was a diffusion controlled process.

Whilst the effect of re-aging Al-Li alloys can be shown with empirical data, Li segregation at the grain boundaries is very difficult to observe (Pasang et al., 2012)<sup>67</sup>. No directly attributable evidence in support of this proposed mechanism has been reported.

#### **2.3.4 Reversion age heat treatments to improve the toughness of Al-Li alloys.**

Work by the Defence Research Agency into the reported loss of fracture toughness properties in Al-Li 8090 by McDarmaid in (1992)<sup>83</sup> and (1993)<sup>84</sup> resulted in the proposition of recovering lost toughness by implementing a reversion aging (RV) heat treatment.

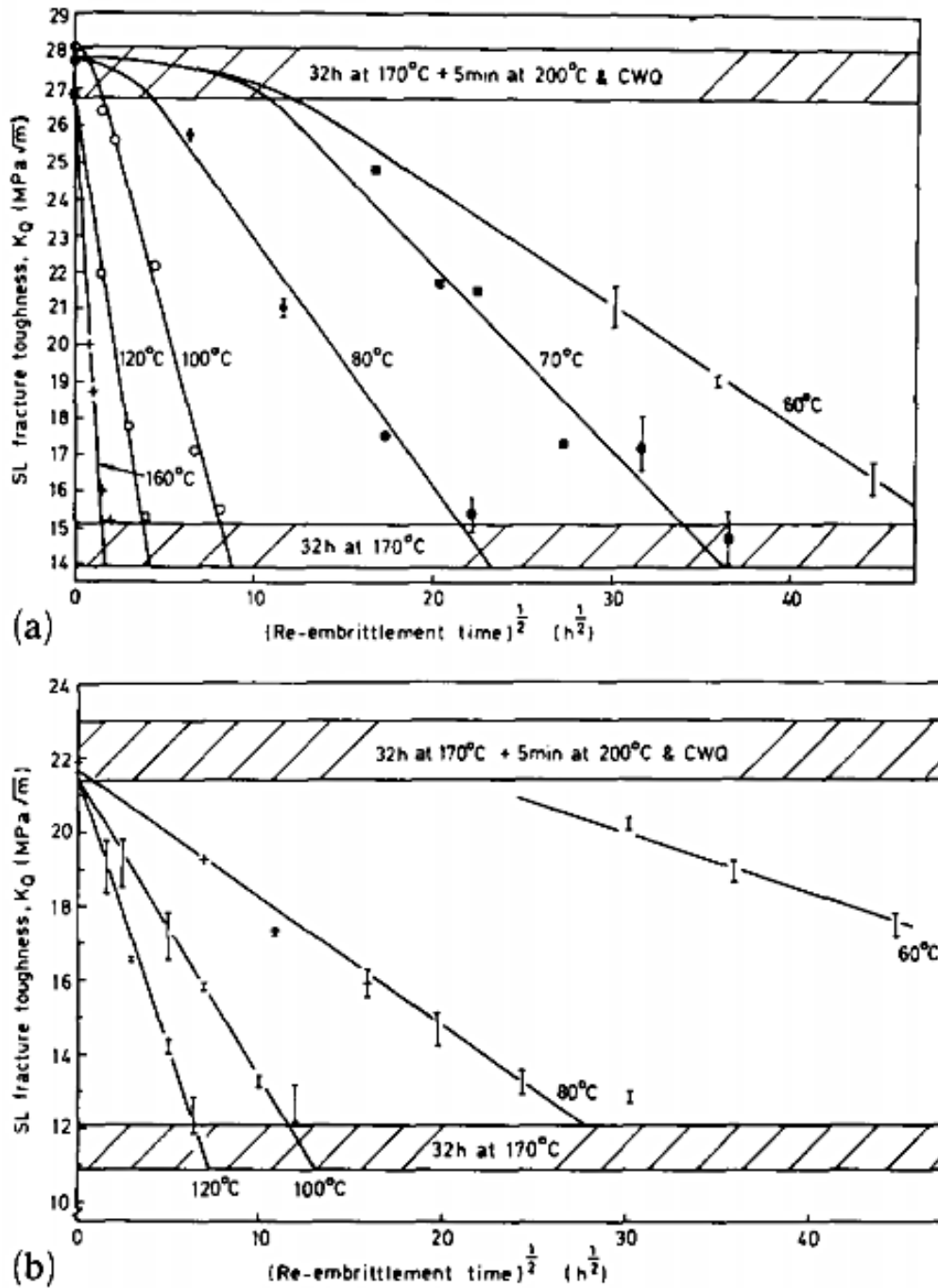


Figure 23: Showing the S-L fracture toughness of Al-Li 8090 plate vs. the square route of re-embrittlement time for 60-160°C for dual aged (170°C for 32 hours + 200°C for 5 mins). a) 25 mm and b) 45 mm thick plate, respectively.

This involved re-solution heat treating, stretching and artificially aging 8090 T81 material to a T62 condition<sup>xiii</sup>, followed by a short additional RV heat treatment of 190°C for 10 minutes. The justification for this work is based upon a measured increase in ductility with an accompanying reduction in strength following the T62 heat treatment. The reported mechanical properties for the alloys investigated are given in Table 5. As can be seen in Table 5, The RV heat treatment gives a further increase in ductility but an additional loss of strength. In the same paper, McDarmaid implies that a (T62+RV) heat treatment could be used to assist in regaining lost fracture toughness properties of 8090 T81 material. Following a 4-week exposure period at 70°C, tensile testing revealed that the T81 and T81+RV materials suffered a 15% reduction in fracture toughness properties, whereas the T62+RV aging material experienced no loss of fracture toughness or strength (McDarmaid, 1993)<sup>84</sup>.

### **2.3.5 Reversion age heat treatments and planar slip.**

However, these 'reclaimed' improvements in fracture toughness and ductility were not found to be a permanent feature. Pitcher (1993)<sup>81</sup> noted that RV heat treated Al-Li 8090 T81 sheet would 'decay' back to its original mechanical properties within several weeks at 70°C and within several years at 20-30°C. Pitcher therefore theorised as to why the post-aging heat treatments (including reversion aging) resulted in a regaining of fracture toughness properties.

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<sup>xiii</sup> Although McDarmaid classifies the heat treatment as the 'T62' condition, a solution heat treat, stretch and artificial age would technically qualify as the T651 condition.

**Table 5: Showing the reported mechanical properties for T81 material subjected to a RV heat treatment, McDarmaid (1992)<sup>83</sup> and (1993)<sup>84</sup>.**

| Specification/Heat Treatment                            | UTS (MPa) | 0.2% (MPa) | Elong (%)           |
|---------------------------------------------------------|-----------|------------|---------------------|
| 8090 T81 Spec (For comparison)                          | 440       | 340        | 12.0                |
| 8090 T81 DRA (Starting material)                        | 432       | 350        | 10.6 <sup>xiv</sup> |
| T62 (30 min at 530°C, 2% stretch and 24 hours at 150°C) | 422       | 322        | 11.5                |
| T62+RV (10 min @ 190°C RV heat treatment)               | 394       | 296        | 12.6                |
| Percentage Change (against DRA 8090 T81)                |           |            |                     |
| T62 (30 min at 530°C, 2% stretch and 24 hours at 150°C) | ↓2.4%     | ↓8.0%      | ↑8.5%               |
| T62+RV (10 min @ 190°C RV heat treatment)               | ↓8.9%     | ↓15.4%     | ↑14.2%              |

Drawing upon earlier work with Stewart and Gupta (1991)<sup>58</sup> using Small Angle Neutron Scattering (SANS), the workers measured the change in volume fraction of  $\delta'$  as a result of a number of dual heat treatments, noting that a reversion heat treatment (such as 190°C for 5 minutes) was sufficient to remove a fine distribution of  $\delta'$  precipitates that were typically 3-4 nm in diameter. Pitcher Stewart and Gupta (1991)<sup>58</sup> surmised that given the propensity for planar slip within the  $\delta'$  precipitates, removal of very fine  $\delta'$

<sup>xiv</sup> Due to missing information in McDarmaid (1993)<sup>74</sup>, reference ambient elongation data has been taken from McDarmaid (1995)<sup>61</sup>.

precipitates would be likely to reduce the overall volume fraction of  $\delta'$ , but this would increase the average  $\delta'$  precipitate diameter throughout the material. This would therefore lead to an increased resistance to planar slip and was the likely reason for the reported recovery in toughness properties.

#### **2.4 Incorporation of Al-Li 8090 into the EH101 development programme.**

As previously mentioned in Section 1.2, research and literature into the Al-Li (relevant to this project) culminated in the 1980's with a large amount of work undertaken at the Defence Research Agency (DRA) Farnborough. Peel, Evans and McDarmaid (1985)<sup>85</sup> reported on the current state of development of Al-Li alloys, resulting in three proposed alloy variants, designated Lital A, B and C. Lital A was designed for medium strength applications (including forgings) and it was intended to be a substitute for conventional 2014 Al alloy in the precipitation hardened (T6) temper.

Around the same time, the EH101 was entering into its development phase. It was reported by Smith (1988)<sup>13</sup> that two 'design philosophies' were considered for the material selection for the EH101 and the proposed advantages of Al-Li alloys met the second philosophy of:

*"...lighter materials which provided equivalent strength and toughness as conventional denser material".*



Even at the early stages of this development period, it is clear that the reported mechanical properties for 8090 and 8091 - the two Al-Li alloys being considered for the main lift frames of the EH101 fuselage – did not always meet minimum specification criteria. The primary intention was always to replace conventional material with Al-Li alloys and in terms of main lift frames, AA 7010 was the main competitor. Therefore, a system of comparative mechanical tests between 8090, 8091 and 7010 were undertaken. Smith (1998)<sup>13</sup> produced results of the mechanical tests, which can be seen in Figure 24. Smith states that whilst the 8090 and 8091 results are relatively inferior to AA 7010:

*“....they nevertheless generally achieve the minimum allowable EH101 main lift frame requirements (S-values) as determined by WHL (Westland Helicopter Laboratory) stress office for longitudinal, long transverse and short transverse directions”.*

Furthermore, it was anticipated at the time that additional quantities of Mg would increase the 0.2% proof stress and elongation properties of the Al-Li samples by ~10%. Preliminary work undertaken by Gregson, Peel and Evans (1986)<sup>86</sup> and Dinsdale, Harris and Noble (1981)<sup>87</sup> did support this hypothesis. However, a report by the Quality Engineering and Test Establishment (QETE) noted that no actual experimentation to prove this statement had been undertaken (Abdel-latif, Beaudet and Roth, 1988)<sup>14</sup>.

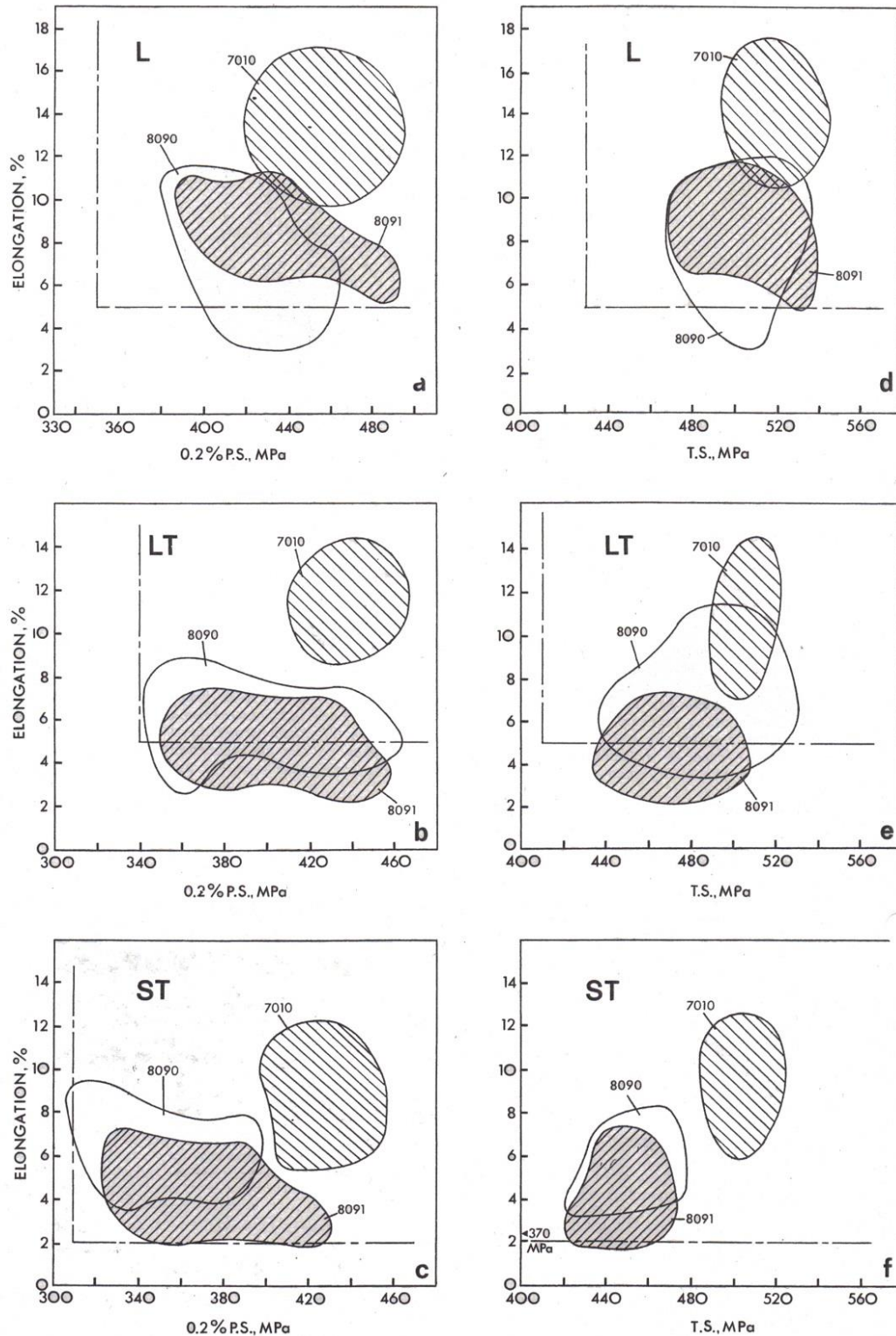


**Figure 24:** Showing the comparison of mechanical properties of Al-Li 8090, Al-Li 8091 and conventional 7010 for the EH/EW101 forgings for use in the main lift frames. Inset axes represent minimum specification criteria. (Smith, 1988)<sup>13</sup>. (L - longitudinal, LT - long transverse and ST - short transverse).

## **2.5 Summary of literature review of Al-Li alloys and the role of Al-Li 8090 as a main lift frame material.**

In summary, Al-Li alloys are exceptionally complex materials that are still not understood in their entirety. For example, precursors to the formation of  $\delta'$  have been predicted, but have yet to be observed (Prasad, Gokhale and Wanhill, 2014)<sup>21</sup>. Whilst the potential increase in stiffness and reduction in density make Al-Li 8090 (and by extension the entire 2<sup>nd</sup> generation of Al-Li alloys) a very attractive material for the aerospace industry. However, the propensity for planar slip to occur as a result of shearing of  $\delta'$  precipitates by the movement of dislocations results in a brittle failure mechanism. Although the mechanical performance is improved by the formation of S precipitates, brittle intergranular failure is still the primary overload failure mechanism.

Additionally, the combination of a dual heat treatment (525-535°C solution heat treatment, 2-5% cold work and 170-180°C 20-50 hours artificial age) provides a partially recrystallised and anisotropic microstructure. Combined with the low performance in ductility, this adds further complexity to the alloy system when compared against conventional aluminium alloys.

Considering these factors, the suitability of using Al-Li 8090 in the manufacture of EH101 main lift frames has been called into question previously (Abdel-Latif, et al. 1988)<sup>14</sup> and is one of the likely reasons for publication by the OEM

(Smith<sup>xv</sup>, 1988)<sup>13</sup>, with predicted improvements in ductility resulting from a proposed increase in magnesium.

With the recent UK MoD theatres of operation, amalgamating the aforementioned factors with of the thermal instability of Al-Li alloys (Nobel, Harris and Dinsdale (1996)<sup>27</sup>, McDarmaid and Starr (1990)<sup>59</sup>, McDarmaid, (1995)<sup>60,61</sup>, Zakharov (2003)<sup>62</sup> and (1999)<sup>64</sup> raises significant questions on the microstructure and mechanical property performance of the Al-Li 8090 main lift frame material in its present day condition.

It should be noted that this work does not intend to attempt to uncover all of the mysteries surrounding Al-Li alloys. Conversely it aims to focus on a small field of interest, whilst taking into account, the complex nature of this alloy system which may impact on the work.

The following section will baseline the as-received Al-Li 8090 main lift frame material against the data provided in the literature to determine how comparable the present day Al-Li 8090 main lift frames are with the original design specification.

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<sup>xv</sup> *It should be noted that A Smith worked for Westland Helicopters at time of publication.*

### **3 Experimental Procedure.**

#### **3.1 Obtaining representative (in-service) main lift frame material.**

Historically, the only available source of Merlin helicopter main lift frame material that had been subjected to in-service loading is limited to the wreckage of crashed aircraft. The issue with this material was that in all instances, the main lift frames had either been subjected to significant impact loads (from crashing) or to aggressive chemical environments (such as the sea bed of the North Atlantic Ocean). For this project, a number of Merlin torsion boxes were obtained with permission from AgustaWestlands that had been removed from in-service aircraft for an investigation into suspected Stress Corrosion Cracking (SCC) in the torsion box section of the structure (blue area, Figure 25).

In total 8 torsion box sections were made available, resulting in 16 'hockey sticks' for the purposes of this project. The suspected SCC mechanism was considered to have no bearing on the potential findings of this project as it is only the yellow lift frame section that is of interest, not the blue box section (Figure 25) as this is not a part of the main lift frame structure - its purpose is designed around the rear undercarriage, and all of the box sections were cut from the main lift frames and were used for other work.

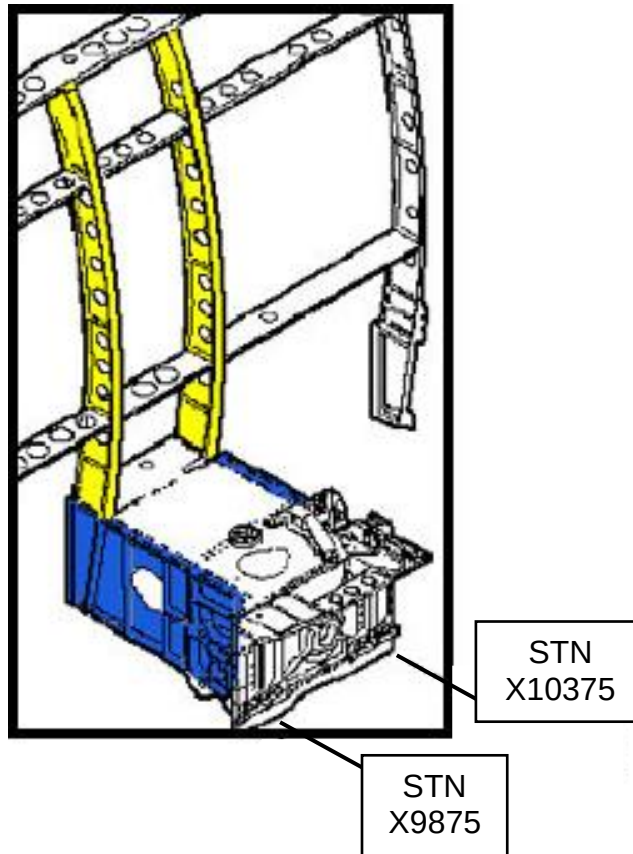


**Figure 25: Showing an illustration of the starboard torsion box structure (expanded image from Figure 3).**

The torsion boxes were delivered in the assembled condition. They were disassembled by the 1710 NAS Repair Department – which meant that qualified military aircraft engineers deconstructed the assembly, ensuring any erroneous stress concentrations and damage to the structure was minimal. As has been previously mentioned – primary aircraft structure is not designed to be replaced and therefore is often not serialised or tracked through a configuration control system. This resulted in difficulties when attempting to identify the parent aircraft and proposed total number of flying hours the frames may have been subjected to. Visual examination of the main lift frames revealed limited information which did enable cross identification against four

aircraft tail numbers, which for the purposes of the OFFICIAL designation of this document were designated Aircraft A, B, C and D respectively. This enabled flying hour data to be extracted up until the date of removal of the lift frames (which were all removed in November 2012).

**Table 6: Showing the documented flying hours for Aircraft A, B, C and D up until November 2012.**

| Aircraft | Fleet | Flying Hours<br>(6 day no fly<br>period) | Flying Hours<br>(as of 21 Nov<br>12) | First Flight | Date<br>delivered<br>to Unit |
|----------|-------|------------------------------------------|--------------------------------------|--------------|------------------------------|
| A        | RN    | Not Known                                | 2065:52                              | 30-Mar-01    | Not Known                    |
| B        | RN    | Not Known                                | 2109:04                              | 15-May-02    | Not Known                    |
| C        | RAF   | 2316:19                                  | 2563:51                              | Not Known    | Not Known                    |
| D        | RAF   | Not Known                                | 2385:48                              | Not Known    | 22-Mar-02                    |

As can be seen in Table 6, each aircraft had achieved in excess of 2000 flying hours and at least three had been flying in excess of 10 years. In addition, the aircraft were a selection of both the RN Mk1 and RAF Mk3 versions and therefore are representative of both fleets and the different operational tasks they perform<sup>xvi</sup>. However, matching the hockey sticks to their parent aircraft was a greater challenge due to the already mentioned absence of serial numbers. This resulted in only six being positively identified against their parent aircraft and associated tail numbers. Each lift frame was labelled with a unique designation number related to its parent torsion box, station number and orientation to the aircraft (port or starboard), Table 7.

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<sup>xvi</sup> *The Merlin Mk2 and Mk4 variants were not in service at this time.*

**Table 7: Showing the identification details for each main lift frame.**

| Frame | Aircraft Designation | Fleet | Position  | Station No. | Information                                             |
|-------|----------------------|-------|-----------|-------------|---------------------------------------------------------|
| A1    | N/A                  | RN    | Port      | X9875       | Sectioned for tensile testing                           |
| A2    | N/A                  | RN    | Port      | X10375      | Tensile testing 53.1°C 1000hr exposure (Groups A and B) |
| B1    | N/A                  | RN    | Starboard | X9875       | Sectioned for tensile testing                           |
| B2    | N/A                  | RN    | Starboard | X10375      | Tensile testing 80.0°C 1000hr exposure <sup>xvii</sup>  |
| C1    | N/A                  | RAF   | Port      | X9875       | 1710 NAS Archive                                        |
| C2    | N/A                  | RAF   | Port      | X10375      | Tensile testing 53.1°C 1500hr exposure (Groups C and D) |
| D1    | Aircraft C           | RAF   | Port      | X9875       | Sectioned for tensile testing                           |
| D2    | Aircraft C           | RAF   | Port      | X10375      | Sectioned for tensile testing                           |
| E1    | Aircraft D           | RAF   | Port      | X9875       | Tensile testing multiple Groups                         |
| E2    | Aircraft D           | RAF   | Port      | X10375      | Ballistic trial No.2                                    |
| F1    | Aircraft D           | RAF   | Starboard | X9875       | Ballistic trial No.3                                    |
| F2    | Aircraft D           | RAF   | Starboard | X10375      | Tensile testing Group E                                 |
| G1    | N/A                  | RN    | Starboard | X9875       | Tensile testing Group F                                 |
| G2    | N/A                  | RN    | Starboard | X10375      | 1710 NAS archive (PMS)                                  |
| H1    | N/A                  | RN    | Port      | X9875       | Ballistic trial No.1                                    |
| H2    | N/A                  | RN    | Port      | X10375      | Tensile testing at ambient temperature                  |

<sup>xvii</sup> Two test pieces from this frame (No 21 and 26 were subjected to ductile failure testing at 530°C for comparison with work reported by Lynch, Muddle and Pasang (2001)<sup>68</sup> (see Section 2.3.1).



Once designated, the lift frames were stripped of their paint layers by plastic media stripping at Vector Aerospace Fleetlands<sup>xviii</sup>. This process culminated in access to a unique sample of the lift frame material, which was representative of operational Merlin aircraft across the UK MoD Services.

### **3.1.1 Structural Integrity and Structurally Significant Items - why primary aircraft structure is not often investigated.**

The Military Aviation Authority (MAA) publishes guidance on a number of technical areas that are deemed essential to ensuring the Airworthiness of aircraft Propulsion, Systems and Structure. This ensures that the aircraft structure is in a suitable condition to be considered operational. It is dominated by the principles of Air Safety and Aircraft Structural Integrity (SI). The MAA have published<sup>xix</sup> a Structural Integrity Handbook which details a series of ‘threats’ to SI:<sup>88</sup>

- A. Overload.
- B. Fatigue, fretting and wear.
- C. Accidental Damage (AD) and Environmental Damage (ED).
- D. Procedural (Design, Manufacturing, Maintenance or Supply) error.

These ‘threats’ are generally managed at a Squadron level (at airstations or equivalent) using conventional engineering and husbandry techniques, with use of more detailed scientific and technical expertise as, and when, required.

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<sup>xviii</sup> Now officially known as StandardAero.

<sup>xix</sup> This freely accessible version is uncontrolled and may not be up to date when viewed.

In addition, aging aircraft are regularly assessed (by what is classed as a structural sampling process) - however this does not typically involve the core skeleton structure of an aircraft (as per the lift frames in this investigation). Furthermore, metallurgical assessment is often limited to information ascertained from failure investigations which are usually focussed on identified or known 'threats' such as those listed above. They are not aimed at assessing the overall condition of the core in-service material microstructure.

Operational service has a very real potential to alter the structure of a material (especially in the more complex materials) in ways that may not become apparent during the early to middle life of an aircraft, but could potentially have significant effects on the late stages of operations – especially with extensions to Out of Service Dates. To address these potential late failures (although primarily aimed at the cyclic loading that affects aircraft), all aircraft Structural Significant Items (SSIs) are designed around a philosophy of 'Damage Tolerance' or 'Safe Life'. These two philosophies are based on either:

- 1) Calculated critical cracks lengths, using fracture mechanics and non-destructive inspection (NDT). This is in order to remove components from service under risk of imminent failure.
- 2) Calculated S-N curves for the number of cycles to failure for a loading spectrum (though fatigue testing) and the application of a 'safe life' for components. Upon completion of this 'life' which is typically given in flying hours, they are removed from service.

SSIs are defined as:

*“Any detail, element or assembly, which contributes significantly to carrying flight, ground, pressure or control loads and whose failure could affect the structural integrity necessary for the continued safe and controlled flight of the aircraft. (MAA02 Issue 5, see also AP 100C-22, MSG-3 and MASAAG 103B)”*<sup>89</sup>

In the majority of aircraft platforms, SSIs are considered to be important but removable/replaceable components on aircraft. Justifiably, undamaged primary aircraft structure (i.e. not having been subjected to a repair or modification) is not subjected to metallurgical sampling processes and is not scrutinised for any change from the original design. However, the core principles of Safe Life and Damage Tolerant philosophies are inexorably tied to the microstructure and therefore mechanical performance of the main lift frame and other primary structure of any aircraft platform. Therefore, any alteration to the mechanical properties of a main lift frame material will significantly impact on any pre-existing justification for continued airworthiness (such as mechanical property based calculations).

The counter-argument to physical sampling of main lift frames is that the removal of material would render the main lift frame useless, making the aircraft unserviceable and subsequently impact operational capability. It is simply not feasible (or economical) to sample primary aircraft structure. In this instance, being able to use material that is representative of in-service aircraft

and which has not had consequential impact on operational capabilities, really has provided a unique opportunity to gain a detailed insight into the condition of the main lift frame material. However, before any data could be obtained for the in-service material, comparison against the original design specification was required to determine whether there had been any fundamental shift in properties since the original design conception and the original safety case justifications.

### **3.2 Material characterisation of the as-received main lift frame material.**

As stated previously, given the length of time between the design and implementation of Al-Li 8090 in the EH101 programme, this project provided an opportunity to establish whether the current in-service Al-Li 8090 forgings were comparable against the original research and design data. The initial task was to determine which properties would be suitable to compare the in-service material against the original specification. As with material specifications and data sheets, the primary features of interest are often the elemental chemical composition and mechanical properties. The research plan for the experimental procedure is shown in Figure 26

### **3.3 Determining the elemental chemical composition of the as-received main lift frame material.**

As has already been discussed, Al-Li 8090 is a quinary alloy composing of Al, Li, Cu, Mg and Zr. The specific composition of which has been slightly adjusted over time, but retains the same core %weight compositions, see Table 8

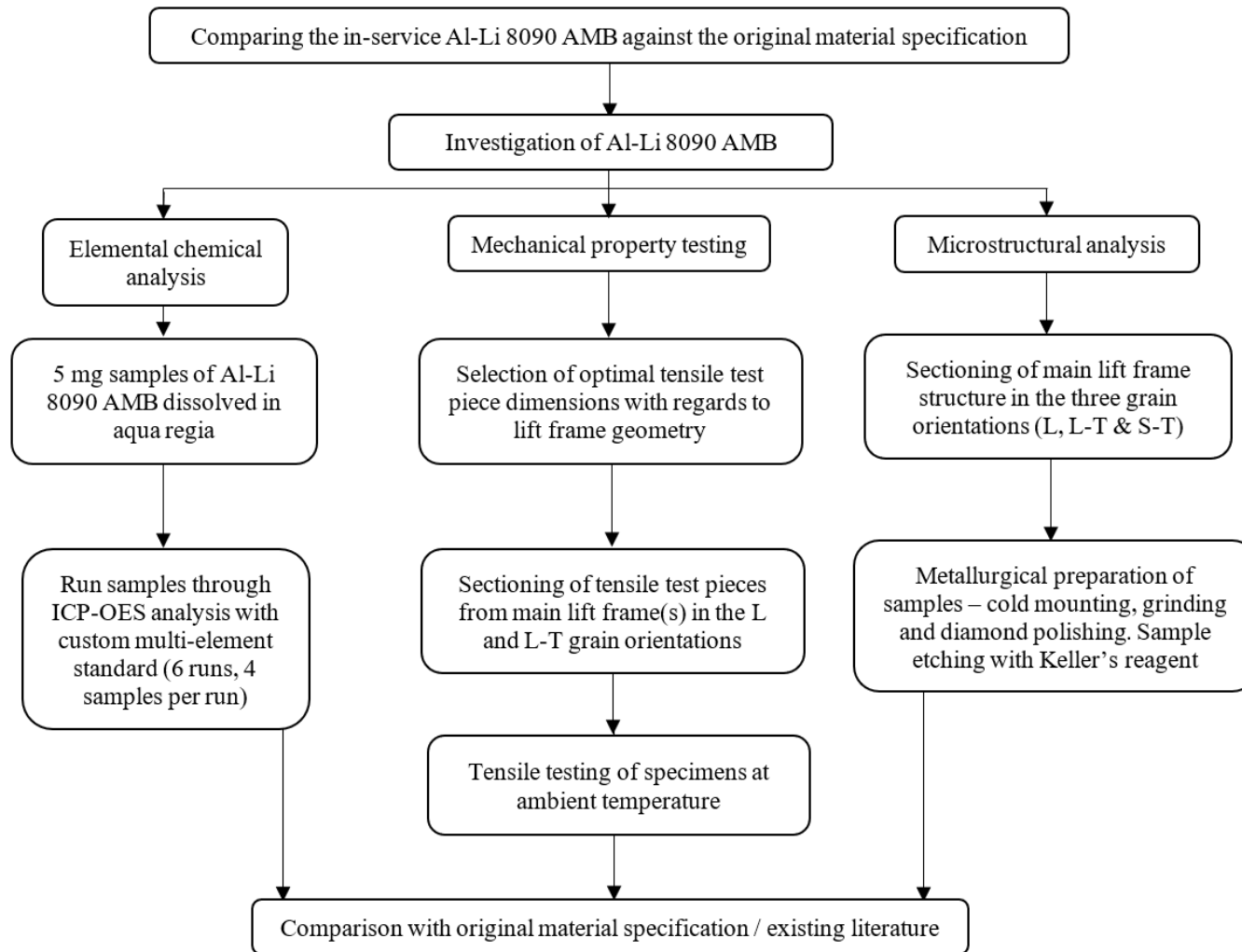


**Figure 26: Showing the research plan for the experimental procedure.**

**Table 8: Showing the specified elemental chemical composition in wt.% of Al-Li 8090 from a number of sources.**

|                                                  | Li      | Cu      | Mg   | Zr        | Al   |
|--------------------------------------------------|---------|---------|------|-----------|------|
| Rioja and Liu <sup>xx</sup> (2012) <sup>10</sup> | 2.4     | 1.2     | 0.8  | 0.11      | Bal. |
| ASM Handbook Vol 2 (1992) <sup>90</sup>          | 2.45    | 1.30    | N/A  | 0.12      | Bal. |
| 8090 (May 1985) <sup>91</sup>                    | 2.2-2.7 | 1.0-1.6 | 0.30 | 0.04-0.16 | Bal. |
| 8090A (Late 1985) <sup>92</sup>                  | 2.1-2.7 | 1.1-1.6 | 0.15 | 0.08-0.15 | Bal. |
| Prasad, Gokhale and Wanhill (2013) <sup>93</sup> | 2.4     | 1.2     | 0.8  | 0.11      | Bal. |

### 3.3.1 Methodology for determining the elemental chemical composition of the as-received material and results.

For determination of the elemental chemical composition of the as-received in-service material, the suite of analytical equipment available at 1710 NAS was considered. Scanning Electron Microscopy with Energy Dispersive X-ray (SEM-EDX) analysis was not considered to be a suitable technique due to the inability of the detection system to give accurate, quantifiable measurements of the lighter elements (notably Li and Mg in this case). In addition, SEM-EDX is considered to be a semi-quantitative technique and the degree of accuracy of the results was not deemed to be sufficient for this project.

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<sup>xx</sup> Rioja and Liu (2012)<sup>10</sup> list 8090 as containing 0.17% Scandium, however later iterations of their data Prasad, Gokhale, and Wanhill, (2013)<sup>93</sup> omit the addition.

Therefore, the decision was taken to determine the elemental chemical composition of the main lift frame material by way of Inductively Coupled Plasma with Optical Emission Spectroscopy (ICP-OES). This technique is based on the ionisation<sup>xxi</sup> of atoms of a solution in an inductively coupled plasma flame, with subsequent detection and measurement by an optical emission spectrometer – with a detection sensitivity of parts per billion<sup>94</sup>.

Samples were taken from a selection of main lift frame material (each weighing approximately 0.5 g). The samples were acid digested into solution using 10% aqua regia via an in-house developed method that has been used by 1710 NAS for multiple air accident investigations. The methodology is provided in Annex B.

Six sections were taken from main lift frames and were submitted for ICP analysis with each section being split into four samples. It must be noted that one of the analytical wavelengths used for the detection of Li gave spurious readings on the equipment.

Therefore the 2<sup>nd</sup> run of each sample was repeated, which resulting in a data set for nine samples (36 individual samples in total) and the outlying result was identified as being an issue with the graphical representation software on the ICP computer, which had no direct effect on the results. The complete ICP results can be found in Annex B and a summary can be seen in Table 9.

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<sup>xxi</sup> *Atomised, ionised and thermally excited*<sup>94</sup>.

**Table 9: Showing the elemental chemical composition of Al-Li 8090 main lift frame material as determined by ICP-OES at 1710 NAS (combination of 36 sample runs) in wt.%.**

| Chemical Composition (wt.%) |      |      |      |      |
|-----------------------------|------|------|------|------|
| Batch                       | Li   | Cu   | Mg   | Zr   |
| 1                           | 1.27 | 0.86 | 0.48 | 0.07 |
| 2                           | 3.55 | 1.35 | 1.13 | 0.17 |
| 3                           | 2.58 | 1.59 | 0.88 | 0.13 |
| Average                     | 2.46 | 1.27 | 0.83 | 0.12 |

### 3.3.2 Conclusion of the findings for the elemental chemical composition.

As can be seen in Table 10, the ICP-OES results are generally consistent with the specified elemental compositions. It should be noted that the early Al-Li 8090 specifications (pre 1988), give a broader range of the elemental compositions than the later specifications, potentially reflecting the developmental or prototype nature of the alloy at that period of time.

The ICP-OES results are inconsistent with Smith's report (1998)<sup>13</sup>, which proposed that increasing the Mg content in billet material would provide superior properties in 8090 forgings. The description used in Smith's report refers to the increase in magnesium by stating;

*'...it is understood from the metal manufacturers that all future AA8090 billet will contain higher magnesium contents of at least 1 (wt.%)...'*



Smith (1998)<sup>13</sup> does not provide the elemental chemical specification requirement for AL-Li 8090 in his report. However, in the same publication as Smith's report (NATO AGARD, 1988)<sup>95</sup> the commercial Alcan (British aluminium manufacturer) Mg requirement for AL-Li 8090 is given as 0.6 wt.% (Smith<sup>xxii</sup>, Gray, Schra, et al., 1988)<sup>96</sup>.

As can be seen in the post 1988 specifications such as those provided by the ASM Handbook Committee (1992)<sup>97</sup> and Rioja and Liu (2012)<sup>10</sup> (Table 10), an increase Mg content of at least 1 wt.% is not present. Although the pre 1988 specifications listed by Roth and Bright (1977)<sup>98</sup> and Edgar Starke and Quist (1985)<sup>99</sup> do accommodate for a 1 wt.% Mg content, as has already been stated, these were prototype alloys.

Therefore, given the results obtained from the ICP-OES testing, it is concluded that the elemental chemical composition of the in-service Al-Li material is chemically consistent with the Al-Li 8090 material specification given by the ASM Committee (1992)<sup>97</sup>.

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<sup>xxii</sup> C.J.E Smith worked for RAE Farnborough. Not to be confused with A Smith who worked for WH at the same time.

**Table 10: Showing the comparison of the ICP-OES results with the original material specification in wt.%.**

|                                                     | Li      | Cu      | Mg      | Zr        | Al          |
|-----------------------------------------------------|---------|---------|---------|-----------|-------------|
| Rioja and Liu <sup>xxiii</sup> (2012) <sup>10</sup> | 2.4     | 1.2     | 0.8     | 0.11      | <i>Bal.</i> |
| ASM Handbook Vol 2 (1992) <sup>97</sup>             | 2.45    | 1.3     | 0.95    | 0.12      | <i>Bal.</i> |
| 8090 (May 1985) <sup>98</sup>                       | 2.2-2.7 | 1.0-1.6 | 0.6-1.3 | 0.04-0.16 | <i>Bal.</i> |
| 8090A (Late 1985) <sup>99</sup>                     | 2.1-2.7 | 1.1-1.6 | 0.8-1.4 | 0.08-0.15 | <i>Bal.</i> |
| Alcan Commercial 8090 (1988) <sup>96</sup>          | 2.4     | 1.2     | 0.6     | 0.11      | <i>Bal.</i> |
| Prasad, Gokhale and Wanhill (2013) <sup>100</sup>   | 2.4     | 1.2     | 0.8     | 0.11      | <i>Bal.</i> |
| ICP-OES Results                                     | 2.46    | 1.27    | 0.83    | 0.12      | <i>Bal.</i> |

### 3.4 Examination of the grain orientation in the as-received main lift frame material.

The definition of grain orientations for this research project have been aligned with the definition presented by Polmear<sup>101</sup> and are illustrated in Figure 27, below. Microstructural examination of sections taken from the main lift frames revealed a typical Al-Li forged structure, with an elongated grain structure in the longitudinal orientation, interspersed with areas of recrystallised grains as can be seen in Figure 28a.

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<sup>xxiii</sup> Rioja and Liu (2012)<sup>10</sup> list Al-Li 8090 as containing 0.17% Scandium, however later iterations of their data Prasad, Gokhale, and Wanhill, R.J (2013)<sup>93</sup> omit the addition.

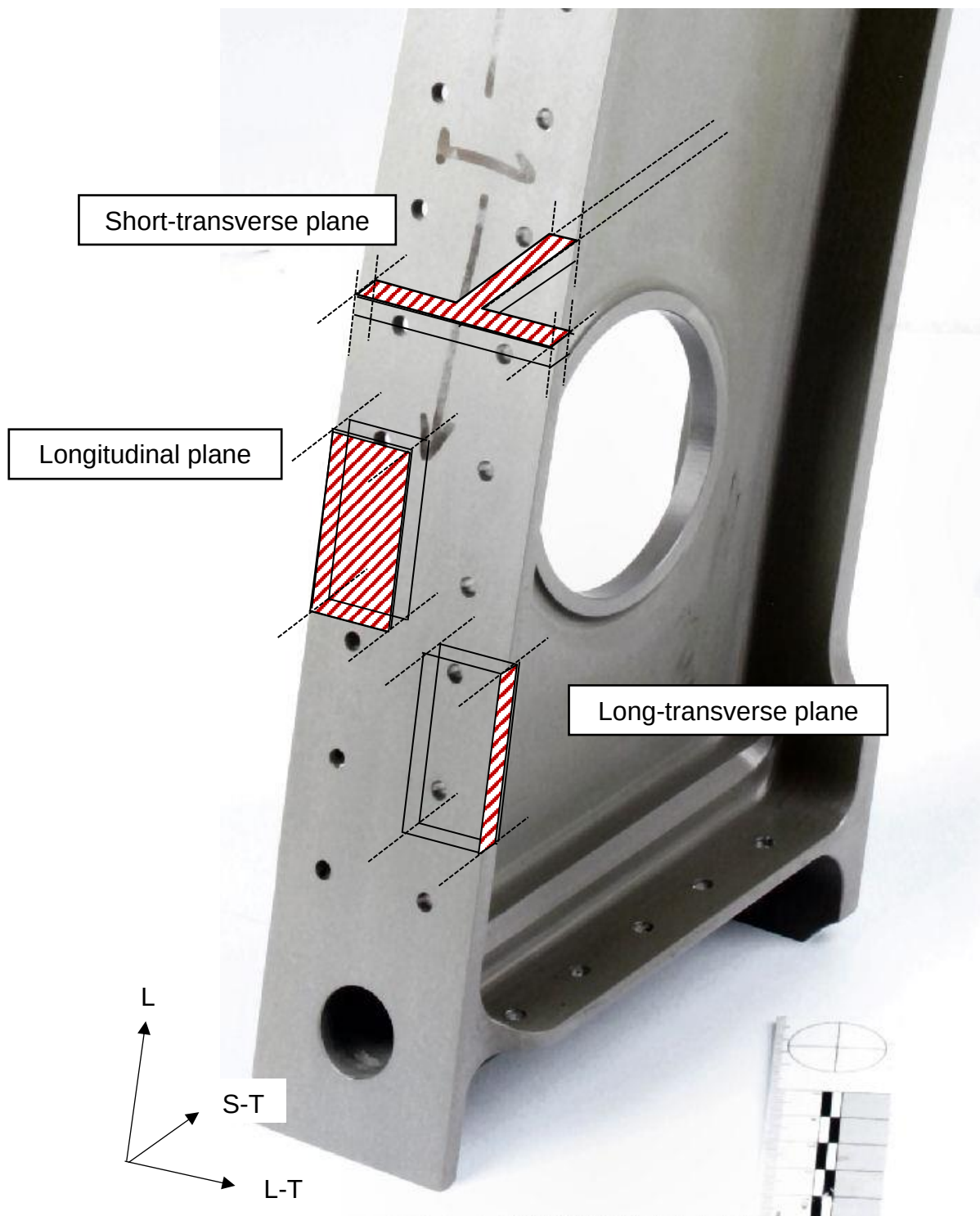


**Figure 27: Illustrating the grain orientations in a section of main lift frame.**

The samples were sectioned using Struers Discotom and Accutom cutting systems, ground and polished to a 1 $\mu$ m diamond polish finish and etched using Keller's reagent.

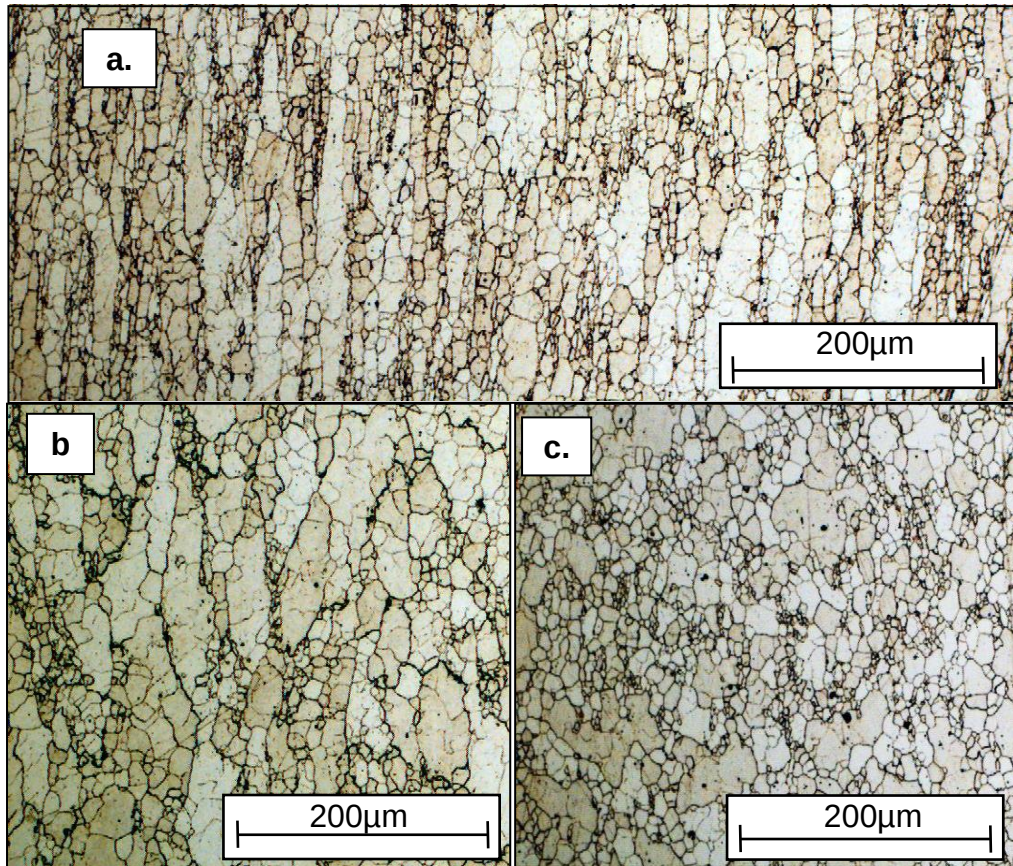


**Figure 28:** Showing the microstructure of the main lift frame material, longitudinal (a), long-transverse (b) and short-transverse (c) orientations, etched using Keller's reagent.

To enable a clear distinction between the grain orientations (for mechanical testing) they shall be referred to as the longitudinal (L), long-transverse (LT) and short-transverse (S-T) orientations, respectively.

### **3.5 Determining the mechanical properties of the main lift frame material - tensile testing.**

Tensile testing has long been established (within the aviation industry) as one of the main destructive testing methods for the qualification of mechanical properties of materials against approved material specifications. In particular, components that undergo solution and precipitation hardening heat treatments are closely monitored to ensure they meet the specified mechanical property requirements.

#### **3.5.1 Methodology used for tensile testing of main lift frame material.**

The standard tensile test piece employed in the qualification of materials used on UK MoD aircraft are specified to meet the requirements of BS EN ISO 2002-1:2005<sup>102</sup>. Annexes within the specification provide details of the critical dimensions for the test pieces<sup>103</sup>, and a representative schematic of a typical 'dogbone' tensile test piece is shown in Figure 29.

However, for this project, the dimensions of a standard size test piece were found to be too large to enable the manufacturing of a suitable number of test pieces from the limited material found to be present on the available main lift frames. An example of 'standard' size test piece in comparison with a section of a main lift frame can be seen in Figure 30.

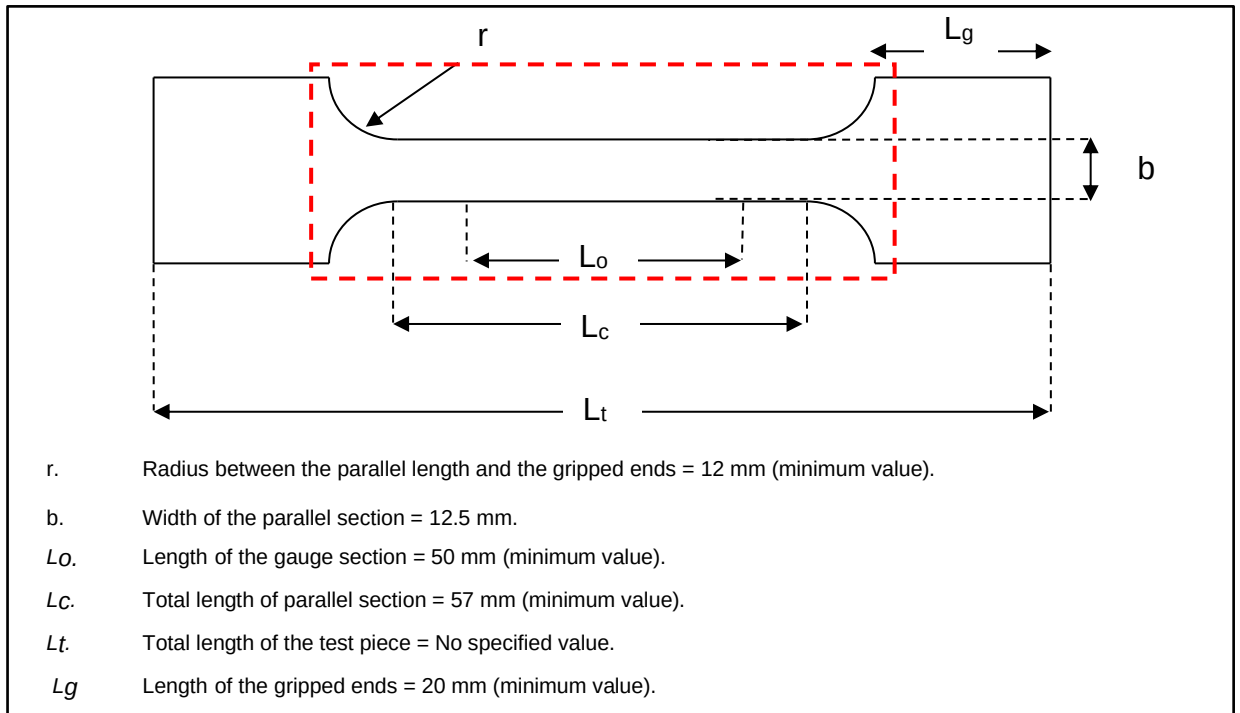


**Figure 29: Showing a schematic of a standard size 'dogbone' shaped tensile test piece, in accordance with: BS EN ISO 2002-1:2005<sup>102</sup>.**

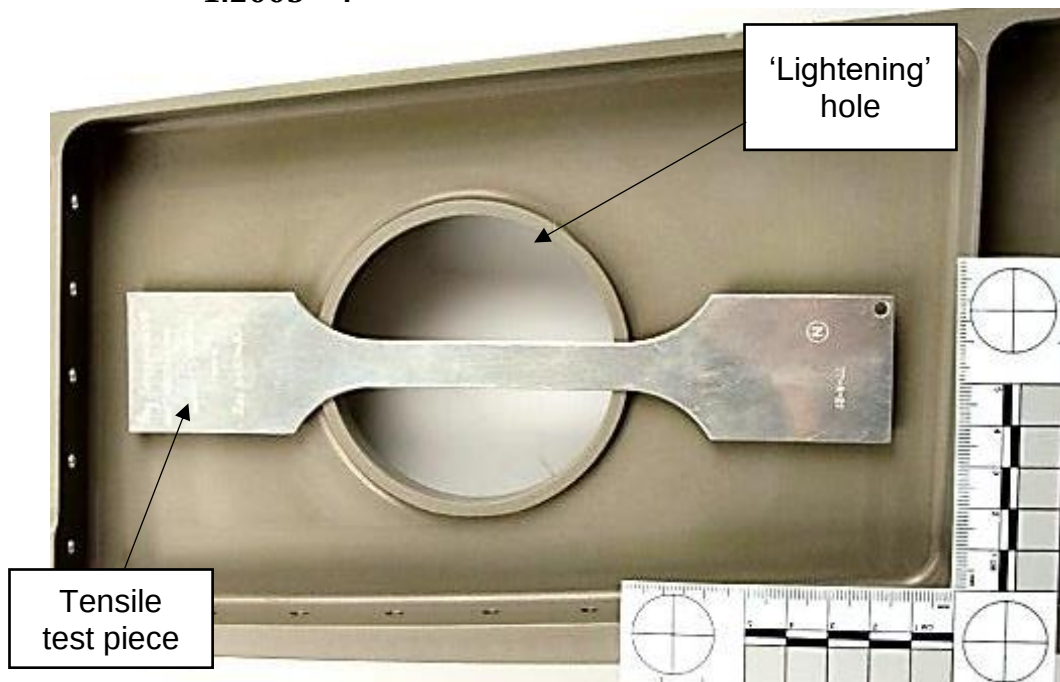


**Figure 30: Comparing a standard sized tensile test piece against a section of main lift frame.**

As can be seen in Figure 30, the ‘lightening’ holes on the lift frames prevented the production of standard sized test pieces and it therefore became apparent that a reduced size tensile test piece would be required for this project. Reduced size test pieces are not described in detail BS EN 2002-1:2005, but the subject is expanded upon in BS EN ISO 6892-1:2009<sup>104</sup> which states that:

*“By using an original gauge length smaller than 20mm, the measurement uncertainty is increased”<sup>104</sup>.*

However, unlike manufacturing standards which typically limit testing to a maximum for two test pieces for the same component, this project intended to obtain the maximum number of available test pieces from the lift frames (i.e. significantly more than two samples to achieve the greatest available data set), whilst adhering to the aforementioned > 20 mm gauge length limitation. The tensile specimens were initially cut from individual sections of main lift frames in both the longitudinal and long-transverse orientations using a XYZ milling machine. This process required the lift frames to be sectioned into four smaller pieces and placed into the milling machine. In addition, a cutting programme had to be designed for each individual section – which required precise alignment for the positioning of each individual section in the correct position for cutting. An example of the original sectioning process is shown in Figure 31. In addition, there is a difference in thickness between the top and bottom of the main lift frames. As shown in Figure 33, the main lift frames were measured to be 2.6 mm thick at the top and 4.3 mm thick bottom.



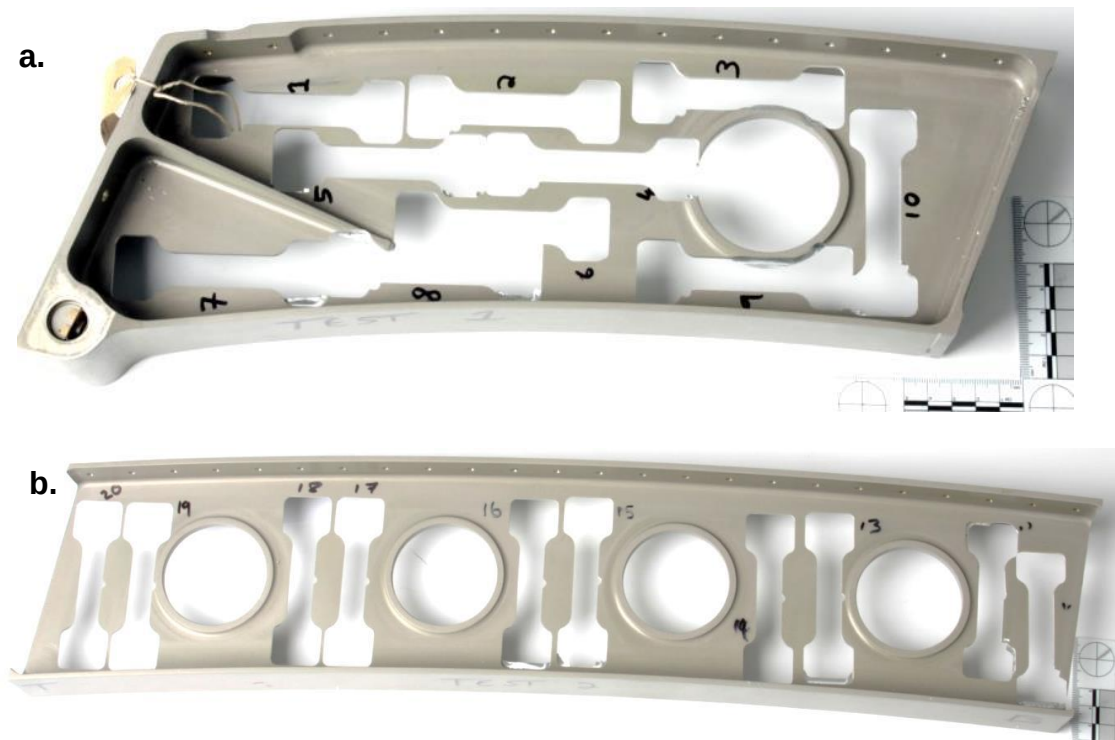


**Figure 31:** Showing a main lift frame post machining of the reduced size tensile test specimens in a) the longitudinal and b) long-transverse orientations.



**Figure 32:** Showing the comparison between a standard tensile test piece and two reduced sized tensile test pieces.



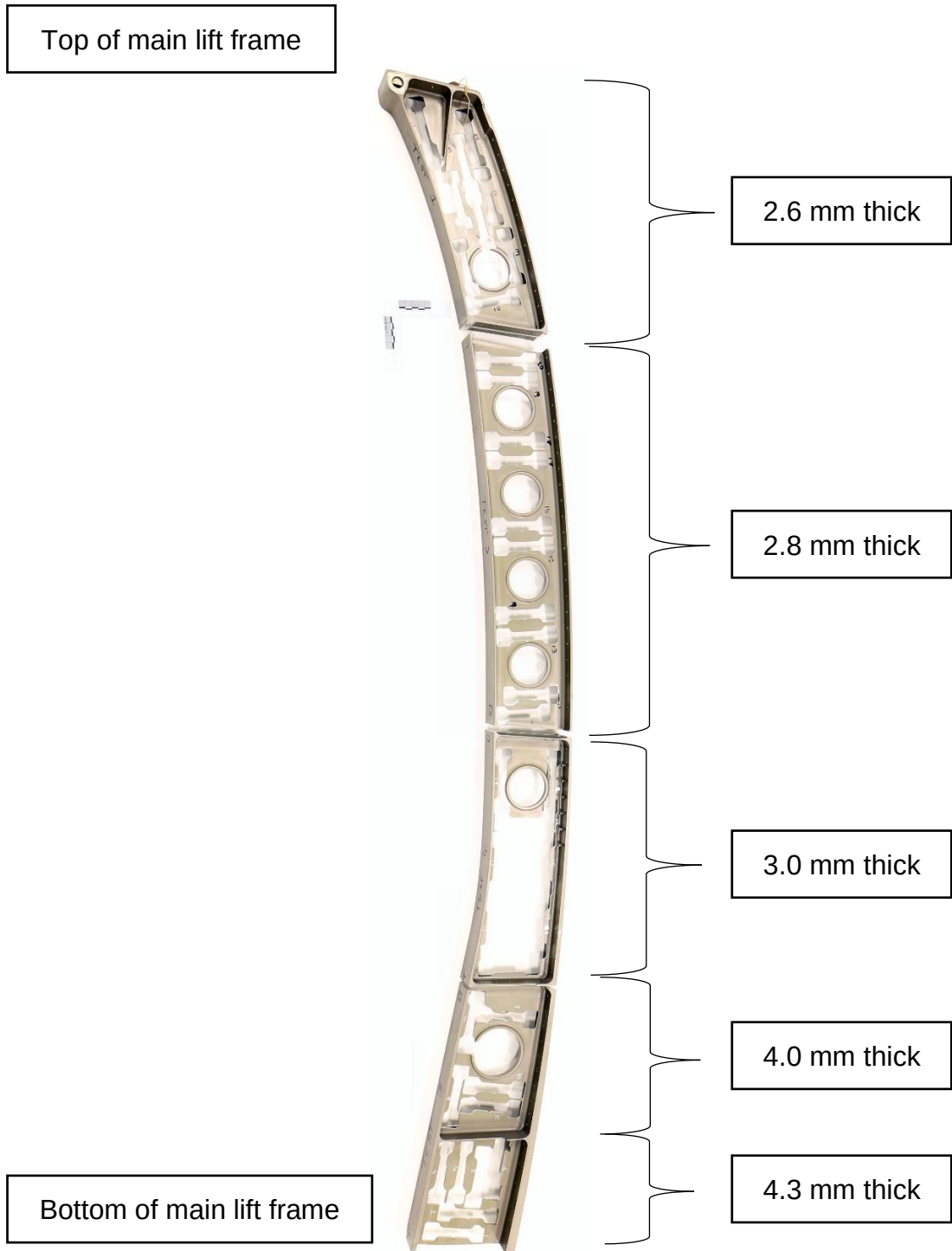


**Figure 33: Showing the change in thickness in the main lift frames.**

Therefore, there was a change in thickness in the tensile test pieces that is dependent on where they had been cut from the main lift frame.

### **3.5.2 Tensile testing terminology.**

For clarity, the following terminology (taken from G.E Dieter, 1988)<sup>105</sup> is used when referring to the measured mechanical properties resulting from tensile tests reported in this research;

#### **3.5.2.1 Ultimate tensile strength (UTS).**

The ultimate tensile strength of a test piece is the maximum tensile stress a material can withstand prior to failing and is calculated by the maximum load attained on the load vs extension stress-strain curve divided by the original cross-sectional area of the loaded test sample. It is strongly argued by Dieter that the UTS value of a material is dependent upon the onset of instability and localised deformation and therefore is of little to no significance in the modern engineering design world, and it is the yield strength value that all engineering designers (using ductile materials) adhere to. However, the UTS of a material is an easily determined and repeatable value which is very useful for the purposes of quality control and specifications.

#### **3.5.2.2 0.2% proof stress.**

The stress required to produce the onset of permanent plastic deformation in a material is the yield stress. In a wide range of materials, determination of the exact moment of yield is relatively hard to define as there is a gradual transition from elastic to plastic deformation. Therefore, an offset yield strength – the stress at which a line on the stress/strain curve that is parallel to the elastic part of the curve offset by a specific strain (typically intersecting the stress/strain curve at 0.2%), is often used and is also commonly referred to as the 0.2% proof stress.

### 3.5.2.3 Elongation.

The ductility of a material is determined by the engineering strain (change in length / original length) up to the point of failure. Engineering strain is often referred to as elongation and is expressed as a percentage of the original length and referred to as % elongation. In this document this will simply be referred to as elongation.

### 3.5.3 Classification of the different material conditions subjected to tensile testing and the terminology used in this work.

There is a significant amount of tensile testing data presented in the following sections. Whilst the material investigated remains the same - Al-Li 8090 main lift frame material in the T852 temper state, there are a number of different material conditions that have been investigated. For the purposes of clarity, it is proposed to provide a clear distinction between these different material states. Therefore, the material conditions and their classification system are as follows:

#### 3.5.3.1 Al-Li 8090 original equipment manufacturer (OEM) specification (8090/S).

8090/S will be used to refer to the OEM specification for Al-Li 8090 T852 for the production of the EH101 aircraft. The minimum specification data for the T852 temper state has been selected from the ASM Handbook (Vol 2)<sup>47</sup>. The minimum specification requirements for the 8090/S material is as shown in Table 11:

**Table 11: Showing the minimum mechanical property requirements for Al-Li 8090 in the T852 temper condition (8090/S)<sup>47</sup>.**

| Longitudinal Orientation |           |                         |                |
|--------------------------|-----------|-------------------------|----------------|
| Mechanical Properties    | UTS (MPa) | 0.2% proof stress (MPa) | Elongation (%) |
| Minimum Specification    | 425       | 340                     | 6.0            |

| Long-Transverse Orientation |           |                         |                |
|-----------------------------|-----------|-------------------------|----------------|
| Mechanical Properties       | UTS (MPa) | 0.2% proof stress (MPa) | Elongation (%) |
| Minimum Specification       | 405       | 325                     | 3.0            |

#### 3.5.3.2 As-received Al-Li 8090 main lift frame material. (8090/AMB).

8090/AMB will be used to describe the as-received Al-Li 8090 main lift frame material. This is the material detailed in para 3.1, in the ambient temperature condition. The data from this material will be used to provide the baseline for all other tensile testing heat treatment conditions undertaken in this work. Further testing groups and their classifications are detailed in Table 13, Section 4.

#### 3.5.4 8090/AMB main lift frame tensile test results.

To obtain a 'baseline' set of data for the mechanical properties of the in-service lift frame material, a main lift frame (Frame H2) has been subjected to tensile

testing<sup>xxiv</sup>. The test pieces for the longitudinal and long-transverse orientations (L and LT respectively) were tested at ambient temperature, (hence 8090/AMB) and compared against the specified mechanical properties of 8090/S. The results of which are shown in Figure 34 and Figure 35.

### **3.5.5 Longitudinal (L) 8090/AMB main lift frame tensile test results.**

#### **3.5.5.1 UTS and 0.2% proof stress.**

As can be seen in Figure 34, the overall results for the ultimate tensile strength (UTS) of the longitudinal (L) 8090/AMB test pieces was far superior to the minimum requirement and compared favourably against the results reported by Smith (1988)<sup>13</sup> (Figure 24). The results for 0.2% proof stress for the longitudinal grain orientation, which can be seen in Figure 34 show that the material met the minimum specified criteria, including the outlier results (initially suspected to be a result of substandard test piece preparation – see Section 4.2.8 for further work). For both the UTS and 0.2% proof stress results of the 8090/AMB material, the average data lines were at 68.9 MPa and 59.1 MPa which are 16% and 17%, respectively greater than the minimum specified standard. Smith's data shows similar results for UTS and 0.2% proof stress, with both data sets comfortably exceeding the minimum property requirements.

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<sup>xxiv</sup> *All of the tensile test samples were subject to tensile loading to destruction in a calibrated Zwick universal testing instrument, conforming to both ISO 7500 (Metallic materials - calibration and verification of static uniaxial testing machines) and ISO 17025 (General requirements for the competence of testing and calibration laboratories) standards.*

### 3.5.5.2 Elongation.

The elongation data for the 8090/AMB material is markedly different. As anticipated with the relationship between strength and ductility for Al-Li alloys, an increase in strength is normally accompanied with a resistance to plastic deformation (i.e. slip), and therefore results in a reduction in ductility. In the case of the 8090/AMB material, the scatter of the results is significant with only 8 of the 19 (42%) test pieces passing the minimum specification criteria with an average reduction of 15.7% below the minimum criteria. Again, test piece preparation was suspected to be a factor in these results, (see Section 4.2.8 for further work).

Smith's data, specifically; the 'UTS vs elongation' and '0.2% proof stress vs elongation' results for the longitudinal orientation (graphs, A and D, Figure 24)<sup>13</sup> were analysed using ImageJ software area calculations<sup>xxv</sup>. The analysis implied an approximate 78% and 63% pass rate for longitudinal UTS vs elongation and 0.2% proof stress v elongation, when compared against the ASM Handbook (1992)<sup>97</sup> minimum required mechanical properties (Table 11). In the UTS and 0.2% proof stress data, Smith's results show that even from the early days of Al-Li 8090, the minimum elongation requirement for tensile test pieces have not consistently been met.

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<sup>xxv</sup> *The calculations were made using pixel-based area measurements from the graphs in Figure 24 as no numerical data set was available.*

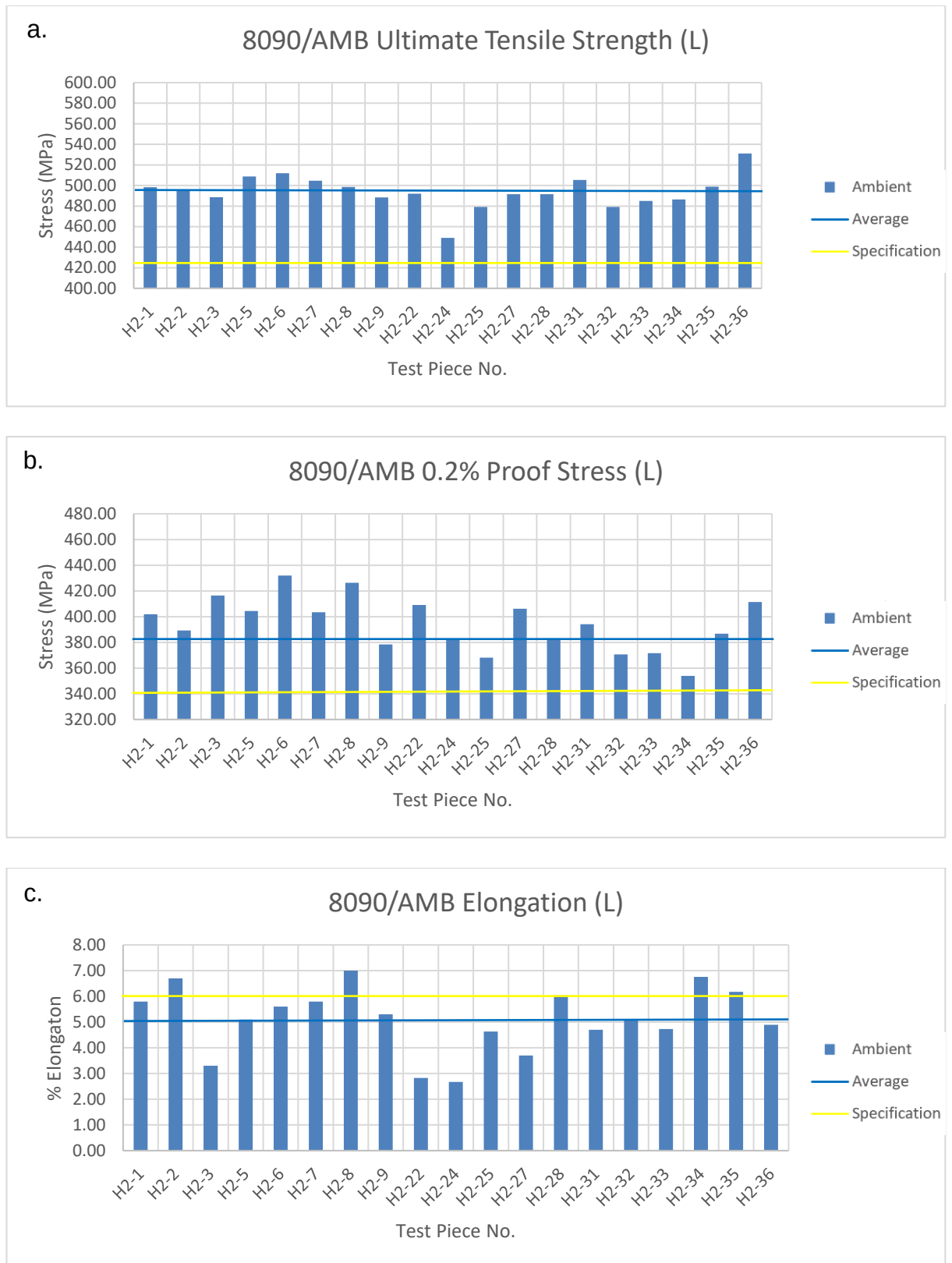


**Figure 34: Showing the 8090/AMB a) UTS, b) 0.2% proof stress and c) elongation test results for the L orientation.**

### **3.5.6 Long-Transverse (LT) 8090/AMB main lift frame tensile test results.**

#### **3.5.6.1 UTS and 0.2% proof stress.**

The tensile test results for the LT orientation are presented in Figure 35. Again, both the UTS and 0.2% proof stress results for the 8090/AMB material are within minimum specifications although with an increase in scatter for the thicker test pieces (which are situated at the lower end of the frame sections) for the 0.2% proof stress results.

Scatter for the UTS results is improved, with a slight increase in value again towards the thicker end of the test pieces (note the test piece numbers – the lower numbers are situated at the upper end of the frames which has a thinner cross section which increases towards the lower end of the frame). For the UTS and 0.2% proof stress results, the average data lines revealed a 100 MPa (19.8%) and 76.7 MPa (19.1%) increase over the minimum standard, respectively

#### **3.5.6.1 Elongation.**

The elongation results for the LT orientation have a significant scatter – as is consistent with the longitudinal results, but with a far improved pass rate of 17 out of 18 tensile test pieces meeting or exceeding the 6% minimum elongation requirement. In addition, there was an average increase in elongation of the 8090/AMB test pieces of 1.35% over the minimum specification requirement, which overall, represents a 31.1% increase.



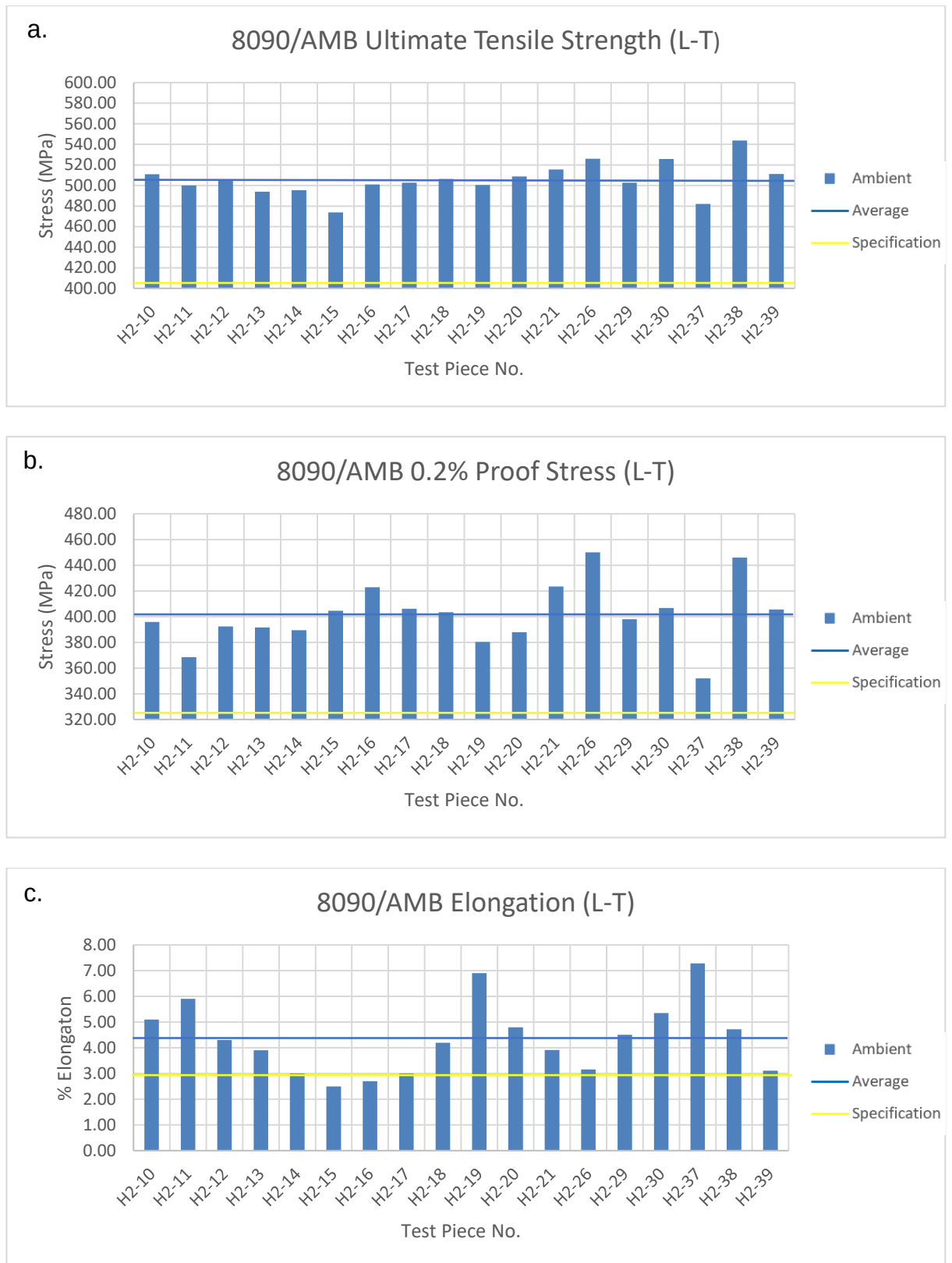


**Figure 35: Showing the 8090/AMB a) UTS, b) 0.2% proof stress and c) elongation test results for the LT grain orientation.**

Separating the elongation results between the L and LT grain orientations revealed a 100% pass rate for the LT orientation and a 40% pass rate for the L orientation, (see Annex A). Comparison of these results indicated surprising consistencies with Smith's results, Figure 24. It should also be noted that all of the test specimens passed the UTS and 0.2% proof stress requirements, all of the failures were associated with the elongation results of the longitudinal test pieces.

**Table 12: Showing the average mechanical properties of the 8090/AMB tensile test pieces compared against the minimum requirements of 8090/S<sup>47</sup> in both the longitudinal and long-transverse grain orientations.**

| Longitudinal Orientation |           |                         |                |
|--------------------------|-----------|-------------------------|----------------|
| Mechanical Properties    | UTS (MPa) | 0.2% proof stress (MPa) | Elongation (%) |
| 8090/S                   | 425       | 340                     | 6.0            |
| 8090/AMB                 | 495       | 394                     | 5.0            |

| Long-Transverse Orientation |           |                         |                |
|-----------------------------|-----------|-------------------------|----------------|
| Mechanical Properties       | UTS (MPa) | 0.2% proof stress (MPa) | Elongation (%) |
| 8090/S                      | 405       | 325                     | 3.0            |
| 8090/AMB                    | 506       | 402                     | 4.4            |

### **3.6 Summary of characterisation of the 8090/AMB main lift frame material.**

The initial metallurgical and mechanical characterisation testing of the 8090/AMB material has shown that the material met the specified criteria for the elemental chemical composition and the majority of the minimum specified mechanical properties (Table 10 and Table 12, respectively). In addition, as stated in Section 3.5.6, there is a notable consistency between the L and LT results reported by Smith (1998)<sup>13</sup>. Therefore, it is accepted that the existing literature appertaining to Al-Li 8090 in the T852 temper state is applicable to the 8090/AMB material used in this investigation. Therefore, this information can be drawn upon for comparison during further testing carried out in this work.

In particular, since the change in behaviour of mechanical properties of the 8090/AMB material is the main objective of this work, a representative temperature for 'operational low temperature exposure' had to be defined and this is detailed in the next Section. A more complete metallurgical characterisation of the microstructure will be detailed in Section 5.

#### **4 Thermal stability of Al-Li 8090 main lift frames and low temperature exposure in operational environments.**

As previously mentioned in Section 2.2.5, Al-Li alloys exhibit a change in mechanical properties when exposed to temperatures in the range of 70-100°C (and above). Whilst a number of authors including McDarmaid and Starr (1990)<sup>59</sup>, Lynch (1991)<sup>82</sup>, McDarmaid (1993)<sup>84</sup> and Pasang et al. (2012)<sup>67</sup> have focussed their efforts on the effect of temperature on the fracture toughness properties of Al-Li alloys, there is little or no information regarding 8090-T852 forgings (specifically forgings that have been subjected to in-service conditions). However, due to the dimensional limitations of the main lift frames, it was not possible to manufacture fracture toughness test pieces that would have been dimensionally consistent with existing data (or to current testing standards). Therefore, as a key requirement of fracture toughness testing is consistency in test piece size, no comparable data could be obtained.

However, much work regarding the thermal sensitivity of Al-Li to temperatures < 100°C (otherwise referred to as low temperature exposure (as per Section 2.2.5)), does provide evidence to suggest that prolonged exposure to temperatures < 100°C can have notable effects on some of the microstructural and mechanical properties of Al-Li alloys, which will be discussed later in this Section.

With consideration of recent UK military operational environments – notably Operations TELIC<sup>xxvi</sup> (Iraq) and HERRICK<sup>xxvii</sup> (Afghanistan) – a question was raised regarding the potential for operational aircraft (including Merlin<sup>106</sup>) to have been subjected to low temperature exposure whilst operating in relatively hot environments. Therefore, a series of experiments was undertaken to identify whether operating in a ‘hot’ environment could have had a permanent or temporary effect on the mechanical properties of Al-Li 8090 T852 main lift frames.

#### 4.1 Aircraft low temperature exposure.

The ‘location’ for the representative temperature data for a ‘hot’ operating environment was chosen to be Camp Bastion, which was the main operating base for British forces during Operation HERRICK and was the primary location for Merlin aircraft. This location and time period were selected due to the following factors:

- 1) It was representative of a location in which Merlin aircraft are known to have operated in.
- 2) Being a more recent operation, the quality of recording and accessibility of data was perceived to be superior to older operations.

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<sup>xxvi</sup> *British Operations in Iraq between 2003 and 2011.*

<sup>xxvii</sup> *British Operations in Afghanistan between 2002 and 2014.*

- 3) It was known by the author that (unrelated) work had been undertaken regarding temperature data recording on aircraft – during Operation HERRICK.

The maximum measured environmental temperature (i.e. the standard air temperature) for Camp Bastion<sup>107</sup> over a period of time<sup>xxviii</sup> during Operation HERRICK is shown in Figure 36. Whilst this may be representative of what a human being on the ground may have experienced, with respect to aircraft structure there are additional factors that have to be taken into consideration.

Aircraft are often exposed to direct sunlight on a concrete (or similar material) hardstanding, which additionally reflects heat towards the aircraft. In addition, aircraft are covered in a variety of protective coatings (for a multitude of purposes) and these coatings are often matt in finish and thermally absorbent. In addition, the outer skin of an aircraft (directly exposed to sunlight) will differ in temperature to a main lift frame or wing spar – (the inner structure that is not directly exposed to sunlight). These factors (and others) are likely to increase the overall temperature of an aircraft above the standard measured air temperature. In addition, aluminium has a thermal conductivity of 205 W/mK which will be a salient factor where aluminium aircraft skin contacts main lift frame material. However, there is little information appertaining to the temperatures that aircraft can be subjected to in hot environments. Pasang et al. (2012)<sup>67</sup> state that structures exposed to ‘strong sunlight’ can reach

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<sup>xxviii</sup> *Three year average for Jan-Aug, two year average for Sep-Dec.*

temperatures of 80°C. There is no reference given as to where this figure was obtained, nor is this explained as either an internal or external temperature. A parallel is drawn to the potential that aircraft (discussed in the paper) may have been subjected to such conditions. More accurate, hot environmental testing on Merlin aircraft did record a peak temperature reading of 77°C in the vicinity of the tail rotor (QINETIQ/UK MOD, 2014)<sup>108</sup>. However, this was attributed to a hot testing environment combined with additional factors. Therefore, given the similarity between 80°C and 77°C, Pasang et al.'s (2012)<sup>67</sup> 80°C was chosen as the representative testing temperature. As can be seen in Figure 36, this prospective temperature, is notably higher than the recorded air temperature at Camp Bastion (a difference of 34.4°C from the highest recorded temperature).

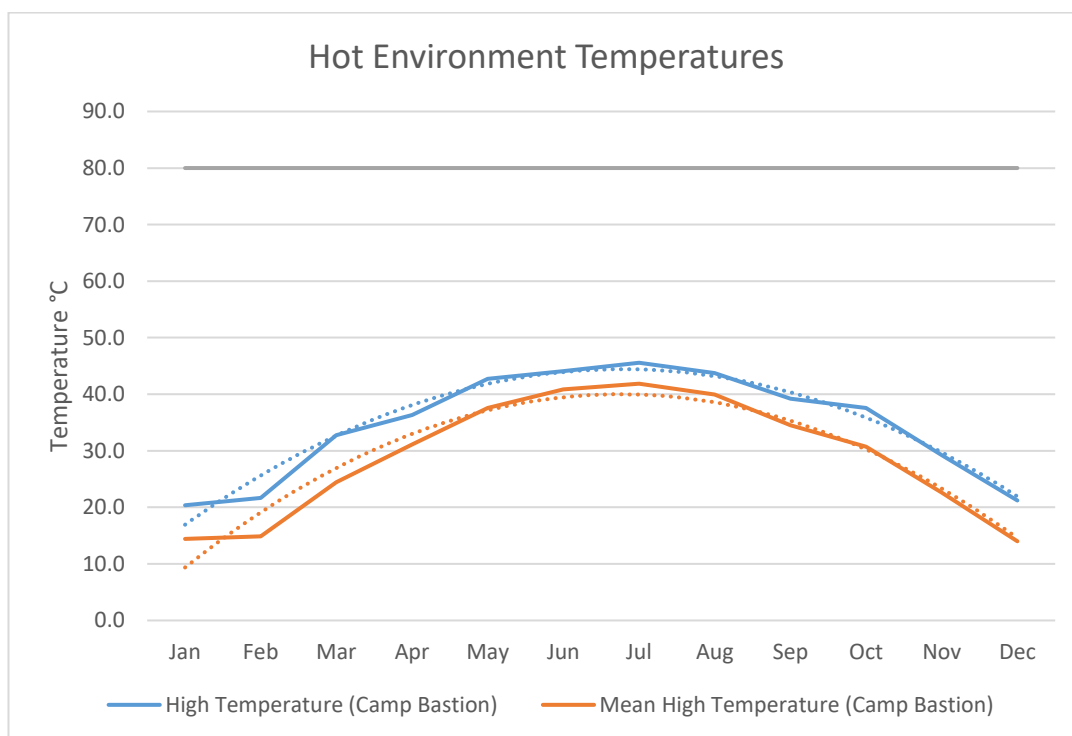


**Figure 36: Showing average air temperature measurements of Camp Bastion during a period of time of Operation Herrick<sup>107</sup> (with Pasang [et al.]’s 80°C for comparison).**

Additional data was obtained through DSTL (the Defence Science and Technology Laboratory)<sup>109</sup> from work that involved temperature data collection on aircraft during Operation HERRICK<sup>xxix</sup>. The data provided was obtained from four aircraft<sup>xxx</sup> covering the period May to September 2014. The temperature sensors were situated by the wing spar (inside the wing, near the fuselage) and were contained within a cast aluminium box, ensuring that there was no contact with direct sunlight.

Due to the significant amount of recorded data that was recorded by DSTL (2016)<sup>109</sup>, the data provided in Figure 37 only covers maximum temperatures that exceeded 50°C. As can be seen in Figure 37, the recorded DSTL temperature data shows that there is an increase over the standard recorded air temperature (a maximum of 12.4°C and an average of 11.1°C). The maximum recorded temperature of 56.1°C is 23.9°C lower than the proposed 80°C value by Pasang et al. (2012)<sup>67</sup>.

As shown in Figure 37, the average recorded temperature for the DSTL data set was 50.1°C and the peak temperature measured was 56.1°C. An average of these temperatures (53.1°C) was chosen to be used as the representative hot environment temperature investigated in this work.

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<sup>xxix</sup> *Some information in this report has been redacted to remove any Official-Sensitive classification information. There has been no alteration to the dates or recorded temperatures.*

<sup>xxx</sup> *It should be noted that these were not Merlin aircraft.*



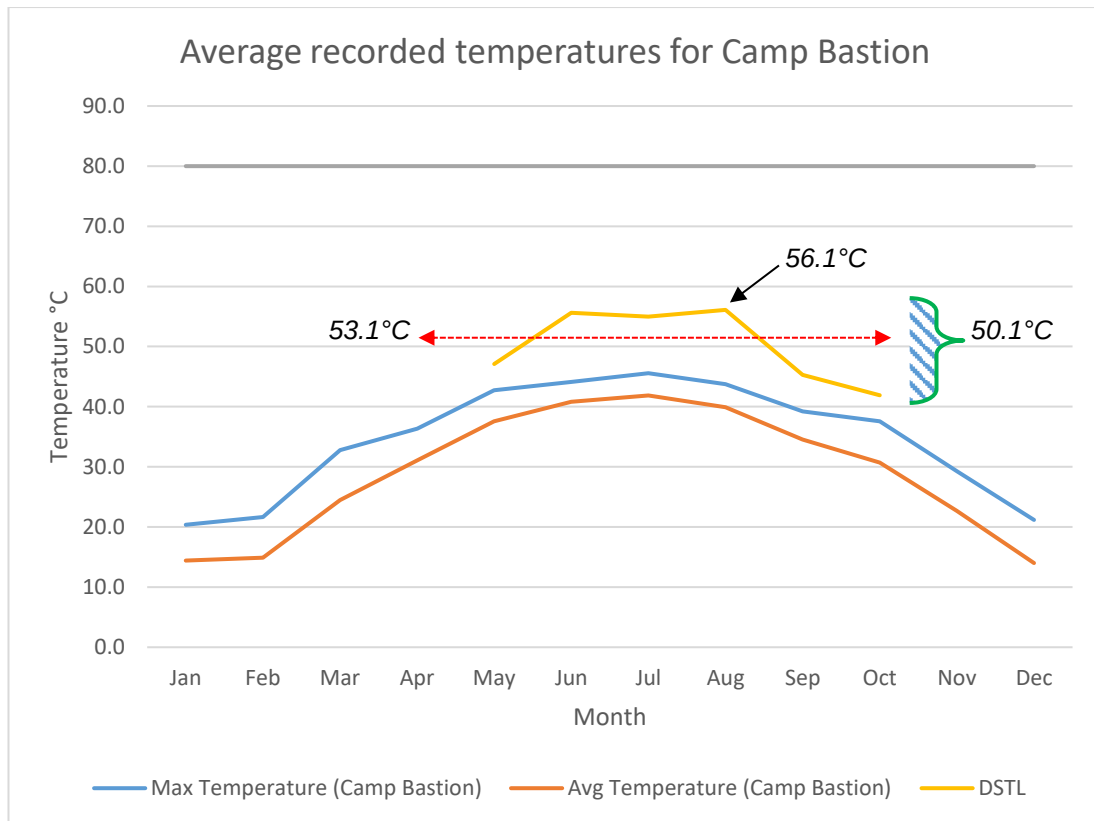


**Figure 37: Showing the recorded temperature data from within the aircraft.**

#### **4.2 Determination of the effect of operational low temperature exposure on the mechanical properties of Al-Li main lift frames.**

With the two determined values for in-service temperatures (80.0°C and 53.1°C) a programme of testing was undertaken to determine whether these temperatures had an effect on the mechanical properties of the in-service main lift frames. In total, five main lift frames were used to manufacture tensile test pieces to the same standards as was used for the 8090/AMB material as detailed in Section 3.5.1.

For comparison with existing data sets - for example work undertaken at DRA Farnborough by McDarmaid (1995)<sup>61</sup>, the length of soaking time at the specified temperatures was initially selected to be 1000 hours (six weeks). Further testing extending this to 1500 hours.

The test pieces were soaked in a locally calibrated, dry environmental chamber that was periodically monitored and checked using an externally calibrated thermocouple. After the 1000 hours had been achieved, the test pieces were subjected to tensile testing (with no appreciable delay between leaving the environmental chamber to testing). As with the 8090/AMB material testing, the location of the test pieces from each main lift frame was recorded. A selection of images showing the location of test pieces sectioned from Frames A2, B2, C2 and H2 are shown in Annex C. The following series of tests were undertaken:

#### **4.2.1 Tensile testing of Al-Li samples heat treated for 1000 hours at 80.0°C and 53.1°C.**

To determine whether the 80.0°C temperature taken from the Pasang et al. (2012)<sup>67</sup> had an effect on the mechanical properties of the in-service main lift frame structure, tensile test pieces sectioned from Frame B2 were subjected to a 1000 hour soak at 80.0°C with the results for the longitudinal orientation test pieces shown in Figure 38. As can be seen from Figure 38, the 1000 hour soak resulted in a minor increase in the UTS – an average increase of 11 MPa against the 8090/AMB data. It also resulted in an average increase in the 0.2% proof stress of 28 MPa when compared against the 8090/AMB material. It

should be noted that test piece B2-3 (Frame B2, No.3) attained what is believed to be an anomalous result for the UTS value and has been omitted from the average ambient data. This was due to poor machining during manufacture.

Comparison of the 80°C elongation results with the 8090/AMB data, revealed an average reduction in elongation of 2.7%. Although this could be interpreted as a relatively low reduction in ductility, due to the specification requirement being such a low initial value (6%), this amounts to a 45% reduction in ductility from the specification requirement and a 36% reduction when compared against the 8090/AMB results (5%), which in both cases, is significant.

Similar to the test pieces soaked at 80.0°C, a set of tensile test pieces sectioned from Frame A2 were subjected to a 1000 hour soak at 53.1°C, followed by tensile testing. The results for which are shown in Figure 39. The results revealed an increase of 40 MPa for UTS (a 7.6% increase against ambient testing) and a 60 MPa increase in 0.2% proof stress (13% increase against ambient testing), which are both superior to the minimum specification requirement. A comparable reduction in elongation as per the 80.0°C soaked samples was recorded, with a 36% reduction from the 8090/AMB results and a 45% reduction from the minimum specification requirement. There was a notable correlation between the reduction in elongation results for the samples soaked at 80.0°C and 53.1°C. The 80.0°C average result is overlaid on the elongation graph (C), Figure 39.

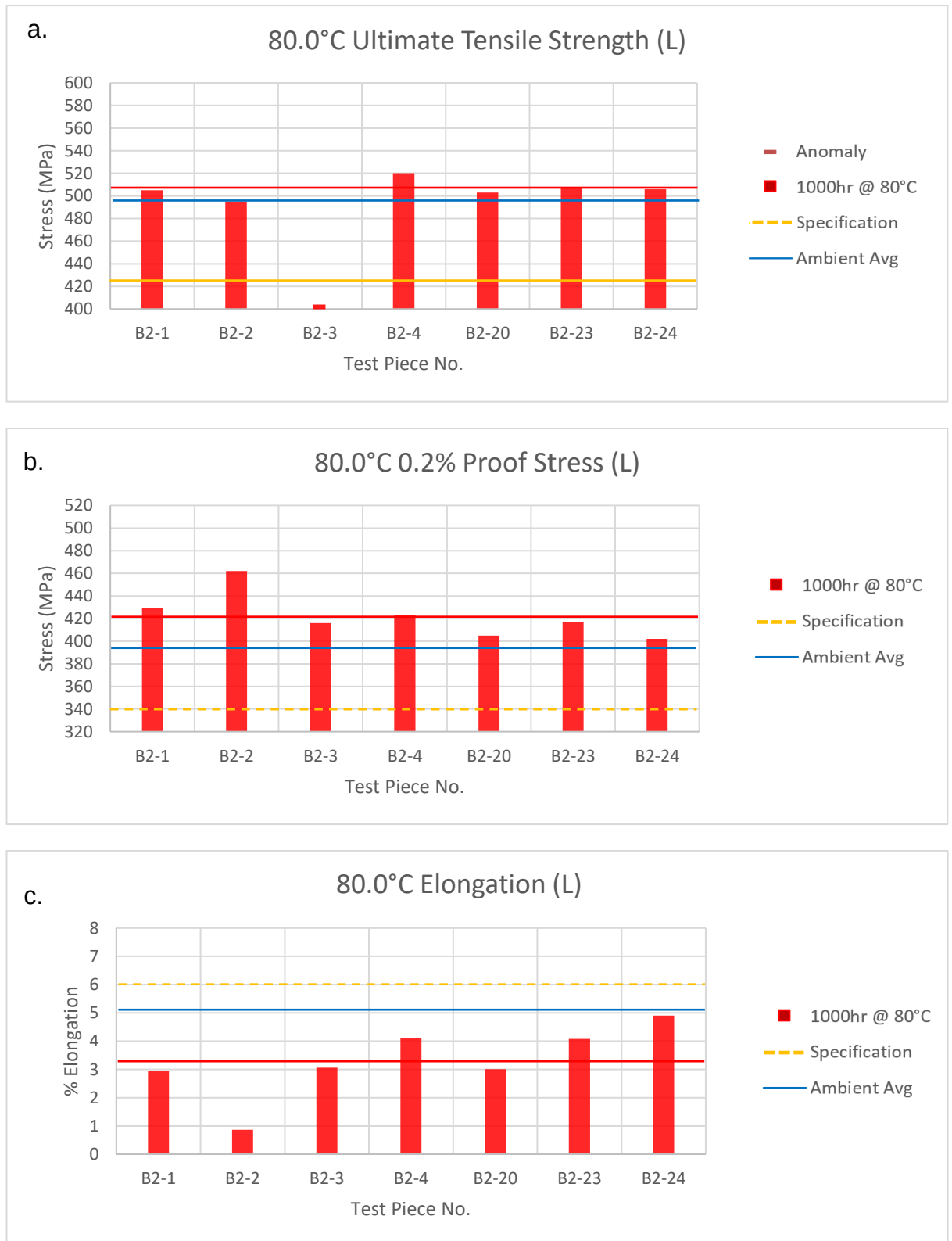


**Figure 38:** Showing the tensile test results for the 8090/AMB and 80°C 1000hr soaked test pieces. a) UTS, b) 0.2% proof stress, C) elongation. The yellow dotted line in each graph represents the minimum specification requirement.

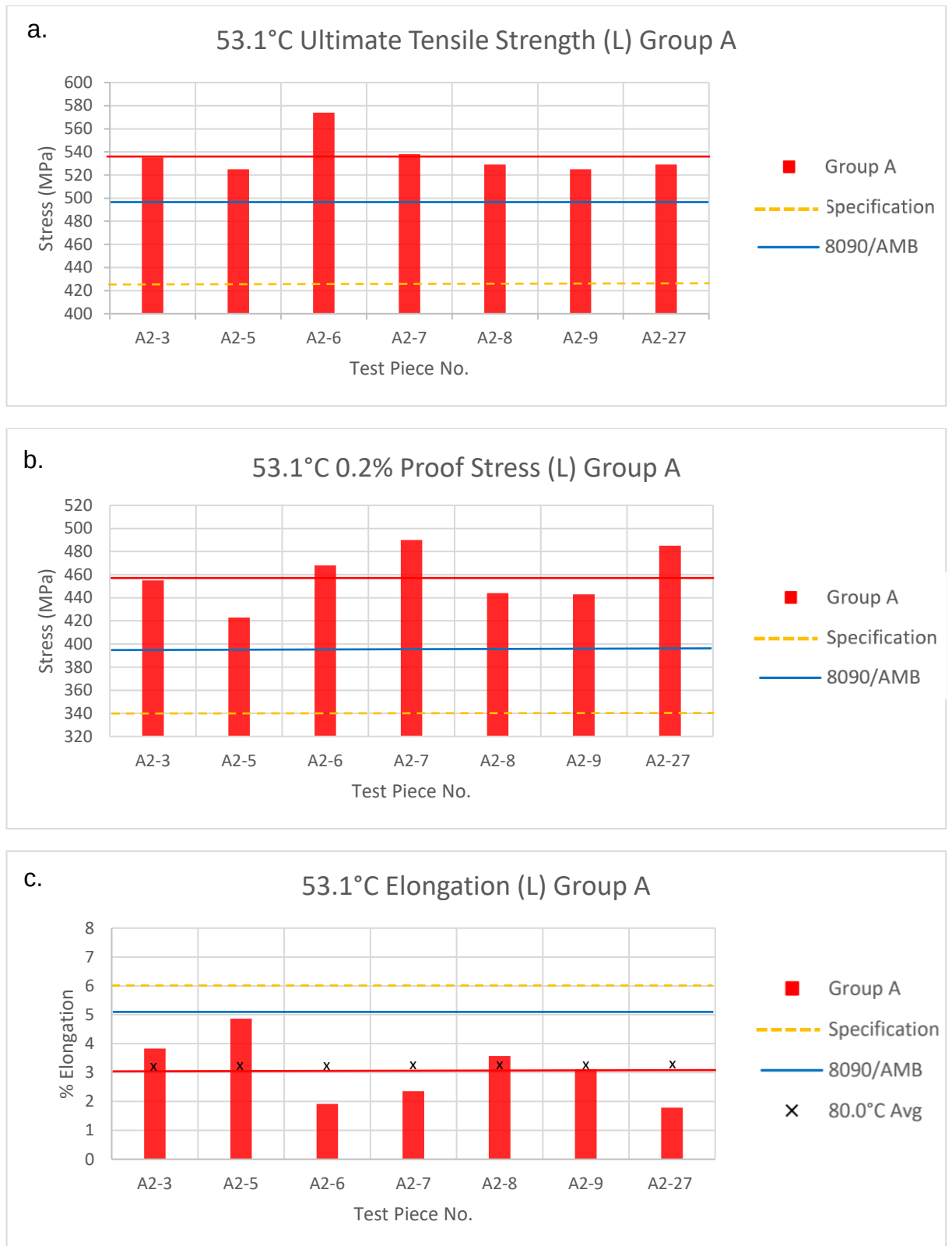


**Figure 39: Showing the tensile test results between the as-received and 53.1°C 1000hr soaking. The dotted yellow line in each graph represents the minimum specification requirement.**

#### **4.2.2 Designation of the tensile testing groups subjected to various 53.1°C heat treatments.**

The following section of this project documents the results and progression of the tensile testing regime on the Al-Li main lift frame material. Throughout this section, reference is made to a number of testing Groups that use similar soaking times and/or temperatures which for clarity, are detailed in Table 13. A summary of the heat treatments and the findings for each Group are detailed in Section 4.3.

#### **4.2.3 Comparison of results for the 80.0°C and Group A (53.1°C) tensile test pieces.**

Whilst taking the scatter of the results into consideration, the initial results for both of the 80.0°C and Group A (53.1°C) test pieces indicate that the 1000 hour soak for both temperatures resulted in an increase in UTS and 0.2% proof stress and a reduction in elongation. The change in mechanical properties for the 80.0°C does not come as a surprise, as it correlates with the findings of others who have examined low temperature exposure effects on the mechanical properties of Al-Li alloys (see Section 2.2.5).

The Group A results indicate that a change in mechanical properties may occur at temperatures lower than originally anticipated. Furthermore, the notable correlation in the reduction in elongation for both temperatures (elongation graph (C), Figure 39) suggests that a consistent and potentially repeatable mechanism may be occurring.

**Table 13: Showing the details of the tensile testing and heat treatment designations.**

| Group | Soak time (hours) | Temperature (°C) | Frame <sup>xxxi</sup> | Orientation | Description                                                       |
|-------|-------------------|------------------|-----------------------|-------------|-------------------------------------------------------------------|
| 80°C  | 1000              | 80               | B2                    | L           | 1000 hours at 80°C                                                |
| A     | 1000              | 53.1             | A2                    | L           | 1000 hours at 53.1C                                               |
| B     | 1000<br>1000      | 53.1<br>Ambient  | A2                    | L           | 1000 hours at 53.1C followed by 1000 hours at ambient temperature |
| C     | 1500              | 53.1             | C2                    | L           | 1500 hours at 53.1°C                                              |
| D     | 1500<br>1500      | 53.1<br>Ambient  | C2                    | L           | 1500 hours at 53.1C followed by 1500 hours at ambient temperature |
| E1    | 72                | 53.1             | F2                    | L           | 72 hours at 53.1°C                                                |
| E2    | 72                | 53.1             | F2                    | LT          | 72 hours at 53.1°C                                                |
| F1    | 72                | 53.1             | G1                    | L           | 72 hours at 53.1°C                                                |
| F2    | 72                | 53.1             | G1                    | LT          | 72 hours at 53.1°C                                                |
| G1    | 72                | 53.1             | F2                    | L           | 72 hours at 53.1°C                                                |
| G2    | 72                | 53.1             | F2 + G1               | LT          | 72 hours at 53.1°C one test piece taken from frame G1             |
| H     | N/A               | Ambient          | E1                    | L           | Additional ambient tensile test pieces                            |

<sup>xxxi</sup> Note that testing Groups and sectioned frames share similar monikers. Therefore, a test piece in Group E named F2-17 is taken from Frame F2, test piece no. 17.

**4.2.4 Material limitations of testing AL-Li 8090 main lift frames.**

Due to limitations regarding the amount of material (and ultimately, the number of available test pieces), the following work centred on the potential effects emanating from exposure to the lower 53.1°C temperature. The justification for this is that the effect of exposure to 70°C and above on Al-Li 8090 has had significantly more investigation than material subjected to temperatures lower than 70°C. Furthermore, designers of rotary wing aircraft have access to heat resistant designs and materials (typically titanium panels), and these could be adopted to mitigate any detrimental effects, if required. Therefore, the decision was taken to focus on the 53.1°C temperature and investigate any effects on the mechanical properties from exposure to this temperature.

**4.2.5 Replicating the reported metastable effect of ambient temperature exposure on the mechanical properties of Al-Li 8090.**

Following on from the reported reversion in mechanical properties of Al-Li tensile test pieces in the findings of Pitcher (1993)<sup>81</sup> and McDarmiaid (1992)<sup>59,60</sup>, regarding reversion age heat treatments and exposure to ambient temperature, Pitcher (1993)<sup>81</sup> concluded that after a period of time at ambient temperature, any toughness increase following the heat treatment of reversion aged Al-Li sheet was predicted to (eventually) revert back to the original mechanical properties. Pitcher (1993)<sup>81</sup> suggested that there were no permanent effects on the mechanical properties from the reversion age heat treatment and therefore theoretically, multiple reversion heat treatments could be employed, as required. Pitcher therefore concluded that there is a metastable nature to the mechanism, responsible for the changes in



mechanical properties. Therefore, in an attempt to determine whether the in-service Al-Li main lift frame material exhibited similar metastable behaviour to Pitcher's observations, a second set of test pieces (designated Group B) were run alongside the original set (Group A). Group B consisted of an identical set of test pieces, also manufactured from Frame A2 and subjected to 1000 hours at 53.1°C at the same time as Group A.

#### **4.2.6 Group B tensile test results (1000 hours at 53.1°C and 1000 hours at ambient temperature).**

To determine whether there was a metastable effect on the mechanical properties of the Al-Li 8090 material, the Group B tensile test pieces were removed from the oven at the same time as Group A and were left for an additional 1000 hours at ambient temperature. The tensile test results for the Group B longitudinal test pieces can be seen in Figure 40. The average data values for Group A are included for direct comparison.

As can be seen in Figure 40, both the UTS and 0.2% proof strength results for Group B show an increase when compared to the 8090/AMB results (24 MPa / 4.6% and 42MPa / 9.7%, respectively), but not to the extent of Group A. Furthermore, the elongation results for Group B are less reduced (0.74% / 14.5% overall value) from the 8090/AMB testing results as compared to Group A (1.84% / 36% overall value).

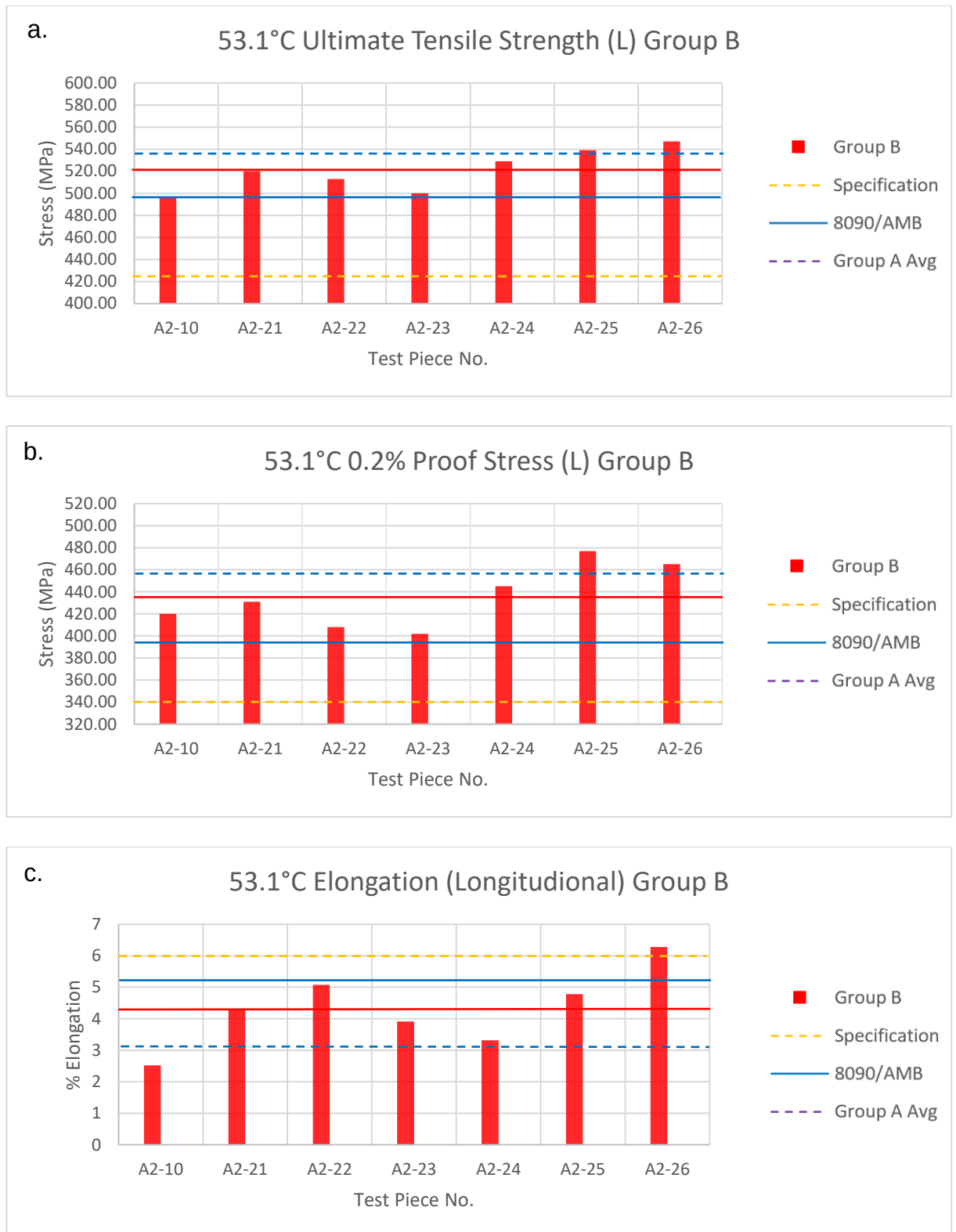


**Figure 40: Comparing the tensile test results for Groups A and B with the 8090/AMB results, a) UTS, b) 0.2% proof stress, C) elongation. The yellow dotted line represents the minimum specification.**

Therefore, as Group B was a part of the same 1000 hour soaking as Group A and both Groups originated from the same main lift frame, the comparative findings between the two Groups indicate that the change in mechanical properties as is evident from the testing of Group A have, in some manner recovered/reverted back towards the 8090/AMB temperature data. This indicates that there is a metastable or equilibrium/non-equilibrium process occurring within the material, which supports the findings of Pitcher (1993)<sup>81</sup> and his work with Al-Li sheet material.

#### **4.2.7 A note on anomalous results and test piece preparation.**

It must also be noted that the 0 hour sample for the 53.1°C tests also exhibited a reduction in elongation. On further inspection of the test piece (Frame A2, Test Piece No.27) it was noted that the preparation of the test piece was in a less than satisfactory condition – machining marks were found to be present on the parallel length of the 0 hours specimen. Any stress concentration on a parallel length of a test piece – for example from the machining process, or accidental damage prior to testing has the potential to act as a stress raiser (by adding a ‘notch’ to the parallel length) which can affect the test result (Khoza, 2016)<sup>110</sup>.

Further inspection of other test pieces identified additional potential stress concentrations that may have caused scatter in the results for both the 80.0°C and Group A test pieces – and it was considered that slight stress raisers on the parallel length of the test pieces could have had an effect on this set of results.

Subsequently, all future testing was subjected to further specimen preparation techniques to remove any stress concentrations. Although each was finished by the 1710 NAS mechanical workshops, to minimise spurious results every manufactured test piece was further finished using a combination of hand filing and abrasion with multiple stages of progressively finer grit sand paper.

#### **4.2.8 Further investigation of experimental results from tensile testing data scatter.**

As is evident in the 8090/AMB results for both orientations (Section 3.5), and subsequent testing (80.0°C, Groups A & B), there is some scatter in the data points for all three mechanical properties, with elongation being the most significant. In an attempt to determine whether the cause of this scatter was due to variables appertaining to this investigation, a second batch of 8090/AMB tensile test pieces were prepared and tested, taking into account: the observations reported in Section 4.2.7 and consideration as to whether a change in cross-sectional area of the test pieces had a noticeable effect on the results. The reason for the second consideration is due to the increase in main-lift frame thickness descending towards the base of the frame.

Further to this, improvements to the machining process at 1710 NAS (procurement of a 4-axis milling machine for use instead of an XYZ milling machine), enabled entire main lift frames to be cut in a single operation and this allowed for test pieces to be orientated in the direction of grain flow (Figure 41 and Figure 42). Therefore, test Group H utilised a selection of test pieces that were inspected for any defects and prepared with 320 and 1000 grit

abrasive paper to ensure any minor machining notches or defects were removed. The results of test Group H were combined with the initial 8090/AMB testing data and the findings were arranged in order of calculated cross-sectional area ranging from the smallest (14.10 mm<sup>2</sup>) to the largest (26.02 mm<sup>2</sup>) (left axis to right axis) in the graphs shown in, Figure 43.

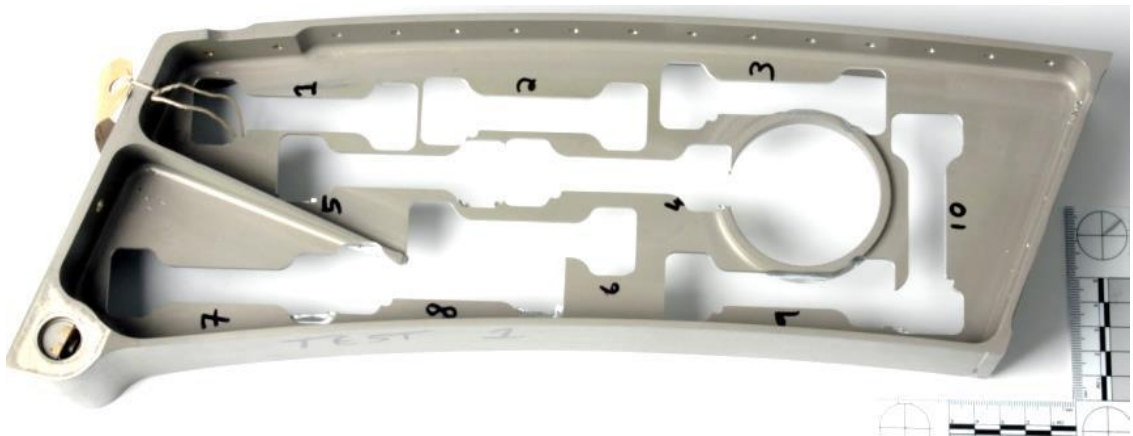


**Figure 41:** Showing the initial machining test piece orientation.



**Figure 42:** Showing the improved test piece manufacturing process, with orientated longitudinal test pieces.

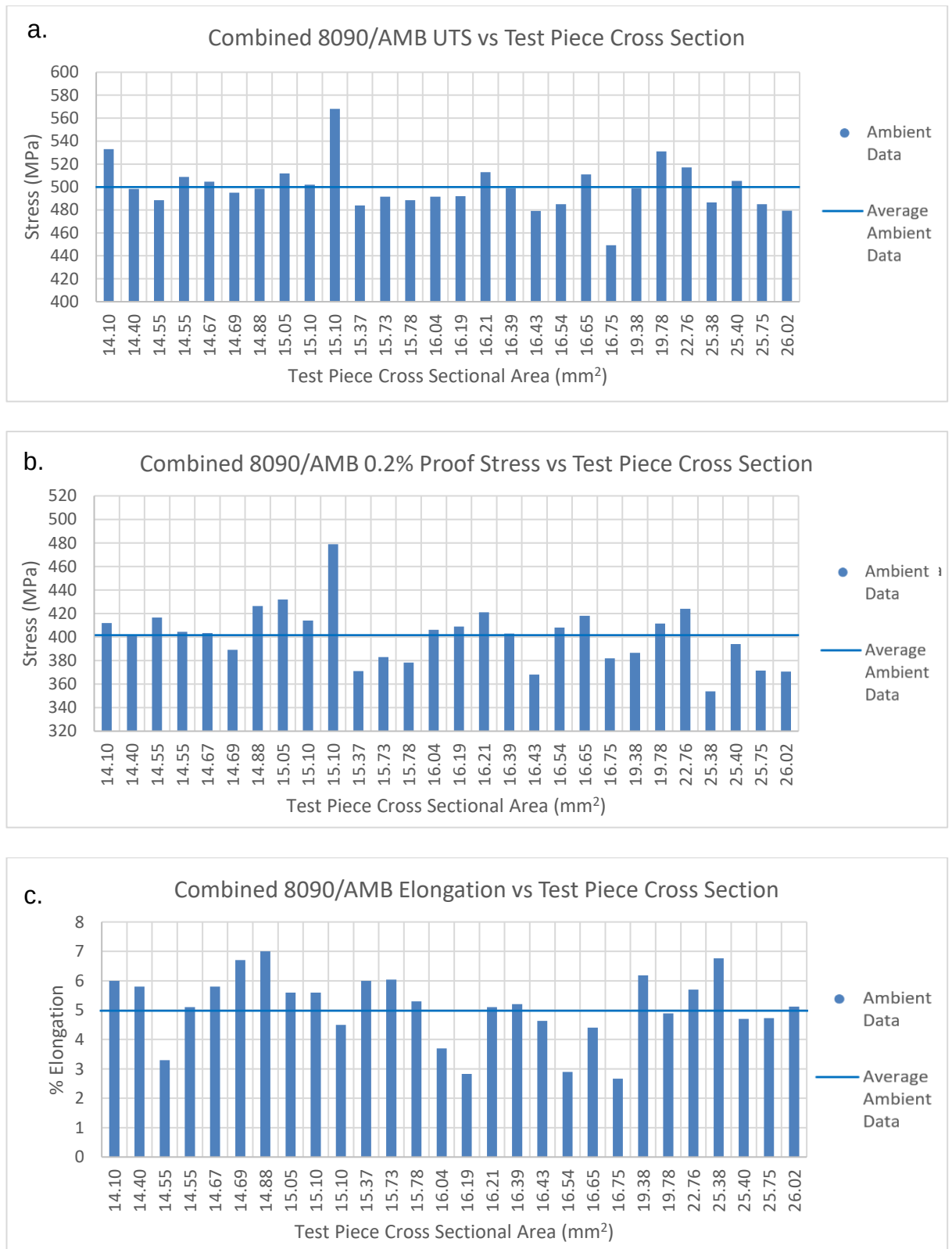


**Figure 43: Showing combined 8090/AMB and Group H tensile data arranged by cross-sectional area, a) UTS, b) 0.2% proof stress, c) elongation.**

As can be observed in the graphs in Figure 43, the results show no correlation between test piece scatter and cross-sectional area, nor do the additional preparation steps reduce the scatter of results in comparison with the as-machined samples. This therefore indicates that the scatter is related to the microstructure of the material and is not affected by the machining and preparation for the testing process.

#### **4.2.9 Tensile testing results for Group C (1500 hours at 53.1°C).**

With the indication that a metastable effect may be taking place during the 1000 hour heating process, the testing for Group C was designed to determine the effect of a further extended time period at 53.1°C on the mechanical properties of the main lift frames. This was deemed to be a period of 0 to 1512 hours (0 to 9 weeks). This would fulfil two requirements: determine whether the findings of Group A were repeatable and determine whether extended exposure at 53.1°C had any further effect on the mechanical properties. To achieve this, six test pieces were prepared, placed in the oven and were removed for testing at intervals.

The tensile test data is presented in Figure 44. As can be seen there is relatively little change in the UTS with a 12.1 MPa / 2.3 % average increase over the average 8090/AMB data with all of the test pieces, (bar the final test C2-9 just falling within the error range). A more notable increase was measured for the average 0.2% proof stress value resulting in a 40.8 MPa / 9.4% increase against average 8090/AMB data in Figure 43.

The elongation results gave a significant reduction in elongation from the average 8090/AMB results with a 2.2% elongation value reduction which equates to a 43% overall reduction. It must be noted that a substantial increase in elongation was detected in sample C2-8 (1176 hours), but it appears to have been an anomalous result – there was no significant alteration in the UTS or 0.2% proof stress results for the same sample. However, the remainder of the results did not reveal a high degree of scatter and remained within the error margin (especially when compared with the 8090/AMB elongation results).

Comparison of the Group C elongation results with data from the other test Groups is shown in Figure 45 and Figure 46. As can be seen in Figure 46, the average elongation values for Group A, Group C and the 80.0°C tests are within 0.4% of each other. These findings indicate that a consistent repeatability of the reduction in elongation has been achieved across a selection of main lift frames. Furthermore, the correlation of the 0 hour piece in Group C (C2-2) and the 8090/AMB data results indicated that the significant reduction in the elongation properties of the Al-Li material (at continual exposure to 53.1°C) occurred within the 0 to 72 hour period.



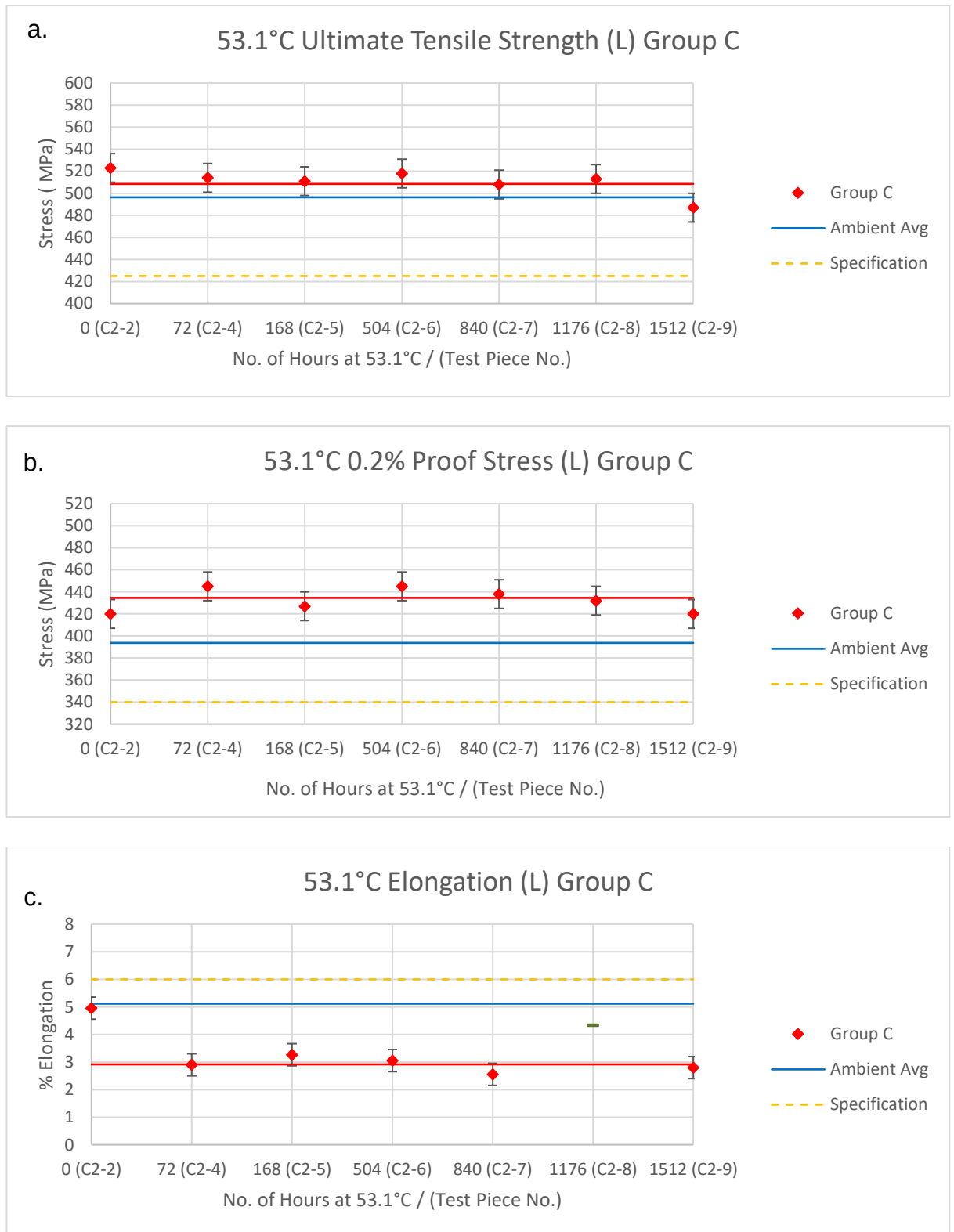


**Figure 44:** Showing the tensile test results for Group C (up to 1512 hours/ 9 weeks soak) at 53.1°C, a) UTS, b) 0.2% proof stress, c) elongation. The yellow dotted line represents the minimum specification requirement.

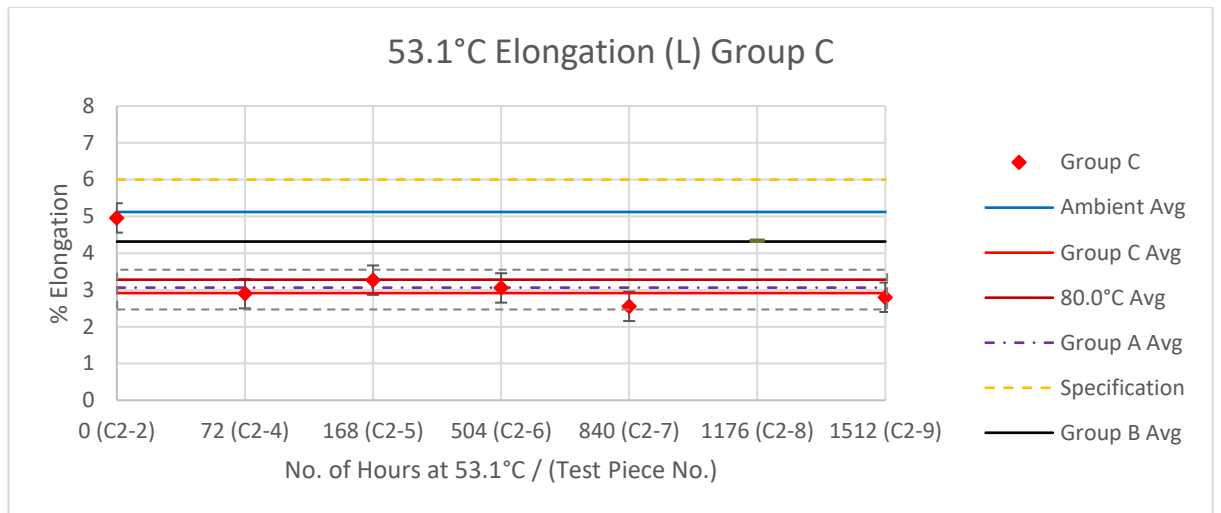


**Figure 45:** Showing the average data results for Group C with the results from previous groups. The yellow dotted line represents the minimum specification. The dotted grey box is the area shown in Figure 46.

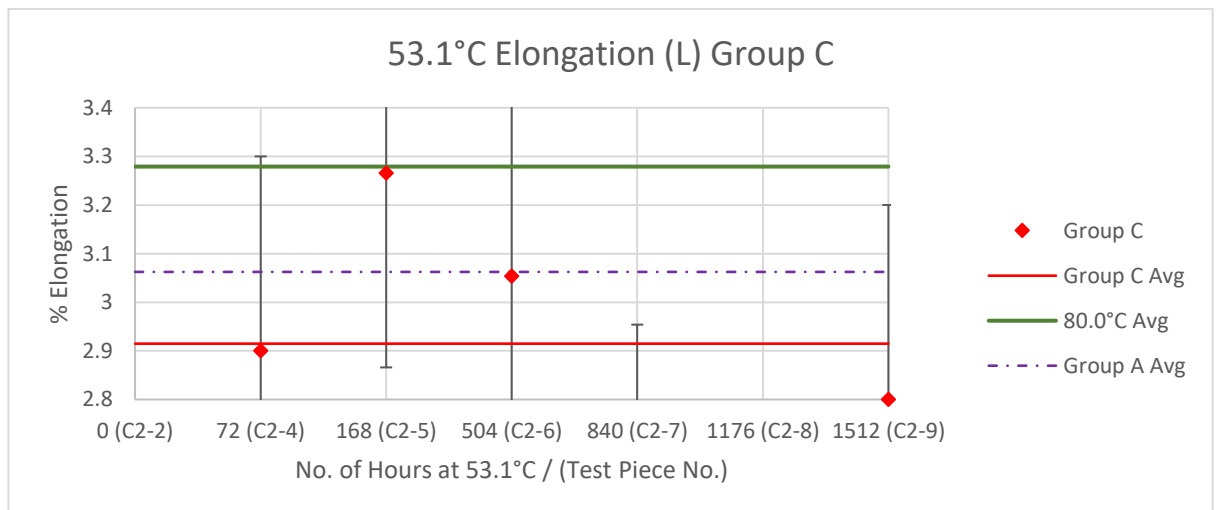


**Figure 46:** Showing an expanded view of the average elongation results for Group C, Group A and the 80.0°C tests.

**4.2.10 Tensile testing results for Group D (1500 hours at 53.1°C and 1500 hours at ambient temperature).**

With Group C determining the mechanical properties from 0 to 9 weeks at 53.1°C, Group D was designed to provide data to help understand what happens to the Al-Li material when left for extended periods of time at ambient temperature, following the same Group C 1512 hour 53.1°C heating conditions. Group D was run alongside Group C at the same time, the samples were removed from the oven at the same time as the last Group C sample (C2-9) and were left at ambient temperature for a period of 9 weeks. The test pieces were subjected to tensile testing at weekly intervals over the 9 week period. The results for Group D are not shown as they do not provide any additional benefit to the work in this investigation, except to demonstrate that no further changes in mechanical properties were recorded after nine weeks of ambient temperature exposure.

**4.2.11 Tensile testing results for Group E (72 hours at 53.1°C).**

As previously mentioned in Section 4.2.9, the Group C testing results indicated that the change in mechanical properties occurred within the first 72 hours of exposure. Therefore, to gain a better understanding of the effects of exposure at 53.1°C on the mechanical properties of the in-service material within this period, Groups E1 and E2 (L and LT orientations respectively) were soaked at 53.1°C for between 1 and 72 hours. Tensile tests were undertaken at 12 hour intervals. The results of which are shown in Figure 47. As can be seen in Figure 47, the results for the UTS values indicate that there is a fluctuation in mechanical properties, with an increase after 12 hours exposure to 53.1°C, a

reduction after 36 hours and a subsequent recovery after 60 hours. The primary finding from the testing was that during the 72 hour period, there was a variable effect on the UTS value of Al-Li lift frame material. The 0.2% proof stress results for Group E1 gave a wide variation of results, with the 0 hours (F2-1) test piece giving a 37 MPa increase over the 8090/AMB testing data and the resultant tensile test pieces gave a continually decreasing 0.2% proof stress result from 0 to 72 hours.

The Group E1 elongation results revealed interesting phenomena, with an initial increase in elongation for a 12 hour exposure period, followed by a reduction after 24 and 36 hours exposure, and a significant increase after 48 hours exposure to 53.1°C. The elongation result after the 48 hours is far superior to the specification requirement of 6% and the highest recorded value for any test piece so far during this work. Post the 48 hour exposure, the elongation steadily decreases, meeting the specification requirement after 60 hours and continuing to decrease to a value consistent with the Group C test after 72 hours of exposure.

#### **4.2.12 Comparison of Group E1 and E2 tensile test results.**

To determine whether there was any notable correlation between the mechanical property behaviour for the L and LT orientations, the LT orientation (Group E2) was subjected to the same tensile testing regime. The results for Groups E1 and E2 (L and LT respectively) can be seen in Figure 48. For UTS, after 12 hours both orientations gave an increase in strength, however the Group E2 test piece at 12 hours gave a significant increase of 73 MPa against

a 12 MPa increase for the L orientation. Post 12 hours, both orientations gave a decrease in strength, albeit with an accelerated decrease for the LT orientation, with the minimum UTS value recorded at around 36 hours, giving a 44 and 119 MPa decrease from the peak 12 hour result for the (L and LT orientations respectively). From 36 to 60 hours, an increase in strength is recorded for both orientations, with a significant recovery for the LT orientation (108 MPa) and a slower increase for the L orientation (30 MPa). After 72 hours of exposure to 53.1°C, the LT test piece had increased mechanical properties and the L test piece had decreased, reporting a 21 MPa less result than the 8090/AMB average value.

The 0.2% proof strength increased for the LT orientation after 12 hours and matched and L orientation which decreased after 12 hours. From 12 to 48 hours of exposure there was minimal difference (1 MPa) between the E1 and E2 results. Once again post 12 hours of aging, there was a steady decrease in 0.2% proof stress. However, the minimum value in the decrease was recorded after 48 hours exposure, not after 36 hours exposure (as was the case for the L orientation). There was a decrease of 49 and 47<sup>xxxii</sup> MPa over the 12 – 48 hour period for the L and LT orientations, respectively.

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<sup>xxxii</sup> Please note that the Group E2 LT sample F2-10 did not give a 0.2% proof stress value and the point on the graph has been calculated to provide an indication of the trend. The premature failure of the test piece is also likely to have affected the elongation result, recording a lower value than is likely to have occurred. These factors need to be kept in mind when looking at the results.

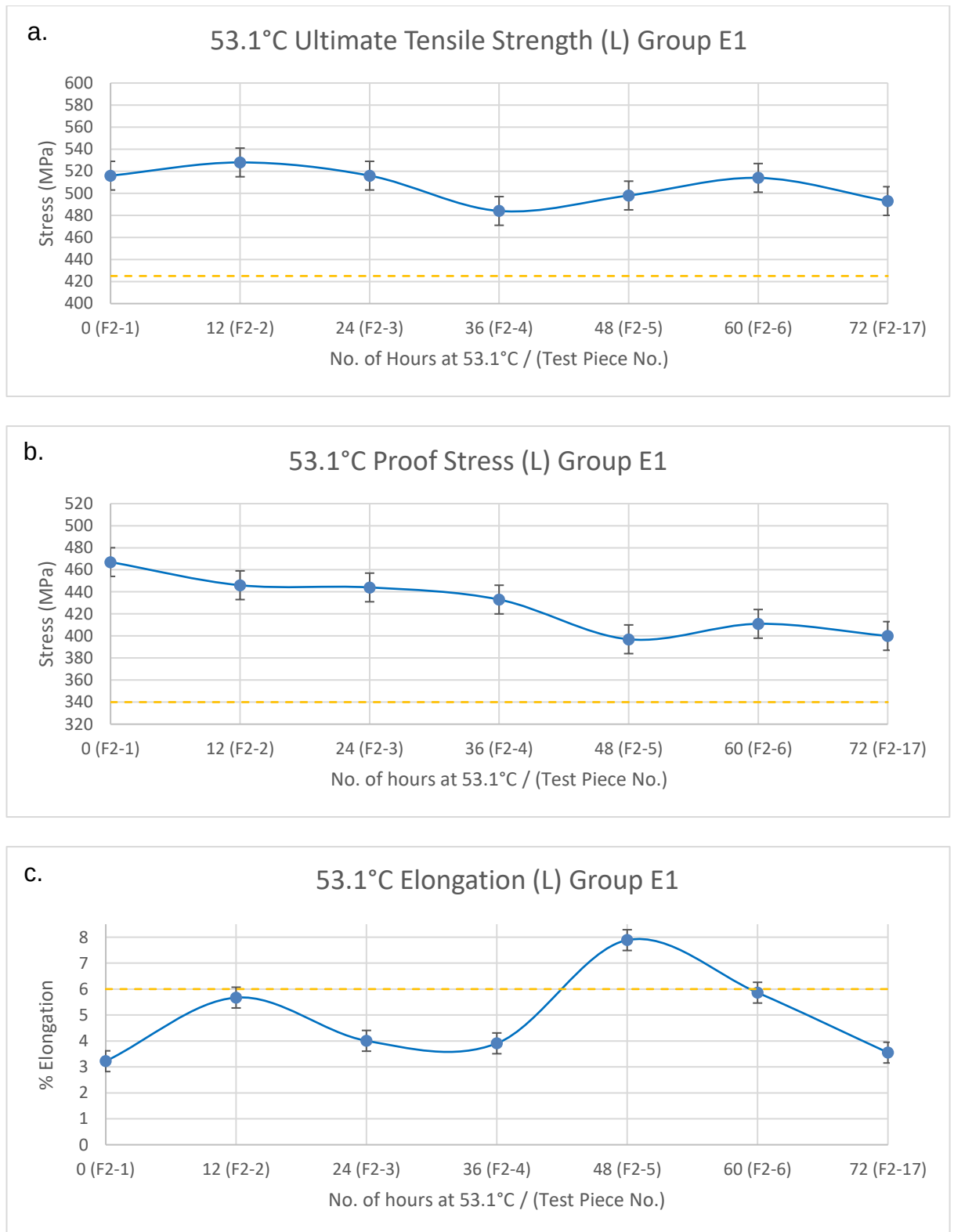


**Figure 47: Showing the tensile test results for Group E (between 1 and 72 hours / 12 hour intervals) at 53.1°C, a) UTS, b) 0.2% proof stress, c) elongation. The yellow dotted line represents the minimum specification requirement.**

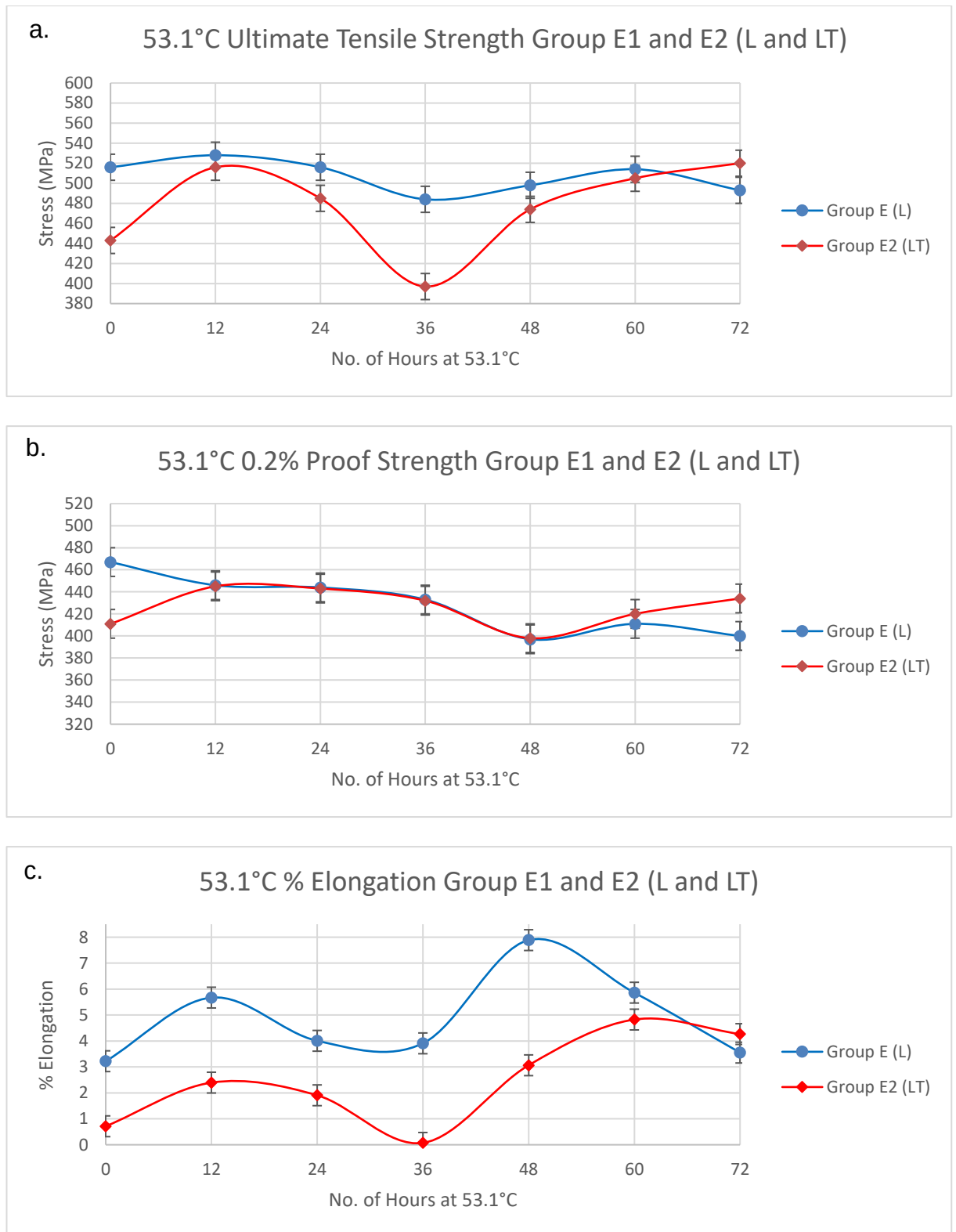


**Figure 48:** Showing the tensile test results for Groups E and E2 (between 1 and 72 hours / 12 hour intervals) at 53.1°C. (Note the Group E2, 36 hour proof strength value has been calculated).  
a) UTS, b) 0.2% proof stress, c) elongation.

From 48 – 72 hours exposure, both orientations initially gave an increase in UTS and 0.2% proof stress, with the LT maintaining the trend to 72 hours and the L decreasing for both UTS and 0.2% proof stress.

The elongation results for both orientations followed visually similar trends from 0 – 48 hours exposure, with a notable peak after 12 hours (as was noted in the UTS for both the L and LT and the L 0.2% proof strength), followed by a decrease from 12 – 36 hours. A second increase in elongation occurs between 36 to 48 hours, with an almost 4% increase for the L orientation and a 3% increase for the LT. The LT results continues to increase to 4.7% after 60 hours aging whereas the L began to decrease. Both orientations gave a decrease in elongation after 60 – 72 hours exposure.

For the 72 hours 53.1°C testing period after an initial 12 hour soak, the Group E1 and E2 test pieces (representing both the L and LT orientations) exhibited a similar pattern of behaviour for their respective mechanical properties. Five of the six test pieces exhibited an initial increase during the 0 – 12 hour exposure period. This initial peak was followed by a decrease for all three property groups for at least 24 hours of exposure with the maximum reduction for UTS and elongation occurring after 36 hours of aging and the 0.2% proof strength after 48 hours. Each reduction was followed by a subsequent re-increase within 12 hours of aging (after 60 hours), but whereas the L orientation peaks and again decreases, the LT samples exhibited a continuous



steady increase up to 72 hours for UTS and 0.2% proof stress, although it should be noted that the elongation sample recorded a small 0.5% reduction.

#### **4.2.13 Tensile testing results for Group F1 and F2 (72 hours at 53.1°C).**

Group F testing was a repeat of Group E (72 hours at 53.1°C with 12 hour testing intervals). As can be seen in Figure 49, there is reduced repeatability between the L (Group F1) and LT (Group F2) orientations when compared against Groups E1 and E2 (Figure 48). Whilst Group F1 was primarily conducted to increase the data set for the 0-72 hour exposure conditions, in contrast to Group E, the Group F1 and F2 test pieces were soaked and tested at separate times<sup>xxxiii</sup> to determine whether a similar pattern would continue to occur.

The UTS results for Group F1 followed a similar pattern to Group E1 with the UTS rising to a maximum peak after 24 hours, a similar peak also occurs after 24 hours with the 0.2% proof stress, but the 0 hours sample gave an increased initial 0.2% proof stress (similar in behaviour to the Group E1 0 hours result), followed by a reduction after 12 hours. The peak therefore occurred after 24 hours exposure (but this value was less than what was reported by the 0 hours value). Both the UTS and 0.2% proof stress give a decrease in properties after 36 hours aging, a reduction of 26 and 61 MPa, respectively.

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<sup>xxxiii</sup> *Group F - 24<sup>th</sup> Jul to 27<sup>th</sup> Jul 17 and Group F2 - 29 Aug to 1<sup>st</sup> Sep 17.*

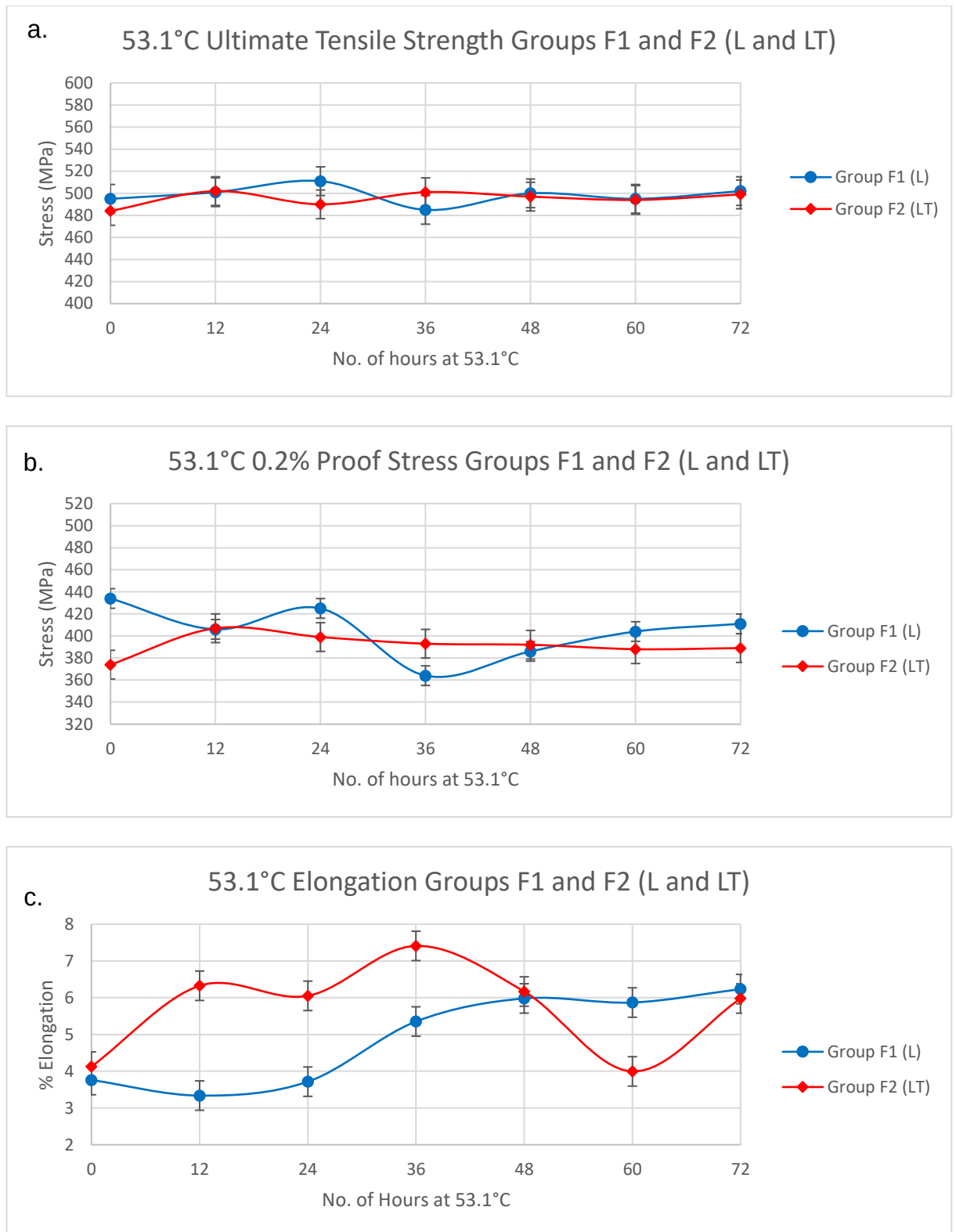


**Figure 49: Showing the comparative tensile test results for Groups F and F2 (72 hours / 12 hour intervals) at 53.1°C, undertaken at different times, a) UTS, b) 0.2% proof stress, c) elongation.**

Both mechanical properties are followed by an increase after 48 hours aging, which continues to a gradual overall increase by after 72 hours (noting the slight dip in UTS after 60 hours). The elongation for Group F1 mirrors the behaviour of the UTS and 0.2% proof stress, after 24 hours aging the elongation was reduced (whereas the UTS and 0.2% proof strength increase) and after 36 hours, the elongation increases to its maximum value, where the UTS and 0.2% proof stress show the maximum reduction.

Comparison between the mechanical property results for the same grain orientation began to show similar pattern behaviour between the testing groups. As shown in Figure 50, the UTS results for Groups E1 and F1 (L orientation) show striking similarity over the 72 hour aging period. Both Groups gave an increase in UTS after 24 hours aging and a significant reduction after 36 hours, with Group E1 reporting 484 MPa and Group F1 reporting 485 MPa, respectively. This reduction was followed by an increase after 48 hours exposure (for both groups). For 48 to 72 hours Group E1 continued to increase in UTS (as shown after 60 hours exposure) whereas Group F1 began to plateau, with a slight reduction after 60 hours. After 72 hours, Group E1 gave another reduction and Group F1 a slight increase, with a difference of 9 MPa between the two Groups at the end of the 72 hour testing regime. The 0.2% proof stress and elongation results for Groups E1 and F1 gave interesting results which show similar behavioural patterns, although the Group E1 reduction in 0.2% proof stress and maximum increase in elongation occurred after 48 hours aging.

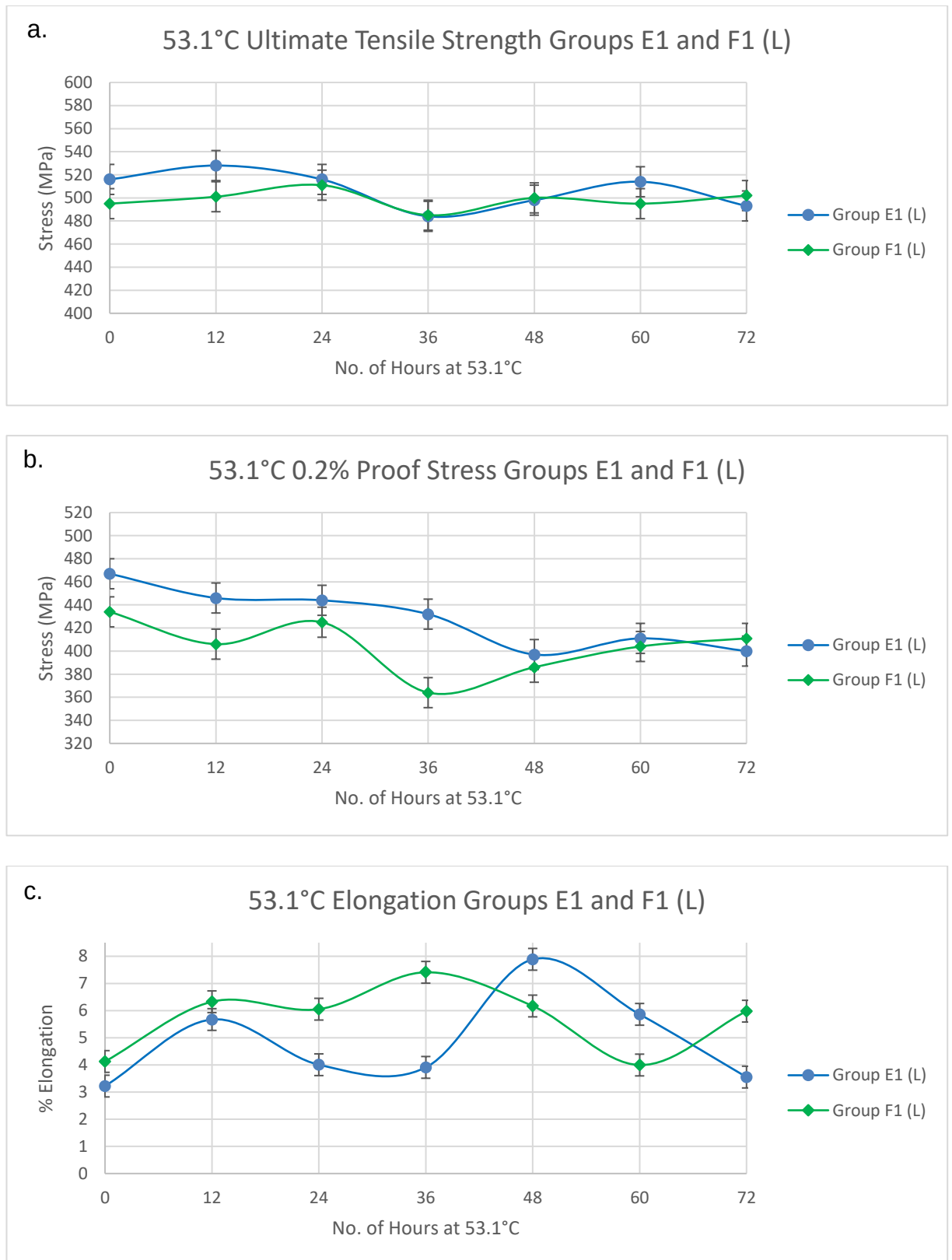


**Figure 50: Showing the tensile test results for Groups F1 and E1 (between 1 and 72 hours / 12 hour intervals) at 53.1°C.**

**4.2.14 Tensile test results for Group G1 and G2 (72 hours at 53.1°C).**

Groups G1 (L) and G2 (LT) testing (once again, to the same criteria as Group E; 72 hours at 53.1°C with 12 hour testing intervals), was undertaken at the same time as Group F2. Groups G1 and G2 were primarily submitted for testing to increase the initial data set to determine whether there was a repeatable correlation with the previously recorded results. As can be seen in Figure 51, the Group G1 (L) results, follow a pattern of increasing strength (0-24 hours for UTS and 12-24 hours for 0.2% proof stress), followed by a reduction in both UTS and 0.2% proof stress after 36 hours, with a partial recovery in UTS and 0.2% proof stress after 48 hours.

The elongation results for the Group G1 (L) tensile test pieces, follows in a opposite pattern to this, showing a decrease in elongation where the UTS increases (after 24 hours and 48 hours), and an increase in elongation where the UTS decreases (after 36 hours) as would be expected. The Group G2 (LT) tensile test group gave UTS results that were comparable to the Group G1 (L) UTS results, with the same increase in strength (from 0-24 hours) followed by a decrease in strength (after 36 hours) and partial recovery (after 48 hours).

The 0.2% proof stress and elongation results gave contradictory data, with the Group G2 (LT) elongation following a similar pattern to the UTS and the 0.2% proof stress results giving an opposite pattern to the UTS results.

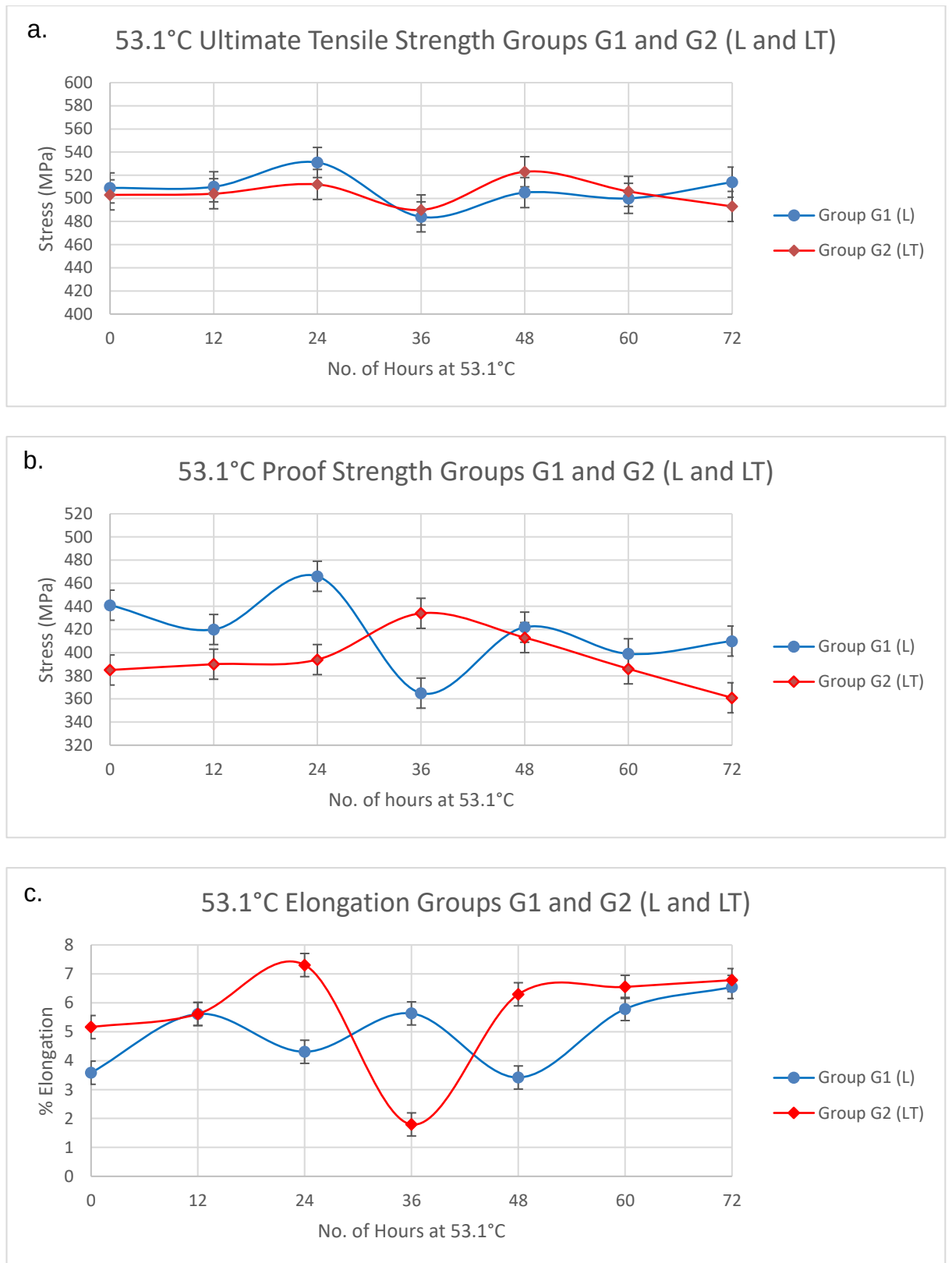


**Figure 51: Showing the tensile test results for Groups G1 and G2 (between 1 and 72 hours / 12 hour intervals) at 53.1°C.**

### 4.3 Summary of low temperature exposure tensile testing results.

To recap the work undertaken within this section, the following Groups of tensile tests have been undertaken:

- 1) 80.0°C soaking for a 1000 hour period (longitudinal orientation).
- 2) 53.1°C soaking for a 1000 hour period (Group A) (longitudinal orientation).
- 3) 53.1°C soaking for a 1000 hour period, with an additional 1000 hours at ambient temperature (Group B) (longitudinal orientation).
- 4) 53.1°C soaking for a 1500 hour period (Group C) (longitudinal orientation).
- 5) 53.1°C soaking for a 1500 hour period, with an additional 1500 hours at ambient temperature (Group D) (longitudinal orientation).
- 6) 53.1°C soaking for a 72 hour period (Group E1) (longitudinal orientation).
- 7) 53.1°C soaking for a 72 hour period (Group E2) (long transverse orientation).
- 8) 53.1°C soaking for a 72 hour period (Group F1) (longitudinal orientation).
- 9) 53.1°C soaking for a 72 hour period (Group F2) (long transverse orientation).
- 10) 53.1°C soaking for a 72 hour period (Group G1) (longitudinal orientation).

- 11) 53.1°C soaking for a 72 hour period (Group G2) (long transverse orientation).

The testing schedule was developed as a dynamic response to the results that were recorded in previous testing groups. The primary findings are as follows:

- i) The experimental results for tensile test group exposed to 80.0°C for 1000 hours, (Figure 38), gave an average increase of 9.6 MPa for UTS, an increase of 28.3 MPa for 0.2% proof stress and a reduction of 1.8% for elongation when compared to the 8090/AMB tensile test results. This equates to an increase of 1.9% for UTS, a 7.2% increase for 0.2% proof stress and a 36.1% reduction in elongation.
- ii) The experimental results for the tensile test group exposed to 53.1°C for 1000 hours, (Group A, Figure 39), gave an average increase of 40.1 MPa for UTS, an increase of 64.6 MPa for 0.2% proof stress and a reduction of 2.1% for elongation when compared to the 8090/AMB tensile test results. This equates to an increase of 8.1% for UTS, a 16.4% increase for 0.2% proof stress and a 40.3% reduction in elongation.
- iii) The tensile test results for Group B (1000 hours at 53.1°C and 1000 hours at ambient temperature, Figure 40), gave an average



increase of 24.2 MPa for UTS, an increase of 41.7 MPa for 0.2% proof stress and a reduction of 0.8% for elongation when compared to the 8090/AMB tensile test results. This equates to an increase of 4.9% for UTS, a 10.6% increase for 0.2% proof stress and a 15.8% reduction in elongation.

- iv) The Group B average overall elongation value was higher than the Group A results, but lower than the 8090/AMB elongation results. As Groups A and B were heat treated at the same time, the only difference between the sample sets was the 1000 hours at ambient temperature following soaking at 53.1°C. This data therefore indicates that the metastable nature and 'recovery' or 'reversion' of mechanical properties as observed by Pitcher (1993)<sup>81</sup> also occurred in the Group B test pieces.
- v) The tensile test results for Group C (1512 hours at 53.1°C, Figure 44), gave an average increase of 12.1 MPa for UTS, an increase of 40.8 MPa for 0.2% proof stress and a reduction of 2.2% for elongation when compared to the 8090/AMB tensile test results. This equates to an increase of 2.4% for UTS, a 10.4% increase for 0.2% proof stress and a 43.2% reduction in elongation.

- vi) Test pieces exposed to 1512 hours at 53.1°C that were sampled at intervals in this time period (Group C) gave relatively little change in mechanical properties over time for UTS and 0.2% proof stress.
- vii) The same testing regime (1512 hours at 53.1°C), indicated that the reduction in elongation occurred within the initial 72 hour time period.
- viii) Comparison of 80.0°C, Group A and Group C tensile test results revealed that the reduction in elongation was repeatable with an average reduction (by comparison with 8090/AMB results) of 36%, 40% and 43% for the 80°C, Group A and Group C, respectively.
- ix) 72 hour testing of both L and LT orientations (Groups E1 and E2) indicated that a pattern of increasing strength (0-24 hours), followed by a sharp decrease in strength (after 36 hours) followed by a partial recovery (48-72 hours) occurred for all three mechanical properties.

#### **4.4 Hardness testing of Al-Li 8090 main lift frame material.**

As a result of the change in mechanical property findings from the tensile testing results in Section 4, a short hardness testing regime was undertaken to investigate whether the hardness value of the Al-Li main lift frame material changed between the 0, 24, 36 and 48 hour periods of exposure to 53.1°C. This testing was undertaken to determine whether the reported changes in strength were reflected with a change in hardness in the material.

A selection of main lift frame material consisting of two tensile test pieces from Frame C2 (C2-7 and C2-10) and a section cut from Frame C2 (C2-Section), were exposed to 53.1°C at tested at 0, 12, 24, 36 and 48 hour intervals with a calibrated Struers Durascan hardness tester, using a Vickers indenter at HV10. There was minimal time between removal of the samples from the oven and the beginning of the hardness testing. The automated hardness testing programme ran for approximately 30 minutes for each set of indents. A graph of the hardness results is shown in Figure 52 and the data tables are given in Annex D.

As can be seen in Figure 52, there was minimal change in hardness recorded for all three samples between 1 and 48 hours exposure to 53.1°C. The maximum variation in HV for the samples was 8 HV for sample C2-7, 9 HV for sample C2-10 and 10 HV for sample C2-Section.

Conversion of the hardness values to show any change in the tensile strength could not be achieved. The reason for this was that the majority of the hardness results did not alter the tensile strength conversion as there was a minimum of a 3HV difference required<sup>111</sup>. Furthermore, these reported figures were within the error margin of  $\pm 4.6\text{HV}$  for the indenter and calibration block that was used to calibrate the hardness tester. These findings therefore indicate that the reported change in mechanical properties as a result of exposure to  $53.1^\circ\text{C}$  did not have any discernable effect on the hardness value of the Al-Li main lift frame material when exposed to  $53.1^\circ\text{C}$  for 0, 12, 24, 36 and 48 hours.

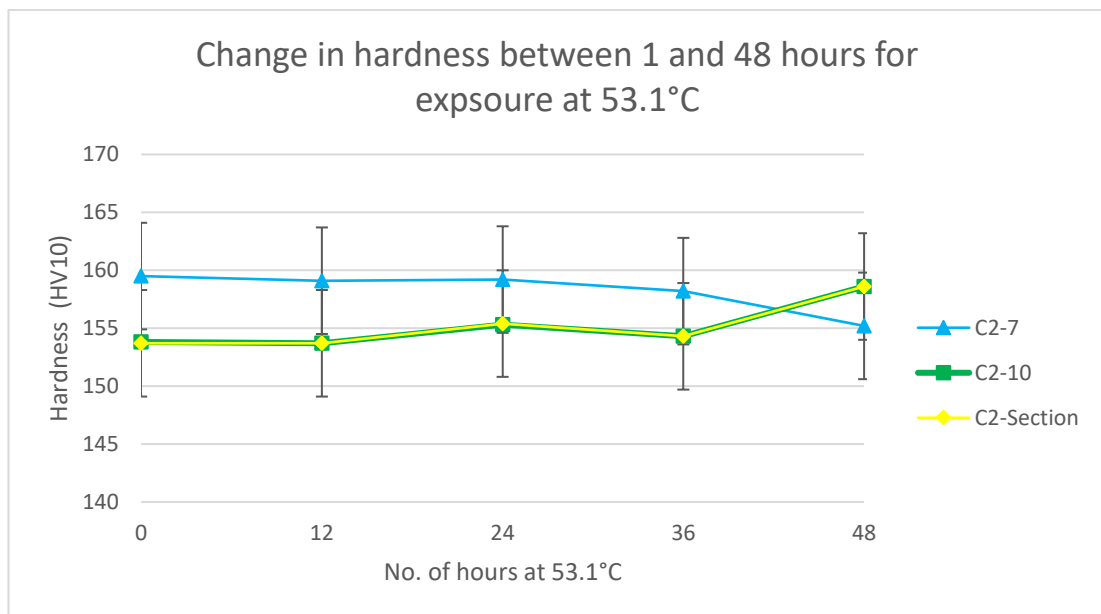


**Figure 52:** Showing the hardness results for samples C2-7, C2-10 and C2-Section between 1 and 48 hours exposure to  $53.1^\circ\text{C}$ .

## **5 Transmission electron microscopy investigation of Al-Li main lift frame material.**

### **5.1 Baseline investigation of the as-received material and development of the sample preparation technique.**

As is evident in Chapter 4, the effect of low temperature exposure on the mechanical properties of Al-Li 8090 is notable over the initial 0 to 72 hour exposure period. Therefore, in an attempt to further understand the mechanism that was causing the changes in mechanical properties, a detailed microstructural examination was undertaken using Transmission Electron Microscopy (TEM) to investigate main lift frame material that had been exposed to both ambient and 'low' temperatures. The primary aim of this was to initially identify and characterise the main strengthening precipitate ( $\delta'$ ) in the 8090/AMB material i.e. that had only been exposed to ambient temperature conditions. This characterisation was then to be compared with precipitates present in the same material that had been exposed to 53.1°C for 36 hours. The heat treatment temperature and time was selected because the tensile test data had shown this time gave the most notable reduction in mechanical properties within the 72 hour exposure period.

#### **5.1.1 TEM sample preparation and transportation.**

TEM samples are required to be very thin foils, free of oxidation, etch pitting and any other contamination. TEM samples can be manufactured by a variety of means, but the decision was taken to undertake sample preparation using the electrolytic thinning process to ensure there was as little mechanical/thermal deformation as possible. To allow for transmission of the

electron beam through the specimen, the ‘useful’ sections of a TEM specimen are in the region of tens of nanometres thick. To obtain such extremely thin cross sections, multiple stages of preparation are required, multiple techniques can be used, and these techniques vary across different materials and scientific disciplines (materials science, biology, etc).

The first stage of the process was the creation of the sample to be thinned. For this project, the initial objective was to create a 3 mm diameter bar from a section of Al-Li 8090. The reason for this is that TEM specimens are 3 mm in diameter since the majority of equipment (samples holder, etc) is designed around this dimension. The 3 mm bar was turned in the 1710 NAS workshops<sup>xxxiv</sup>. To create the thin ‘discs’ the bar was sectioned using a Struers Accutom-5. This machine is capable of undertaking multiple cuts at varying thickness with a positioning accuracy of 5  $\mu\text{m}$  (reportedly)<sup>112</sup>. However, the Al-Li material was noted to regularly spall during cutting at thicknesses below 100  $\mu\text{m}$  (resulting in lost test pieces) and the successful sections did not exhibit a uniform thickness, which became an issue for further preparation techniques.

A second technique was undertaken using a ‘punching method’ in the Struers Tenupol-5 electrolytic thinning equipment. Whilst the equipment is predominantly used for the second stage of test piece manufacture, the

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<sup>xxxiv</sup> *Although turning a bar on a lathe would induce stress concentrations at the outer surface of the bar, electrolytic thinning ‘punches’ through the centre of the sample, which in this case, was still representative of the bulk sample material.*

machine can be used in conjunction with acid resistant tape to 'punch' (in reality etch) 3 mm diameter disks from a larger section of material. The punching is a more aggressive version of electrolytic thinning. The results of the punching process did produce a number of samples with an approximate 3 mm diameter, however the process did etch the edges of the samples, resulting in additional material loss and burning (over etching) of the samples. From this process four viable TEM samples were produced and were used in the first batch of TEM analysis at Sheffield Hallam University. Ultimately, the electrolytic 'punching' process was found to be too inconsistent and aggressive to reliably produce samples that would be suitable for TEM analysis. A third approach, again used the Struers Accutom-5 at 1710 NAS, however to minimise the loss of material instead of a 3 mm bar, a block of Al-Li was partially sectioned with multiple cuts. The result was similar in appearance to cooling fins on a heat exchanger from which thin sheets of material could be taken when required.

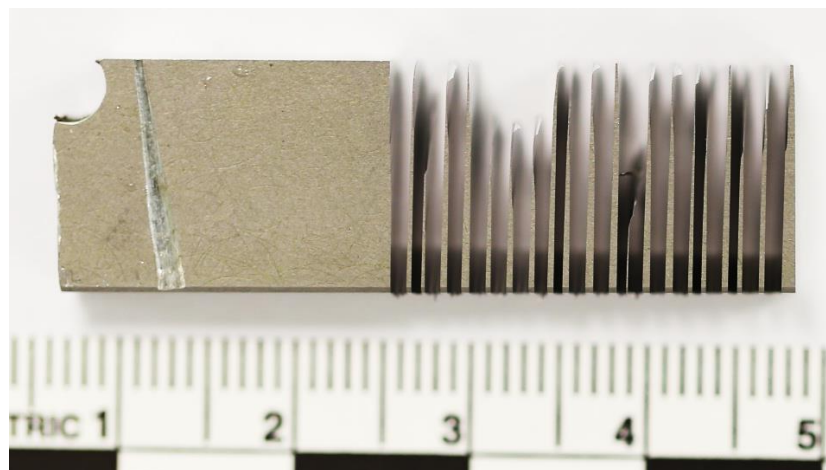


**Figure 53:** Showing the sliced 'heat exchanger' section of Al-Li main lift frame material.

These thin sheets (approximately 100 - 250  $\mu\text{m}$  thick) were then hand ground to a thickness of  $< 100 \mu\text{m}$ . Additional equipment was purchased from Fischione Instruments to assist in the first stages of preparation. Firstly, a Model 130<sup>113</sup> specimen punch was used to produce 3mm diameter specimens from the thin sheets of Al-Li. Secondly, a Model 160 specimen grinder<sup>114</sup> was used with multiple grades of sand paper to uniformly grind the samples to a thickness  $< 100 \mu\text{m}$ . Both instruments are shown in Figure 54.



**Figure 54: Showing (left) the Model 130 punch and (right) Model 160 grinder used for mechanical preparation of the TEM specimens. Images © Fischione Instruments.**

The second phase of specimen preparation was electrolytic thinning (and electropolishing). Electrolytic thinning is undertaken in a dedicated piece of equipment, in this case a Struers TenuPol 5<sup>115</sup> was used. The system causes impingement of a jet of negatively charged electrolyte against a positively charged sample, (the same as an anode in an electrolytic cell) with the resulting material dissolving from the anode into the electrolyte. As the sample progressively thins, a hole will appear in the sample. At this point infrared light, which has been directed at the specimen will be detected on the opposite side and the system cuts the electrical power, stopping the process. An illustration



of the process can be seen in Figure 55. The electro thinning process can be further controlled by changing the voltage of the electrolytic cell (between 1 – 30 volts) and by cooling the electrolyte to reduce the chemical activity – the cooler the electrolyte, the smoother the resultant sample surface.

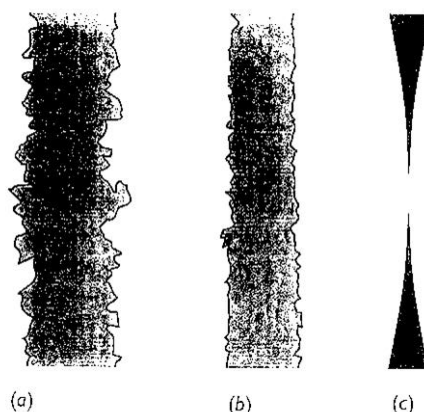


**Figure 55: Showing the effect of electro thinning on a specimen (a) the original rough sample, (b) thinner and smooth and (c) perforated and smooth.**

The electrolyte used for the electrolytic etching carried out in this project was originally a mixture of a Struers solution (Electrolyte A2) and 70% perchloric acid<sup>xxxv</sup>. Initial thinning of samples was undertaken at room temperature, however to minimise oxidation and over etching of the samples (due to heavy etching), the electrolyte was cooled by immersing the electrolyte container in a bath of a liquid nitrogen and methanol to -30°C. In addition, perchloric acid should be cooled for good Health and Safety practice.

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<sup>xxxv</sup> Perchloric Acid is exceptionally hazardous. There are multiple Health and Safety requirements to be met before work with perchloric acid can be undertaken.

This process was used for the first three batches of TEM samples created at 1710 NAS. Examination of each sample batch revealed evidence of: etch pitting, oxidation and an absence of suitably clear, thin sections for analysis. Therefore, the decision was taken to change the preparation process. An electrolyte consisting of a 3:1 ratio of methanol to nitric acid was used at a temperature of -20°C as detailed during work undertaken by NASA (Wang and Shenoy, 1998)<sup>116</sup>. However, a range of voltages were tested to identify the optimal polishing parameters, which were determined as +20V at -20°C with a 'pump value' of 23<sup>xxxvi</sup>.

Due to the distance between the sample preparation location at Her Majesty's Naval Base (HMNB) Portsmouth and the TEM located at Sheffield Hallam University (SHU), being over 200 miles, it became apparent that oxidation of the samples during the transit to the TEM would become an issue (especially as the literature states a maximum of a five minute delay between thinning to putting the sample in the TEM)<sup>117</sup>. Initial transportation attempts using gelatine capsules at room temperature was found to be unsuccessful.

To address this issue, attempts were made to minimise the rate of oxidation of the samples. Firstly, the samples were kept cool within two thermally insulating containers (polystyrene boxes) with additional cold blocks contained within.

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<sup>xxxvi</sup> Which is a unique scale on the Struers Tenupol-5 equipment, therefore no units are given.

Furthermore, each sample was immersed in heptane with a very low water content to act as a further barrier against oxidation. Samples were held in freezers at both 1710 NAS and SHU when they were not being investigated. This approach was found to be successful and the samples were in a suitable condition for examination in the TEM at SHU.

### 5.1.2 TEM calibration.

The Transmission Electron Microscope at Sheffield Hallam University is a FEI/Phillips CM 20 model with a LaB6 emitter and a maximum accelerating voltage of 200kV. The system is capable of enabling the taking of photographic plates and is also fitted with a digital camera for digital imaging and measurement. Calibration of the camera constant relevant to the TEM was undertaken by measuring the electron diffraction ‘rings’ of a polycrystalline thalious chloride standard. Using the camera constant equation (derived from Bragg’s law using small angle approximation):

$$\lambda L = d_{hkl} r_{hkl} \quad (1)$$

Where:

- $\lambda$  = wavelength of the TEM accelerating voltage (nm).
- $L$  = camera length (mm) which is the distance between the sample and the camera film/screen.
- $d_{hkl}$  = interplanar spacing (nm).
- $r_{hkl}$  = the diffraction ring radius measured from a polycrystalline sample, or the distance between the reflection attributable to the transmitted beam and the reflection of interest on a selected area diffraction pattern (SADP) of a single crystal (mm).

Calculation of the accelerating voltage (using DeBroglie's equation – see Annex E for further information):

$$\lambda = \sqrt{\frac{1.5}{V}} \text{ nm} \quad (2)$$

With an accelerating voltage of 200kV:

$$\lambda = \sqrt{\frac{1.5}{200 * 10^3}} \text{ nm}$$

$$\lambda = 0.0027 \text{ nm}$$

However, the velocity of electrons is close enough to the speed of light so relativistic corrections are required:

$$\lambda = \left[ \frac{1.5}{((200 * 10^3) + (10^{-6}) * ((200 * 10^3)^2))} \right]^{1/2} \text{ nm}$$

$$\lambda = 0.0025 \text{ nm}$$

A table of calculated accelerating voltages and wavelengths can be found in Annex E. Using the polycrystalline thallus chloride standard, the diameter of a number of rings were measured. The diffraction pattern image (Figure 56) was taken at a camera length of 70 mm, this was determined by the software algorithms for data inputs by SHU and is recorded on the image.

The scale bar on the image was measured using a steel ruler to be 30 mm which using ImageJ software, gave a digital scale of 4.9111 pixels/mm. All further measurements were made digitally using ImageJ software. The first ring was measured to be 31 mm in diameter and the lattice spacing for the first thallos chloride ring given in the supplied standard data sheet is 0.384 nm. Therefore, to find the Camera Constant L for 70 mm at 200kV:

$$\begin{aligned}\lambda L &= d_{hkl} r_{hkl} \\ &= 0.384 * \frac{31}{2} \\ &= 5.952 \text{ nm/mm}\end{aligned}\tag{1}$$

And checking this Camera Constant value with the second ring (measured to be 43.7 mm):

$$\begin{aligned}d_{hkl} &= \frac{\lambda L}{r_{hkl}} \\ &= \frac{5.952}{(43.7/2)} \\ &= 0.272 \text{ nm}\end{aligned}\tag{4}$$

Calculations for the remainder of the visible rings in Figure 56 can be seen in Table 14.

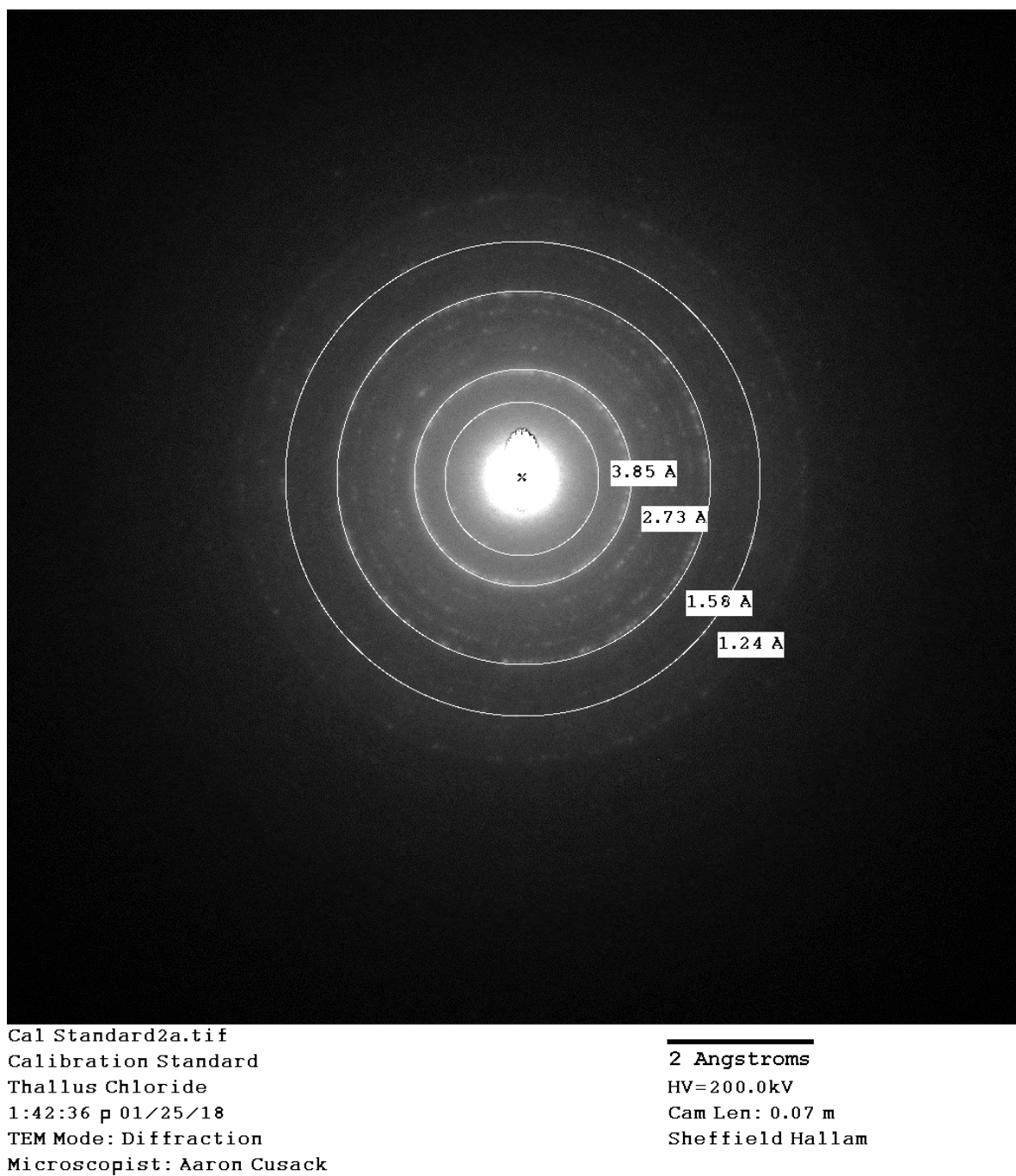


**Figure 56:** Showing a TEM micrograph of the thallous chloride diffraction rings and measurements given using software algorithms at SHU.

**Table 14: Showing the interplanar spacing measurements for the thallos chloride standard (200kV accelerating voltage and 70mm camera length).**

| Evaporated Thallos Chloride |                                      |      |                       |       |                        |
|-----------------------------|--------------------------------------|------|-----------------------|-------|------------------------|
| Ring Number                 | Ring diameter<br>(Given in standard) |      | Measured<br>(by hand) |       | Measured<br>(Software) |
|                             | (nm)                                 | (Å)  | mm                    | (nm)  | (Å)                    |
| 1                           | 0.384                                | 3.84 | 30.7                  | 0.384 | 3.85                   |
| 2                           | 0.272                                | 2.72 | 43.4                  | 0.272 | 2.73                   |
| 3                           | 0.249                                | 2.49 | 53.2                  | 0.222 | -                      |
| 4                           | 0.192                                | 1.92 | 61.6                  | 0.192 | -                      |
| 5                           | 0.172                                | 1.72 | 68.7                  | 0.172 | -                      |
| 6                           | 0.157                                | 1.57 | 75.2                  | 0.157 | 1.58                   |
| 8                           | 0.136                                | 1.36 | -                     | -     | -                      |
| 9                           | 0.128                                | 1.28 | -                     | -     | -                      |
| 10                          | 0.121                                | 1.21 | 96.9                  | 0.122 | 1.24                   |

As can be seen in Table 14, the hand measurements were found to be the most accurate for the determination of interplanar spacing.

### **5.1.3 TEM analysis for characterisation of 8090/AMB main lift frame material.**

As-received ambient temperature (8090/AMB) TEM samples were prepared, stored and transported in accordance with Section 5.1. Examination of the thinned 8090/AMB samples readily revealed a distribution of a spherical precipitates homogenously distributed throughout the bulk of the material, Figure 57a and Figure 57b. Indexing of the matrix of a sample was undertaken with the diffraction pattern shown in Figure 58.

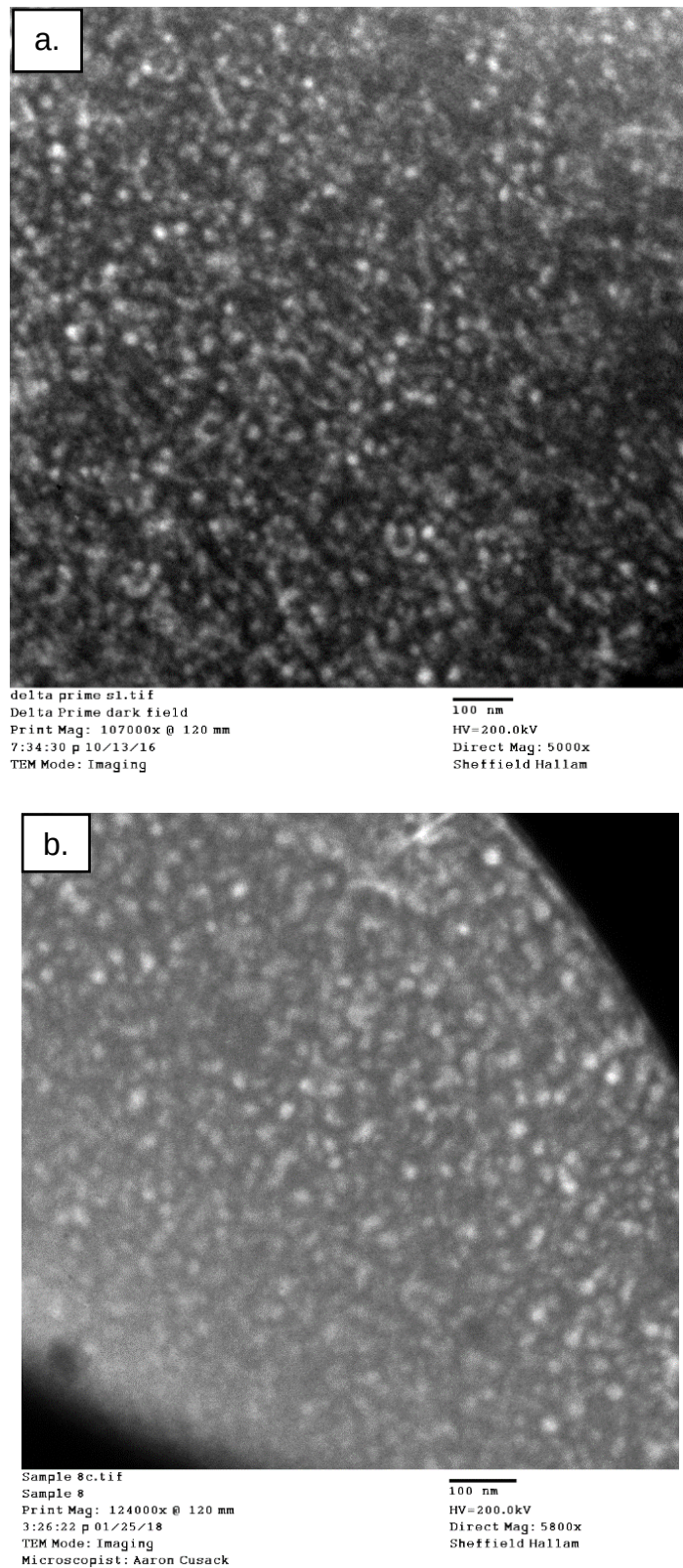


**Figure 57:** HRDF micrographs showing the distribution of a homogenous spherical precipitates in two 8090/AMB samples (a – 2016, b – 2018).



Using a known indexed pattern from established literature for an FCC structure (Williams and Carter, 2009)<sup>118</sup>, (L and M were measured to be 2.594 and 1.816, respectively), enabled the matrix reflections to be indexed, Figure 58. The indices of the reflections are also the indices of the crystal planes that would have been strongly diffracting with the beam travelling in the  $\langle 100 \rangle$  direction with respect to the 8090/AMB foil.

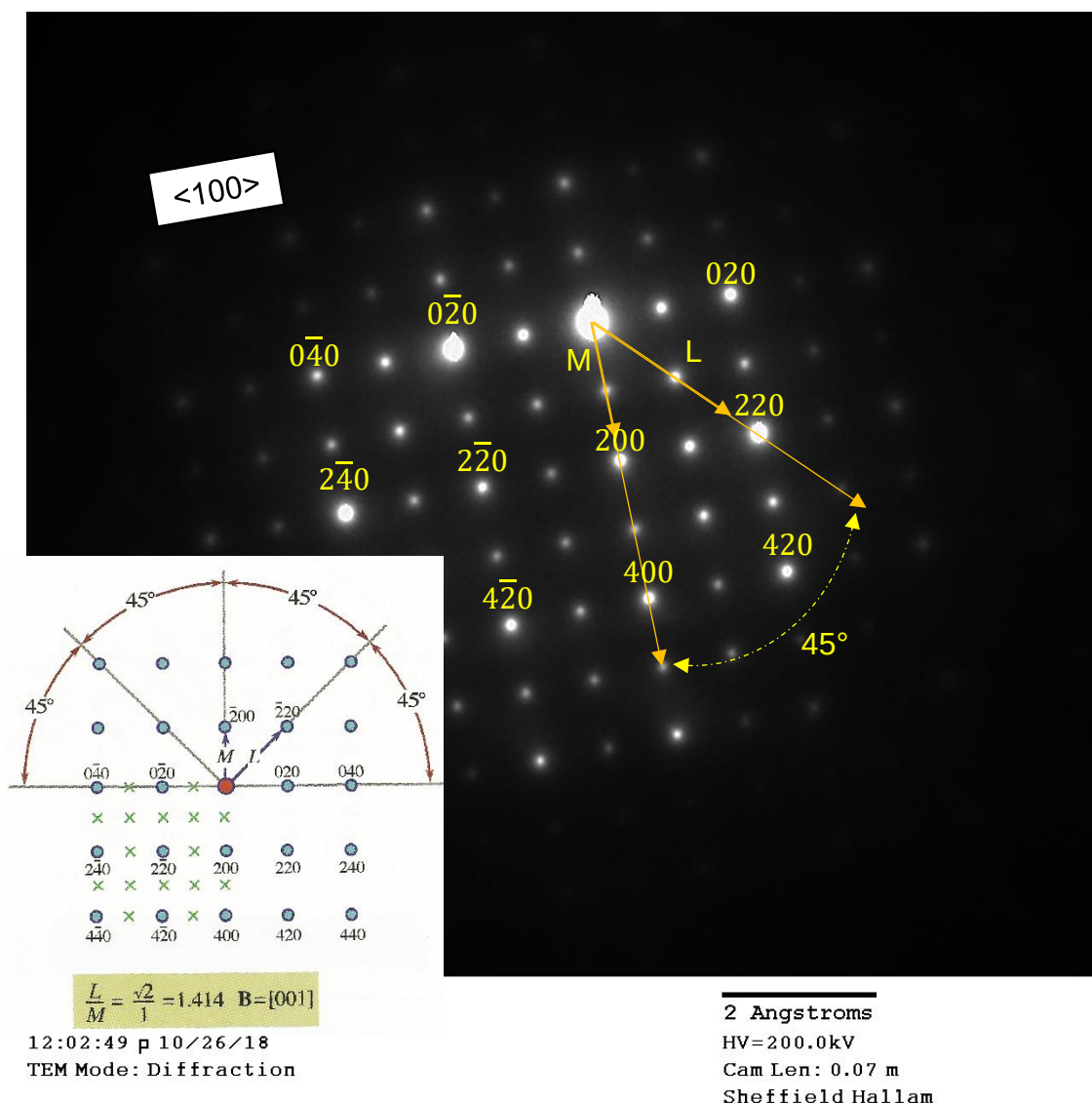


**Figure 58: Indexing the AI matrix using a standard FCC pattern from Williams and Carter (2009)<sup>118</sup>.**

Figure 57a and 56b were obtained using dark field imaging at similar magnification. It should be noted that each image was taken from separate ambient temperature samples (Figure 57a in 2016 and 56b in 2018). Spacing between reflections of interest that could be employed to determine an interplanar spacing measurement was undertaken on a Selected Area Diffraction Pattern (SADP) taken from the specimen shown in Figure 57b, which was measured to be 27.5 mm (yellow line, Figure 59). The following calculation was used to determine the interplanar spacing for a cubic crystal (Stanford University Lecture)<sup>119</sup>:

$$\frac{1}{d^2} = \frac{(h^2 + k^2 + l^2)}{a^2} \quad (5)$$

Where:

- $d$  = interplanar spacing.
- $a$  = lattice parameter (0.404 nm).
- $hkl$  = miller indices.

Therefore:

$$d = \frac{a}{\sqrt{(h^2 + k^2 + l^2)}} \quad (6)$$

The calculated  $h^2+k^2+l^2$  values for a range of miller indices is given in Table 15. The measured distance between reflections on the SADP image in Figure 58 for the (200) reflection was 27.50 mm. The  $d$  spacing for the (220), (400) and (420) reflections were subsequently calculated and were found to

compare well with physical measurements of the distances on the SADP image, confirming the cubic orientation of the reflections. For a (100) reflection, the measured distance was 13.75 mm ( $a = 13.75 \times 1$ ). Taking into account the camera constant at 70mm for a (100) reflection:

$$\begin{aligned}
 d_{hkl} &= \frac{\lambda L}{r_{hkl}} \\
 &= \frac{5.952}{13.75} \\
 &= 0.434 \text{ nm}
 \end{aligned}
 \tag{4}$$

This results in an interplanar spacing of 0.434 nm. As the Al matrix and  $\delta'$  are cubic, in a number of crystal plane orientations for example the  $\langle 100 \rangle$  orientation, the interplanar spacing and lattice parameter are the same.

**Table 15: Showing the calculated d values for a range of miller indices. Those relevant to the SADP in Figure 58 are highlighted in yellow.**

| h | k | l | $h^2 + k^2 + l^2$ | $\sqrt{(h^2 + k^2 + l^2)}$ | d<br>(calculated) | d<br>(measured) |
|---|---|---|-------------------|----------------------------|-------------------|-----------------|
| 1 | 0 | 0 | 1                 | 1.00                       | 13.75             | -               |
| 1 | 1 | 0 | 2                 | 1.41                       | 19.45             | -               |
| 1 | 1 | 1 | 3                 | 1.73                       | 23.82             | -               |
| 2 | 0 | 0 | 4                 | 2.00                       | 27.50             | 27.50           |
| 2 | 1 | 0 | 5                 | 2.24                       | 30.75             | -               |
| 2 | 1 | 1 | 6                 | 2.45                       | 33.68             | -               |
| 2 | 2 | 0 | 8                 | 2.83                       | 38.89             | 38.30           |
| 2 | 2 | 1 | 9                 | 3.00                       | 41.25             | -               |
| 2 | 2 | 2 | 12                | 3.46                       | 47.63             | -               |
| 4 | 0 | 0 | 16                | 4.00                       | 55.00             | 54.83           |
| 4 | 2 | 0 | 20                | 4.47                       | 61.49             | 61.40           |

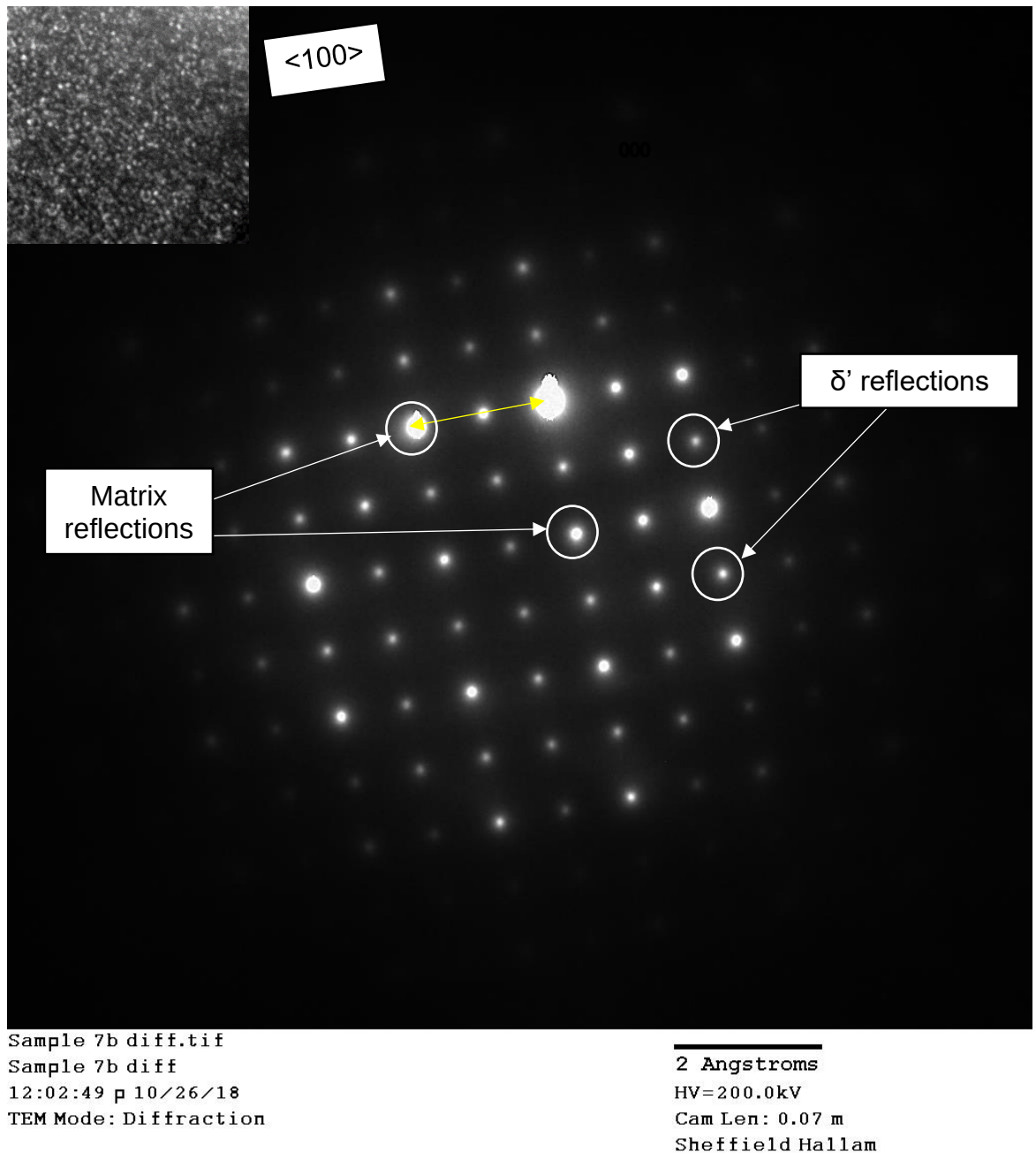


**Figure 59: SADP showing both matrix and  $\delta'$  reflections. The selected area was therefore composed of both matrix and  $\delta'$  within the 8090/AMB sample.**

Given the aforementioned prevalence of  $\delta'$  as a strengthening precipitate (and the subsequent work in Section 4.2), it is highly likely that this SADP contains both reflections of  $\delta'$  precipitates and the Al matrix. The zone of the SADP in

Figure 59 is  $\langle 100 \rangle$  as the beam in this direction was impinging on the foil perpendicularly to the plane of the foil. It is known that the  $\delta'$  crystal planes are homogenous with the lattice and therefore reflections appear aligned with the matrix planes but at half the spacing. This means that in real space, the equivalent hkl planes in the  $\delta'$  structure are spaced at 2x the spacing of the matrix. Measurement of these reflections gave a  $d$  value of 13.70 nm and a resultant interplanar spacing of 0.431 nm. The lattice parameters for the Al matrix and  $\delta'$  precipitates are 0.405 nm and 0.404 to 0.401 nm, respectively.

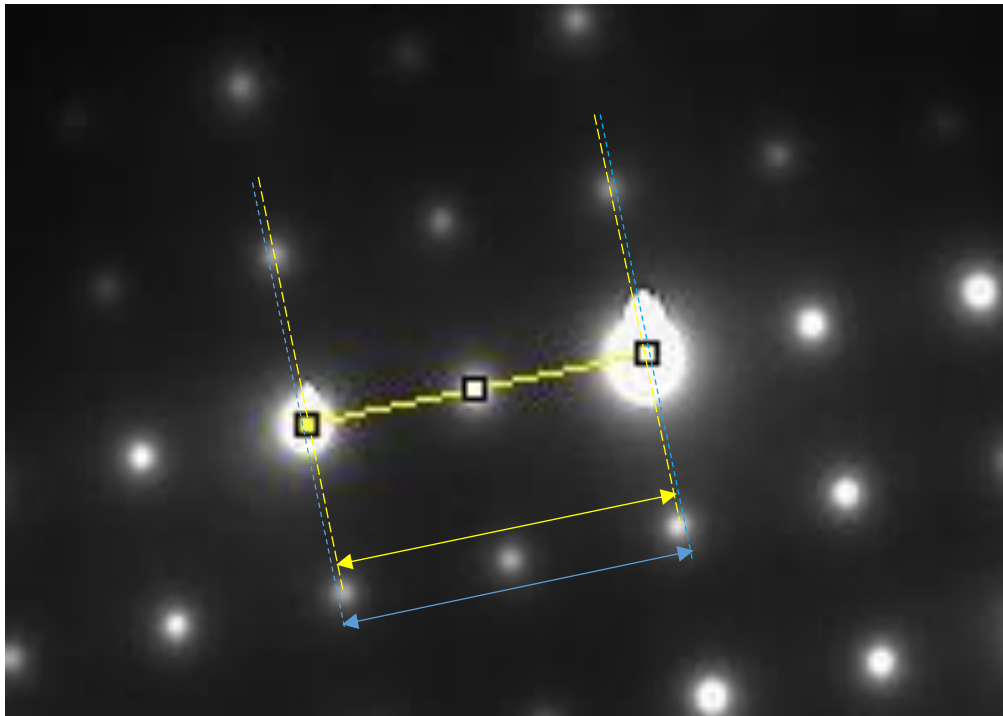


**Figure 60:** Showing the variation in measurement between a 0.434 nm lattice spacing (in yellow) and 0.404 nm lattice spacing (in blue).

Although the lattice space calculations were adrift by  $\sim 0.03$  nm, it is likely that an aberration in a TEM lens or slight tilt to the orientation of the sample is

responsible for this. However, the difference in measurement required for achieve a 0.404 nm interplanar spacing was relatively minor, as is shown in Figure 60. Therefore, the reflections marked in Figure 59 are likely to be Al matrix and  $\delta'$  reflections obtained by electron diffraction of the crystal planes within the matrix and  $\delta'$  precipitates.

Direct comparison with illustrations provided by NASA (Wang and Shenoy, 1998)<sup>116</sup>, confirmed the beam orientation to be consistent with the [100] axis, Figure 61. Using the SADP, a single  $\delta'$  reflection was isolated using the objective aperture and moved to the optical axis of the TEM. This blocks other reflections from contributing to the image in Dark Field. Hence, the  $\delta'$  precipitates could be imaged in Dark Field. An example of HRDF imaging of the  $\delta'$  in an 8090/AMB sample can be seen in Figure 62. The HRDF imaging enabled precipitate diameter measurements to be undertaken. However, thresholding the images (for automatic analysis) within the ImageJ software was difficult due to similarity in greyscale colours and eventually manual measurements (using the ImageJ line measuring capability) was deemed to be the most suitable approach.

Measurements of  $\delta'$  precipitate diameters for the material in the ambient temperature condition gave in an average particle diameter of 24 to 25 nm (depending on whether the measurement was undertaken by hand, or by the Image J software, respectively). The results are detailed in Annex F.

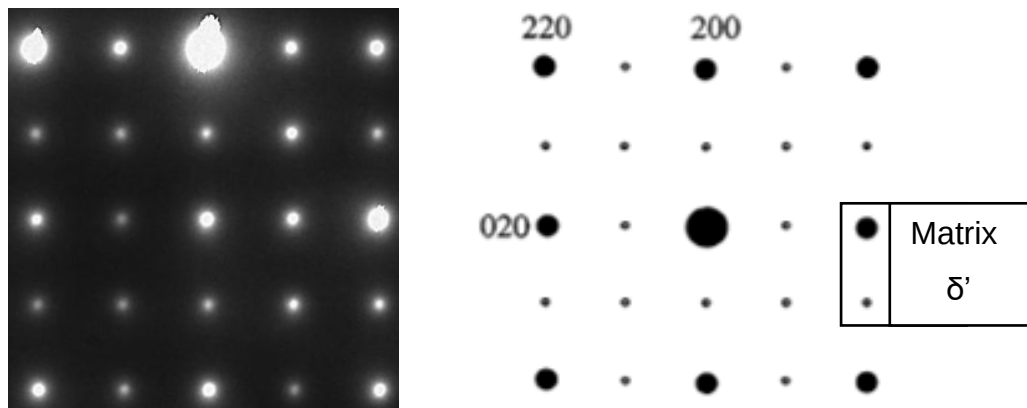


**Figure 61:** Showing the comparison between a diffraction pattern containing both  $\delta'$  and matrix reflections and a NASA illustration of the [001] orientation (Wang and Shenoy, 1998)<sup>116</sup>.

Therefore, the Al-Li main lift frame material was found to have an average particle diameter that was less than the stated critical diameter of ~50 nm for Orowan looping to occur (Edgar, Starke and Quist, 1988)<sup>3</sup>, however this is not unexpected. As previously discussed in the literature review, the  $\delta'$  precipitates are susceptible to shearing by dislocations and hence, the material is susceptible to planar shearing which leads to brittle intergranular failure. Although it should be noted that no evidence of such shearing was present as these samples had not been subject to any form of loading.

Also prevalent throughout the material were lath-shaped precipitates, consistent with either T or S precipitates, precipitating both homogeneously (Figure 63) and heterogeneously (Figure 64).

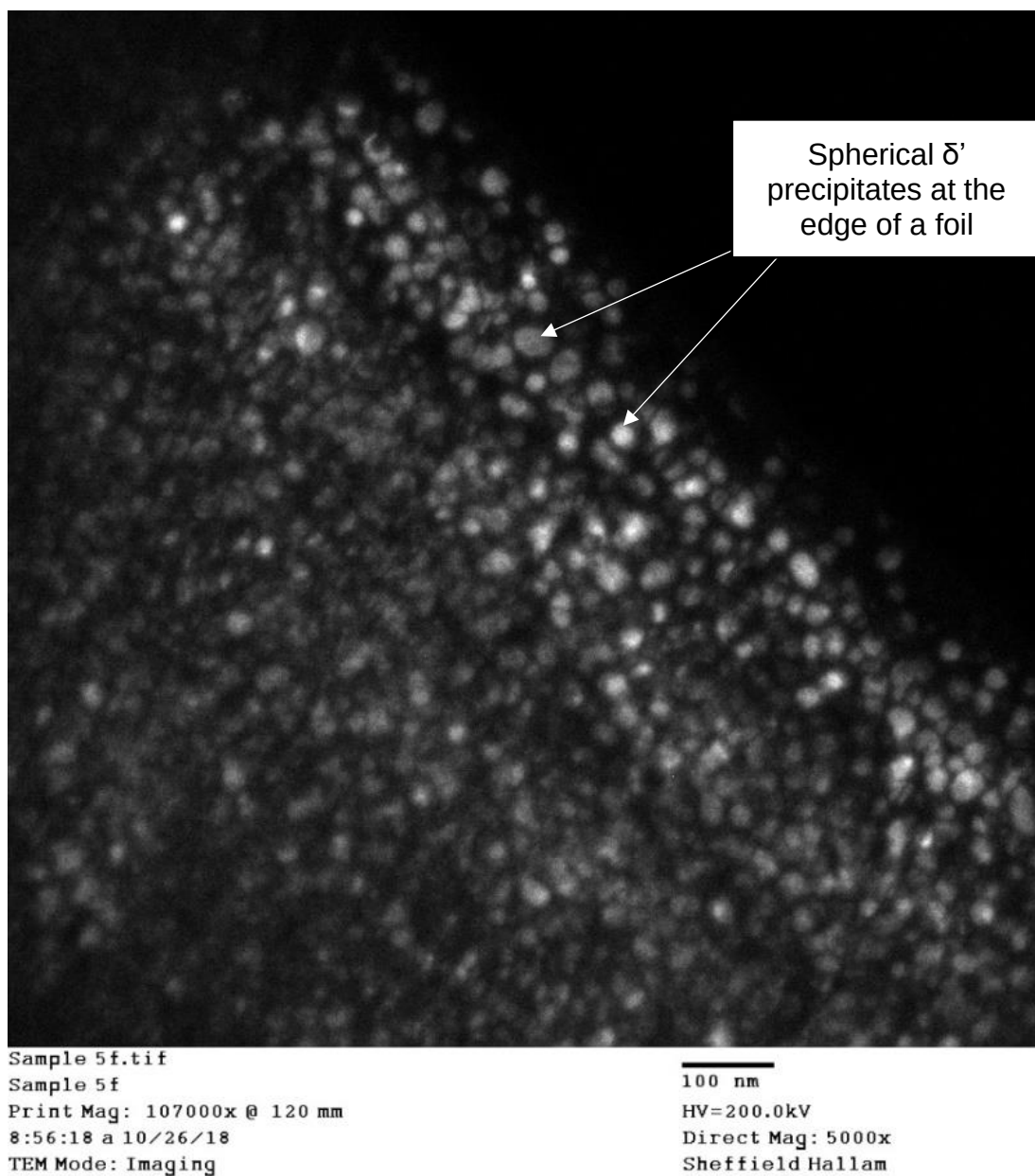


**Figure 62:** HRDF image showing the homogeneously distributed spherical  $\delta'$  in 8090/AMB material. The arrows point at  $\delta'$  precipitates.



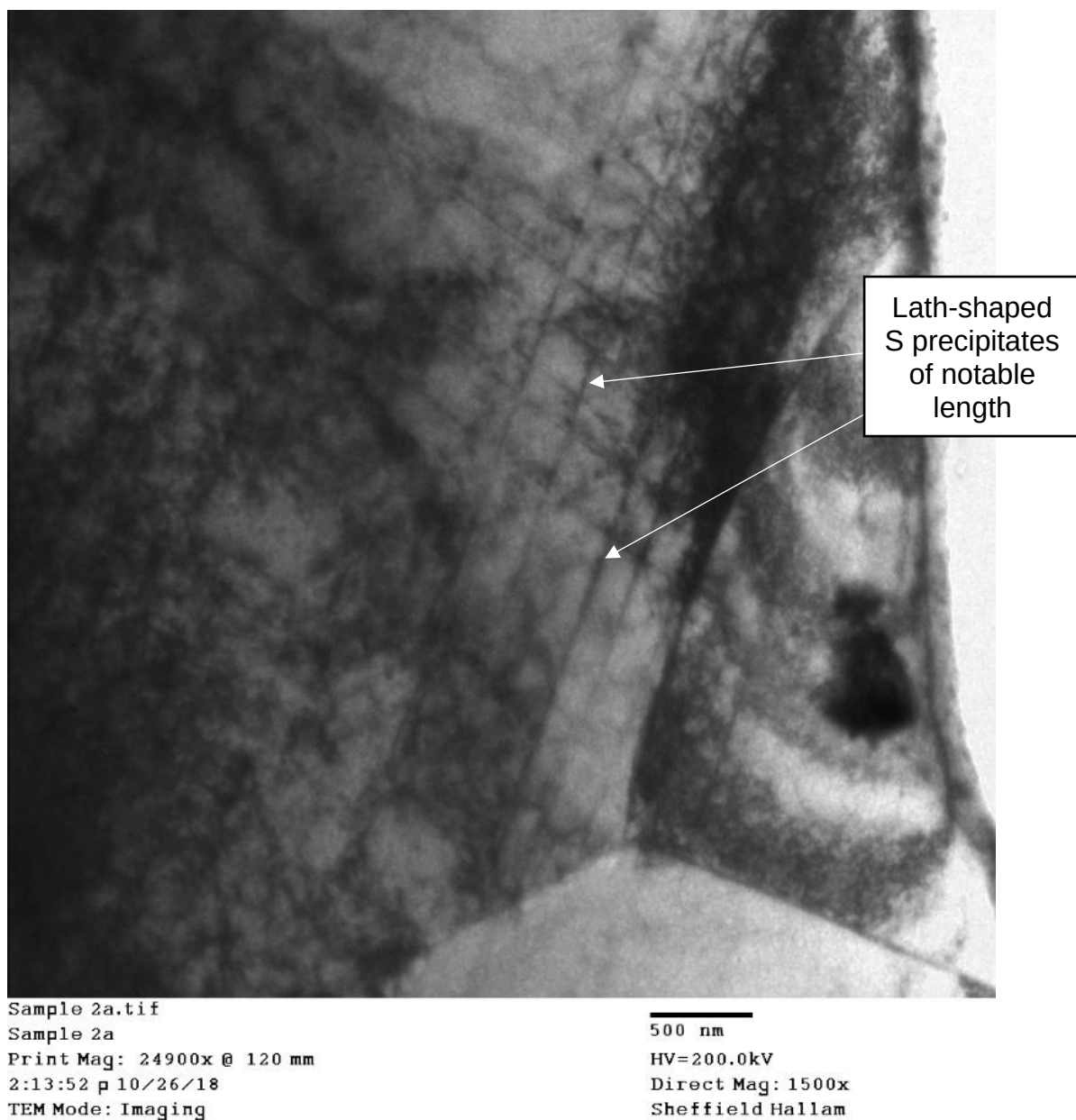


**Figure 63:** TEM bright field image showing homogenous S precipitates precipitation in 8090/AMB material. The arrows point at the lath shapes.

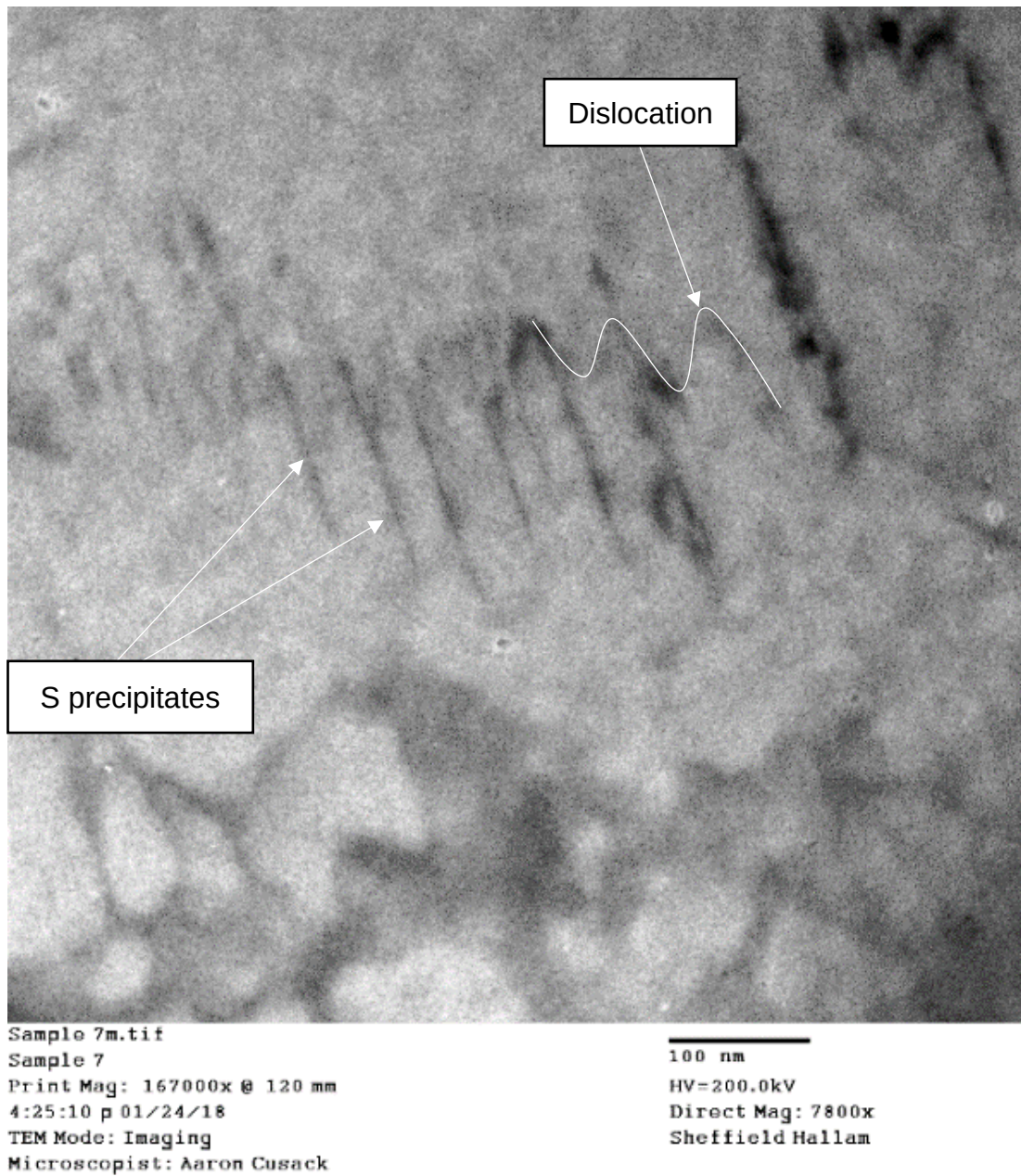


**Figure 64:** TEM brightfield image showing heterogeneous precipitation of S precipitates on a screw dislocation in 8090/AMB material.

However, given the relatively low amount of Cu and availability of Mg within the solid solution, referral to Section 2.1.2 shows that it is the S precipitate which is more likely to be present in the Al-Li 8090 main lift frame material (and indexing of SADP of a precipitate confirms this later in this section). The length of the homogeneously distributed S precipitates was noted to be considerable, with some laths in Figure 63 measured to be up to 3  $\mu\text{m}$  in length. The heterogeneously formed S precipitate laths such as those shown in Figure 64 averaged 182 nm in length.

A SADP obtained from a region of the thin foil containing lath shaped precipitates Figure 65, revealed a typical cubic pattern, associated with the matrix and  $\delta'$  with additional 'streaks' showing between the cubic reflections. A clearer example of the streaky reflections is shown in the SADP obtained from a sample of main lift frame material exposed to 53.1°C for 36 hours (8090/53.1°C/36hrs), Figure 66.

These reflections are typically associated with the S precipitates. The streaks indicated that the suspected S precipitates has a crystallographic aligned relationship with the face centre cubic matrix and are thin laths. This is consistent with the findings of Djaaboube and Thabet-Khireddine (2012)<sup>120</sup> who calculated a number of orientation relationships of the S precipitates and compared them to TEM SADPs, Figure 67. Indexing of the S precipitates and the matrix/  $\delta'$  with the pattern provided by Djaaboube and Thabet-Khireddine (2012)<sup>120</sup> is shown in Figure 68.

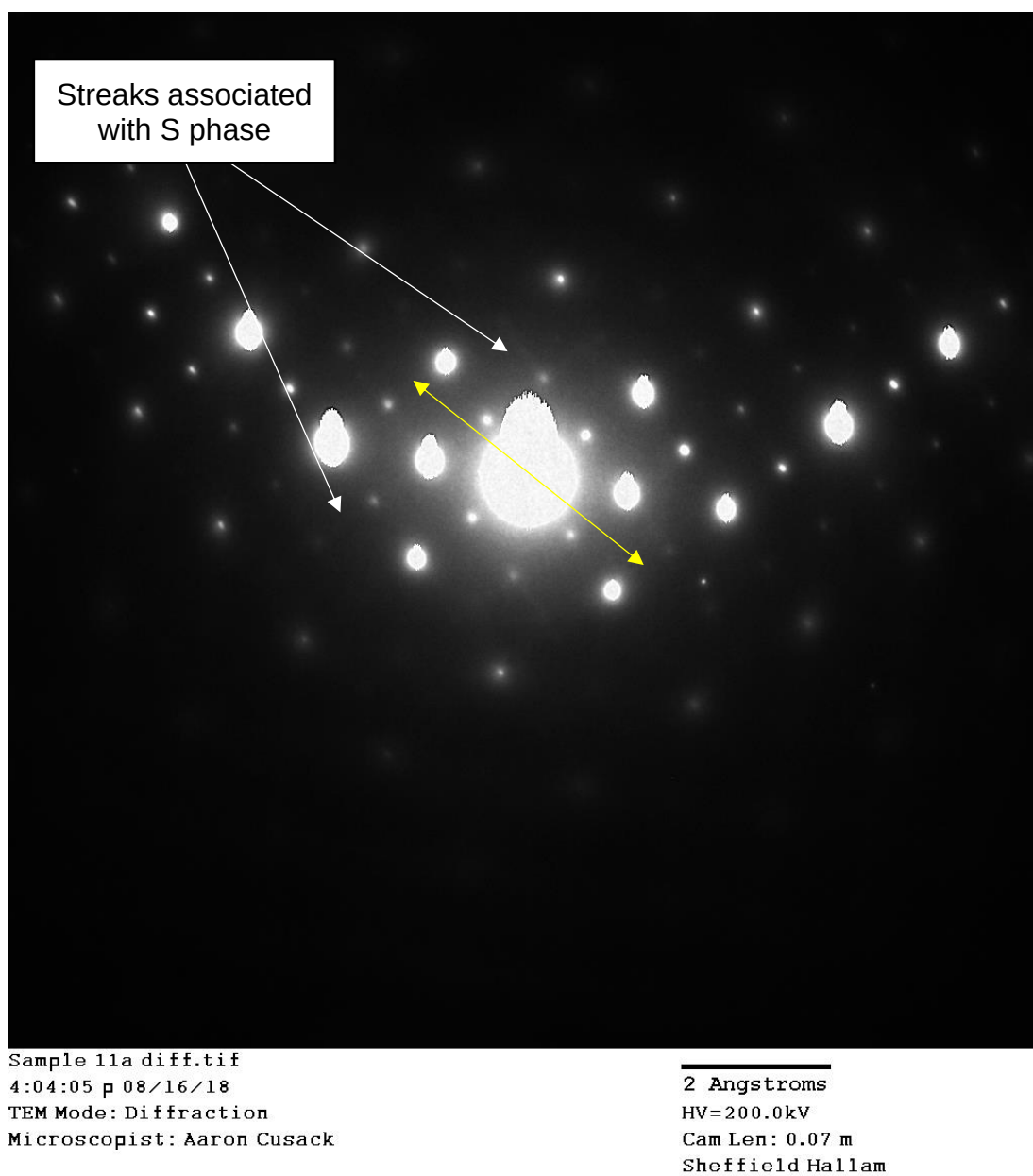
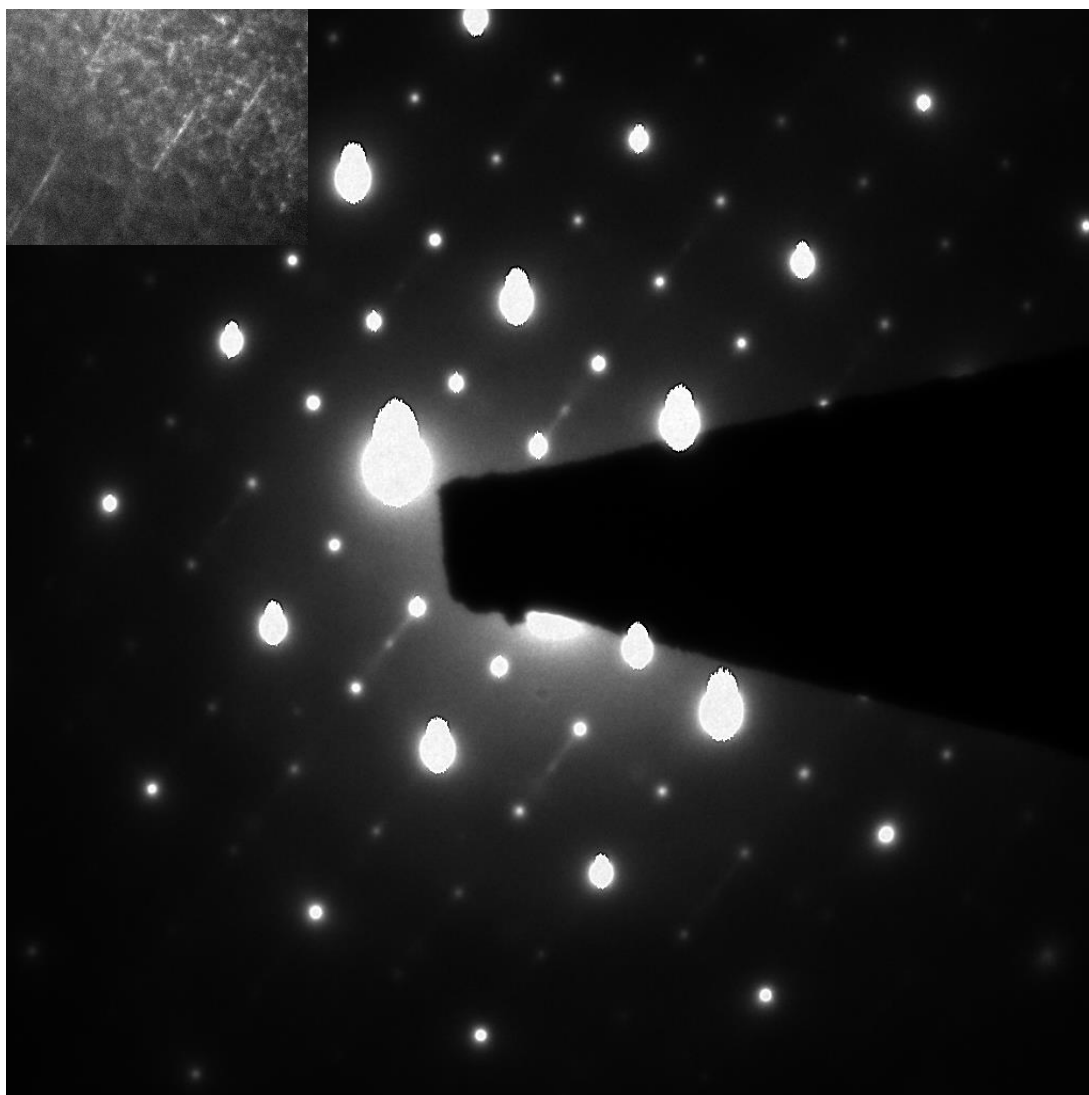


**Figure 65:** Showing the SADP taken from the region of the lath shaped precipitates in an 8090/AMB sample. Yellow line represents the location for a d spacing measurement.



Sample 6h diff.tif  
Sample 6h diff  
2:51:32 p 10/25/18  
TEM Mode: Diffraction

5 Angstroms  
HV=200.0kV  
Cam Len: 0.1 m  
Sheffield Hallam

**Figure 66: SADP obtained from the region of the lath shaped precipitates within an 8090/53.1°C/36hrs sample.**

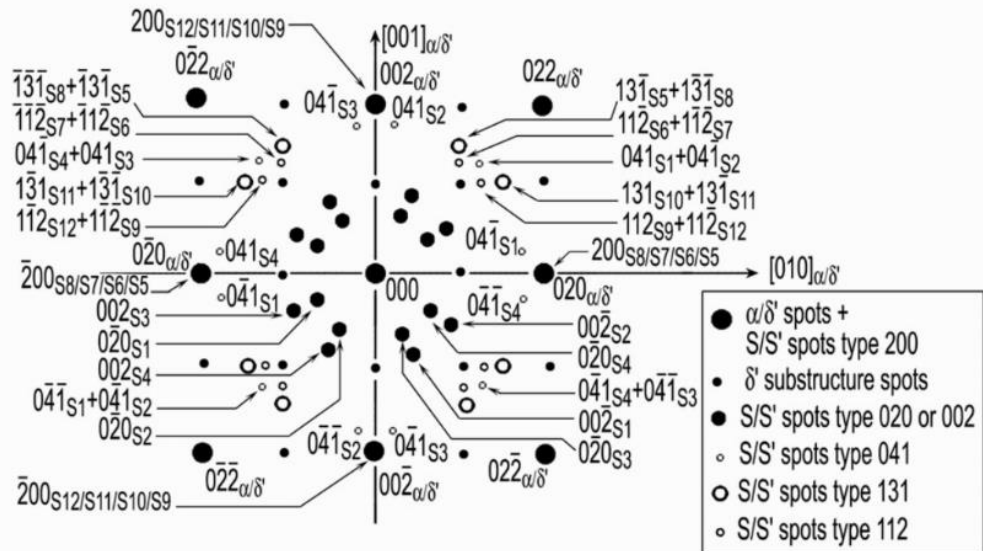
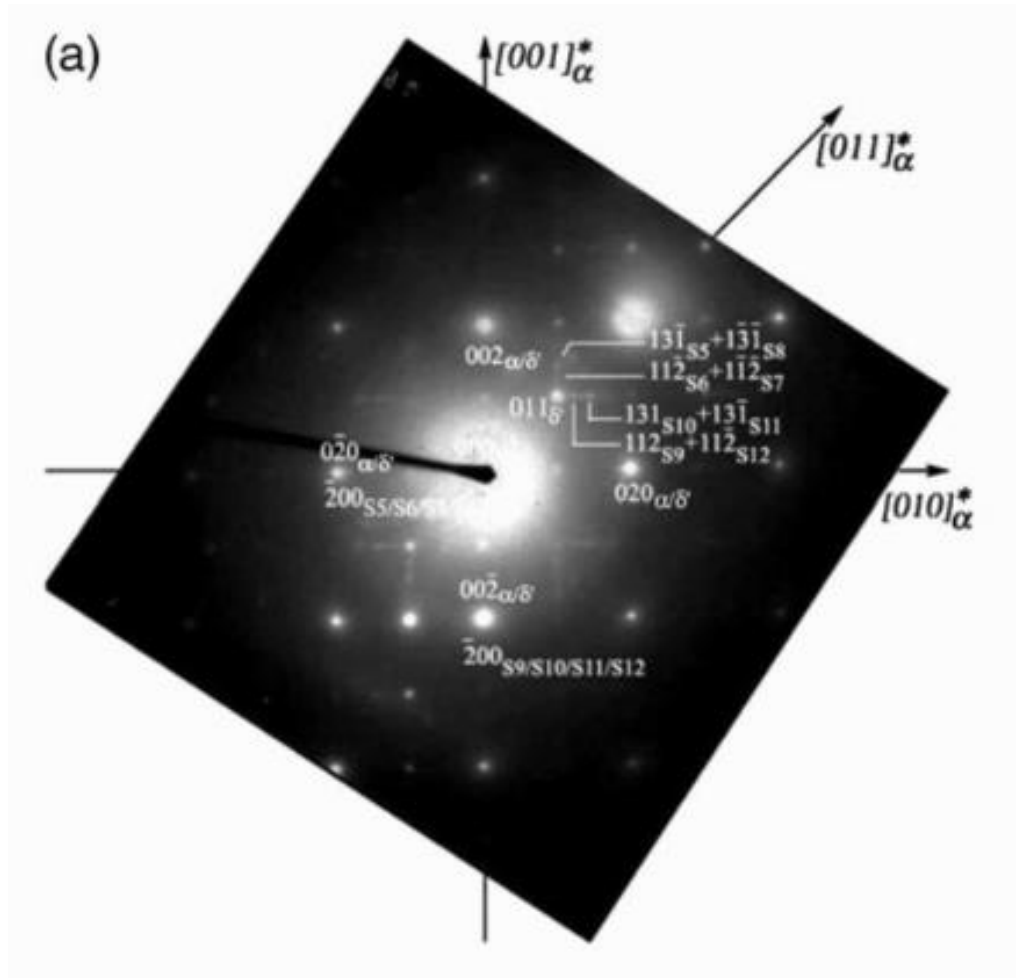


**Figure 67:** Showing Djaaboube and Thabet-Khireddine's SADP containing reflections with measured spacings consistent with those anticipated of the S Precipitates, (2012)<sup>120</sup>.

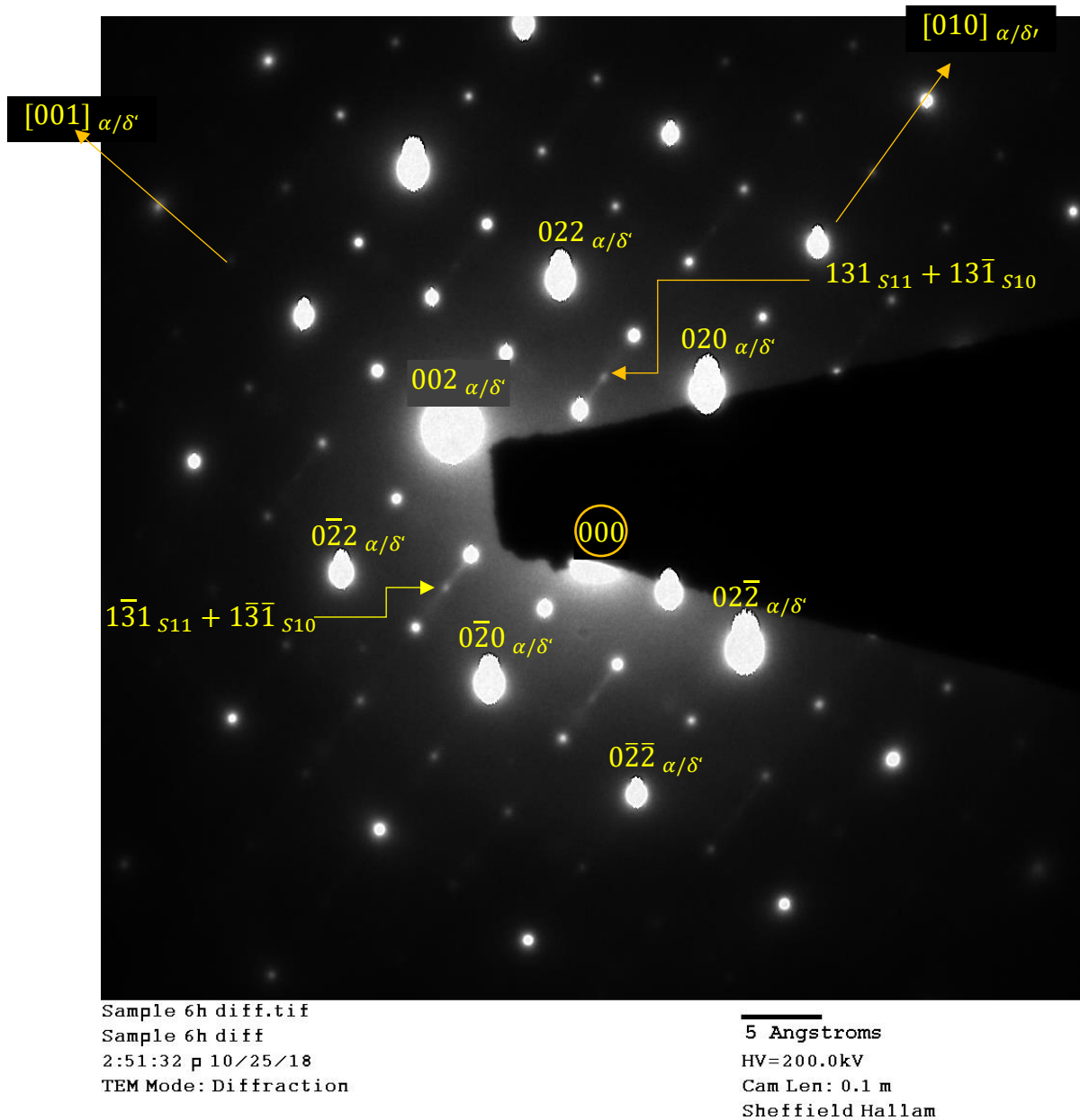


**Figure 68:** Showing the indexed S precipitate reflections from Djaaboube and Thabet-Khireddine's work, (2012)<sup>120</sup>.

As the S phase is an orthorhombic crystal, it has three lattice parameters  $a$ ,  $b$  and  $c$ . The following calculation was used to determine the interplanar spacing for an orthorhombic crystal (Stanford University Lecture)<sup>119</sup>:

$$\frac{1}{d^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \quad (7)$$

Where:

- $d$  = interplanar spacing.
- $a$  = lattice parameter (0.400 nm).
- $b$  = lattice parameter (0.923 nm).
- $c$  = lattice parameter (0.714 nm).
- $hkl$  = miller indices.

Therefore, for a (131) reflection:

$$\frac{1}{d^2} = \frac{1}{0.1600} + \frac{9}{0.8519} + \frac{1}{0.5098}$$

$$\frac{1}{d^2} = 18.7758$$

$$d = 0.2308 \text{ nm}$$

The measured  $d$ , using the image in Figure 65 (as the camera length is different in image Figure 68) was measured to be 53.12 mm. Accounting for the camera length:

$$d_{hkl} = \frac{\lambda L}{r_{hkl}} \quad (4)$$

$$= \frac{5.952}{(53.12/2)}$$

$$d = 0.2240 \text{ nm}$$



As can be seen these results, confirm that the measured interplanar spacing for the SADP of the S precipitates is consistent with the measured interplanar spacing, confirming that they were S precipitates.

#### **5.1.3.1 Summary of TEM analysis for characterisation of 8090/AMB material.**

TEM examination of the 8090/AMB material has successfully identified and indexed the two primary precipitates of interest,  $\delta'$  ( $\text{Al}_3\text{Li}$ ) and S ( $\text{Al}_2\text{CuMg}$ ). Both the spherical  $\delta'$  and lath shaped S precipitates were observed using TEM, with the  $\delta'$  precipitates being identified using High Resolution Dark Field imaging, (Figure 62) and the long homogenous and shorter heterogenous S precipitates identified using Bright Field imaging. The  $\delta'$  precipitates were indexed using a SADP and a standard FCC index pattern Williams and Carter (2009)<sup>118</sup>. The average diameter of the  $\delta'$  precipitates was measured to be between 24 and 25 nm.

The S precipitates were indexed using a SADP and an indexing pattern by Dijaaboube and Thabet-Khireddine (2012)<sup>120</sup>. The shorter heterogenous formed S precipitates (such as on dislocations Figure 64), were measured to be an average of 182 nm in length, and the larger homogenous S precipitates were measured to be up to 3  $\mu\text{m}$  in length.

#### **5.1.4 TEM analysis of low temperature exposure material.**

Further to determining the effect of exposure to 53.1°C on the mechanical properties of the Al-Li lift frame material, a series of temperature soak tests

were undertaken on a number of thin foils with the intention of observing the precipitates of the material after 36 hours exposure to 53.1°C. Five foils were prepared from bulk material (but not subject to the final electropolishing stage). Following the 36 hour soak, the samples were frozen in heptane, electropolished in the Tenupol 5 and re-immersed in heptane for transport to Sheffield Hallam University.

#### **5.1.5 TEM examination of Al-Li 8090 samples heat treated at 53.1°C for 36 hours (8090/53.1°C/36hrs).**

TEM analysis of the samples soaked at 53.1°C for 36 hours (hereon referred to as 8090/53.1°C/36hrs) revealed the presence of  $\delta'$  and S precipitates (Figure 69 a and b) and this was confirmed with SADP images taken in the same region (Figure 66). Measurements of  $\delta'$  precipitate diameters resulted in an average particle diameter of 24 nm, the results of which can be seen in Annex F.

Therefore, given the measurements of  $\delta'$  diameters after exposure to ambient temperature (24 – 25 nm) it can be assumed that the 53.1°C for 36 hour soaking period has not had any effect on the  $\delta'$  precipitate diameters.

No heterogeneously distributed S precipitate laths (akin to those shown in Figure 64) were observed in the 8090/53.1°C/36hrs samples. The homogenous precipitates (Figure 69) were measured to average 185 nm in length. No significantly large laths (as shown in Figure 63) were observed.

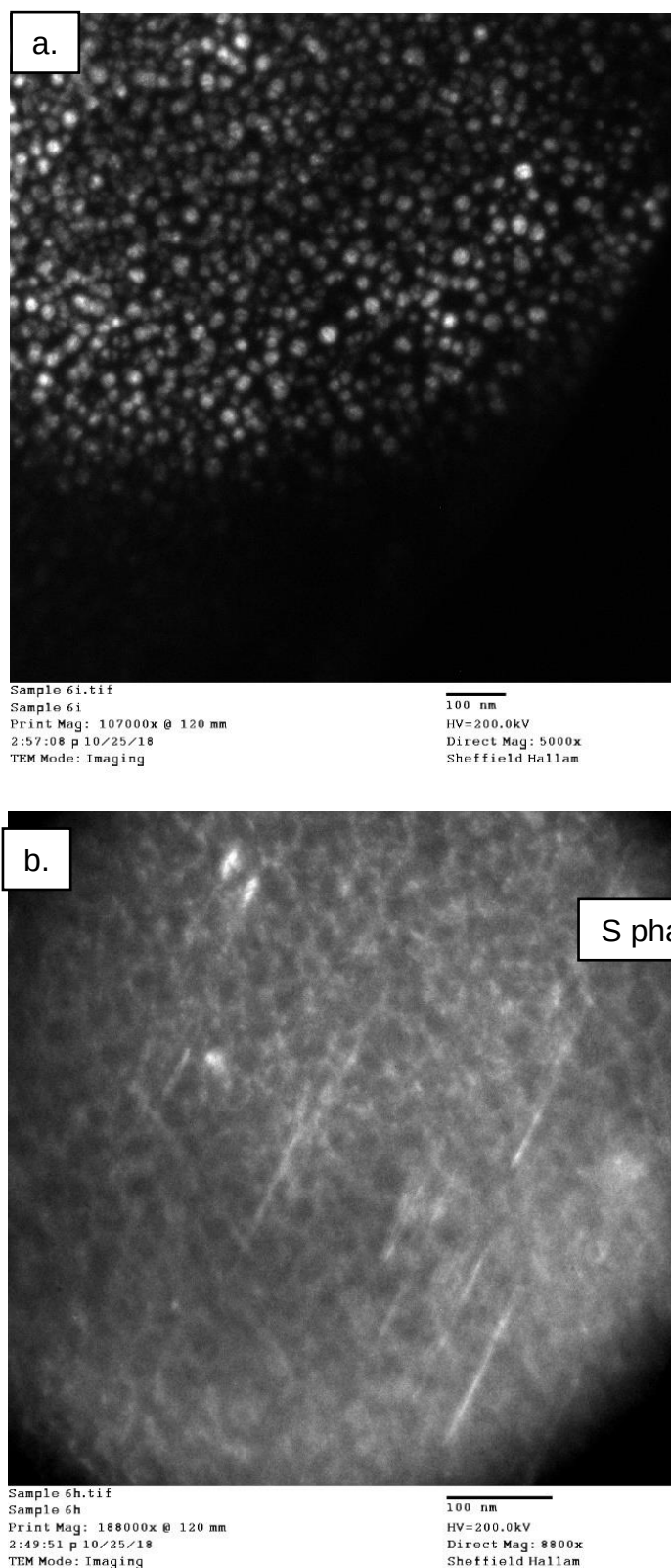


**Figure 69:** TEM high resolution dark field images showing: a)  $\delta'$  precipitates and b) S precipitates in a 8090/53.1°C/36hrs sample.

## **5.2 Summary of TEM examination of 8090/AMB and 8090/53.1°C/36hrs samples.**

TEM examination of the 8090/AMB material has identified the presence of the main strengthening precipitate ( $\delta'$ ). A combination of brightfield, darkfield, HRDF and SADP has been used to confirm the presence of these precipitates. Comparison between the 8090/AMB and the 8090/53.1°C/36hrs samples has shown that no apparent change has occurred to the  $\delta'$  precipitate diameters. With regards to S precipitates, the 8090/53.1°C/36hrs samples show an absence of the significantly large homogeneously distributed precipitates.

As TEM is a highly localised technique, and analysis is undertaken over a small area of material, it is arguably, not representative of the bulk Al-Li main lift frame material. Therefore, Differential Scanning Calorimetry (DSC) analysis was proposed to gain a greater understanding of the effect that exposure to 53.1°C had on the behaviour on the  $\delta'$  and S precipitates over a larger sample of material. An introduction to DSC is included in Section 6.1

## **5.3 A note on oxide removal by using a precision ion polishing system (PIPS) on Al-Li 8090.**

### **5.3.1 Sample preparation and testing.**

Thin foils made from 8090/AMB main lift frame material, were intended to be subjected to 36 hours at 53.1°C. However, a number of difficulties were presented during this period. Firstly, the samples had been suspended in heptane and stored in a freezer to minimise oxidation. The time period between preparation and the second usage (a period of six months) presented

significant opportunity for oxidation to occur. Secondly, the samples could not be removed from the heptane otherwise significant oxidation was assured. Hence the decision was made to soak the samples whilst immersed in the heptane, using bespoke tripod holders. The samples and solution were soaked for 36 hours at 53.1°C, returned to the freezer box and transported to Sheffield Hallam University.

Initial examination of the samples revealed evidence of significant oxidation, rendering each sample (25 in total – 10 of which were subjected to 53.1°C for 36 hours) unusable. Therefore, as the samples were not able to provide any benefit to the project in their current condition, an attempt to remove the oxide layer was undertaken using a Precision Ion Polishing System (PIPS). Concerns were raised about the heat input of an ion milling system resulted in minimizing the time, beam angle and input to determine whether suitable results could be achieved. A final approach using dual beams at 3° for 1 hour was found to be sufficient to remove sufficient oxide from the samples that imaging could be undertaken. Prior to placing the specimens in the TEM that samples were cleaned in a Hitachi ZONE TEM cleaner.

### **5.3.2 The potential effect of PIPS on Al-Li TEM samples.**

It is known that energy input during the ion milling process can change the structure of materials. Work undertaken by Kim and Carpenter (1987)<sup>121</sup> who were using their equipment in the late 80's, initially determined that ion beam thinning had the potential to heat a thin film up to 370°C, which is more than required thermal energy for microstructural changes to occur in aluminium

alloys<sup>xxxvii</sup>. One of the explanations provided in Kim and Carpenters' abstract indicates that poor thermal energy dispersion from a sample during milling was the cause of heating. They recommended the use of high thermal conductivity materials in the design of specimen holders. Viguier and Mortensen (2001)<sup>122</sup> analysed the resultant structure of aluminium thin foils after a number of heat treatments and the compared the resultant structures against a number of ion milling systems. They determined that a 5 kV with a beam angle of 15° for 3 hours resulted in peak temperatures of 400°C, again concluding that heat dissipation through the sample holder was a key factor in reducing specimen heating.

It should be noted that in the case above, the equipment was being used for its intended purpose - milling of a sample with the beam directed on the sample. The PIPS setup for removing the oxide layer of the Al-Li frame samples was set at 3 kV with a beam angle of 3° for a maximum of 1 hour. Although attempts were made to try and measure the temperature of the Al-Li samples during the milling process, this could not be achieved and whilst the samples were never noted to be hot or even warm upon extraction it was unknown as to what temperature the samples reached during milling. This observation was also made and subsequently discounted by Viguier and Mortensen (2001)<sup>122</sup>.

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<sup>xxxvii</sup> *Note: Kim and Carpenter (1987)<sup>121</sup> is cited in Viguier and Mortensen (2001)<sup>122</sup>. No full text was available; however, the abstract provided the above information.*

A table of collated data regarding PIPS systems (including the Gatan system in use at SHU) by Viguier and Mortensen (2001)<sup>122</sup>, notes that using a gripped specimen holder with a 5 kV operating voltage and 8° incidence angle (again assumed with a 3 hour mill) resulted in TEM observations of sample structures consistent with samples heat treated at 240°C. Viguier and Mortensen (2001)<sup>122</sup> concluded that by using appropriate thermally absorbent materials, peak temperatures of 373K (100°C) can be readily achieved.

Therefore, the question stood as to whether a 1 hour 3 kV 3° PIPS session was sufficient to change the structure of the Al-Li main lift frame material. Considering the focus of this work was on the  $\delta'$  precipitates, the thermal energy input into the material would likely result in either: coarsening or dissolution of the precipitates, or transformation of the  $\text{Al}_3\text{Li}$  into AlLi ( $\delta$  precipitates).

Comparison with the Time Temperature Transformation diagram for a binary Al-Li alloy (Figure 70) shows that at exposure at 240°C for 60 minutes will enable the growth of  $\delta'$  precipitates, whereas this temperature is too low for the emergence of the equilibrium  $\delta$  precipitates. Therefore, given the possibility that PIPS had the potential to alter the  $\delta'$  precipitates in the TEM, a short experiment was undertaken on oxidised samples that had been soaked at 53.1°C for 36 hours.

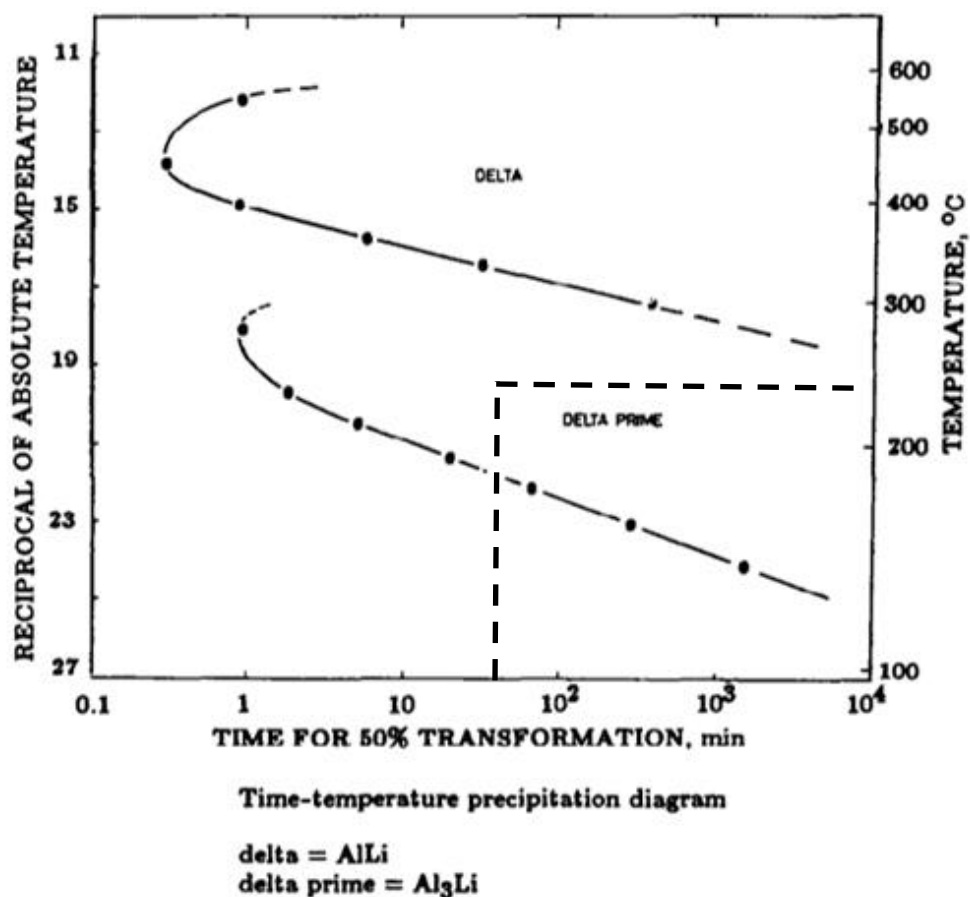


Figure 70: Time-Temperature-Transformation diagram for 4% Al-Li (ASM Handbook, 1992)<sup>123</sup>.

### 5.3.3 Analysis of 8090/53.1°C/36hrs+PIPS and 8090/AMB+PIPS samples.

TEM Examination of a sample soaked at 53.1° for 36 hours subjected to PIPS (8090/53.1°C/36hrs+PIPS), revealed evidence of the growth/coarsening of a bimodal distribution of homogenous  $\delta'$  and in addition what was suspected to be  $\beta$ , Figure 71. These precipitates were far larger in diameter than the as-received  $\delta'$  precipitates and were measured to be between 35-45 nm in diameter.



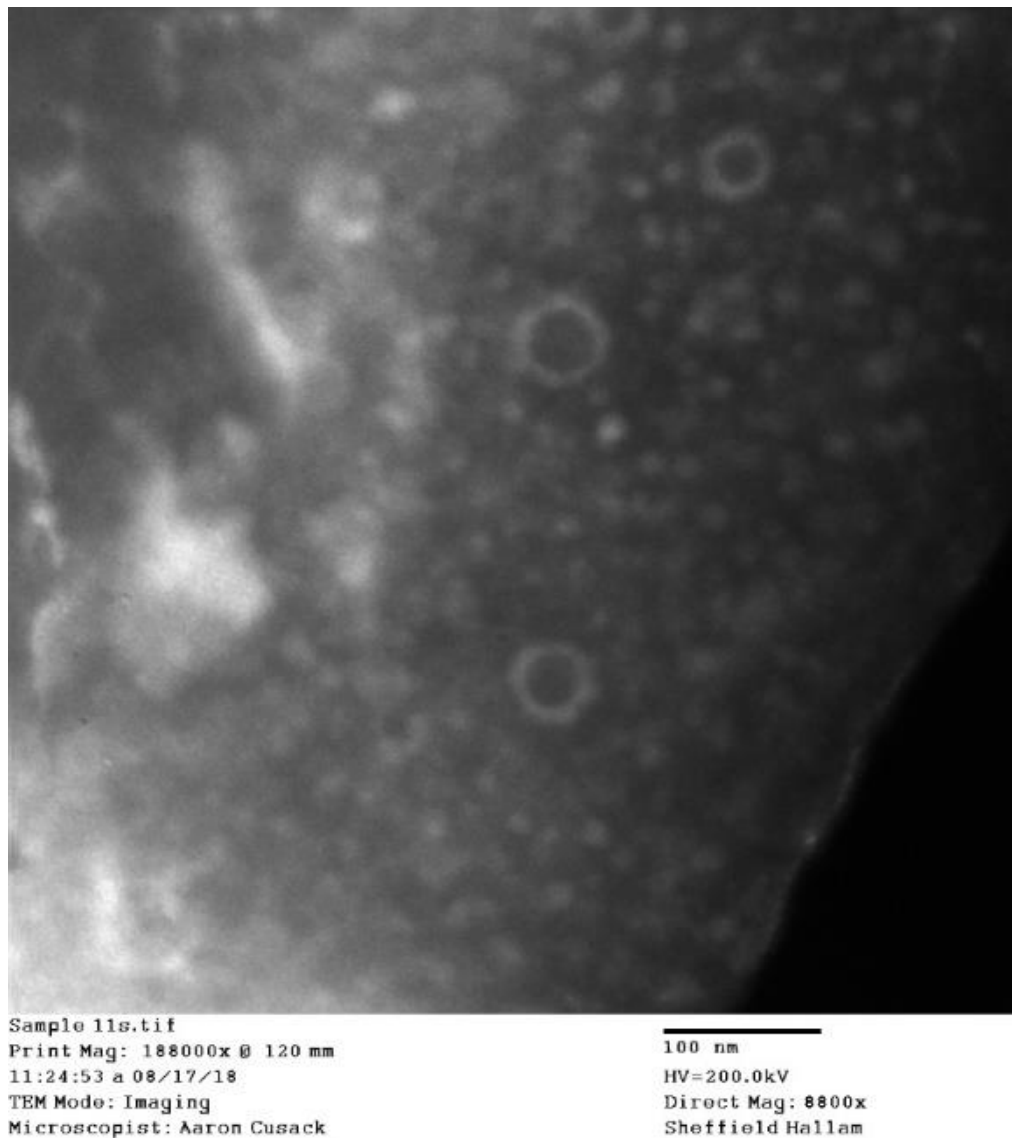


**Figure 71: TEM micrograph of a 8090/53.1°C/36hrs+PIPS sample showing suspected heterogeneous distribution of bimodal  $\delta'$  and  $\beta$  interspersed with the homogenous  $\delta'$ .**

The  $\delta'$  precipitates in the 8090/53.1°C/36hrs+PIPS sample were measured to have an average diameter of 16.1 nm, which was 7.9 nm less than what was measured for the 8090/53.1°C/36hrs material that had not been subjected to PIPS. In addition, there was evidence of nucleation of new heterogeneous  $\delta'$  precipitates localised at dislocations, Figure 72.

In an attempt to determine whether the PIPS system did have an effect on the ambient temperature main lift frame material, a 8090/AMB sample from the same set of thin foils was subjected to 3 kV 3° for 1 hour.

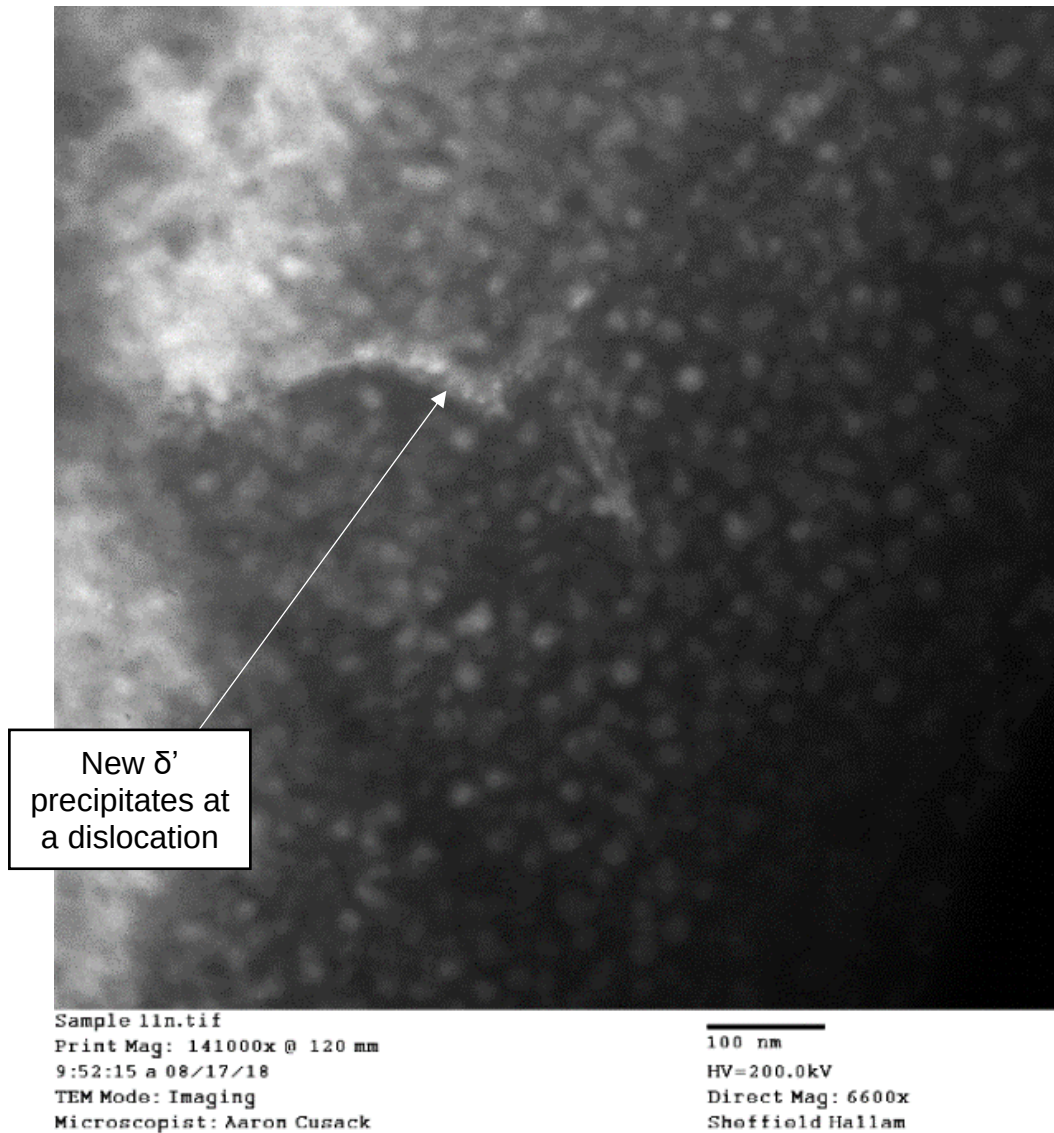


**Figure 72:** TEM high resolution dark field micrographs showing the reduction in precipitate diameter of the native  $\delta'$  and nucleation of a new precipitates at a dislocation in a 8090/53.1°C/36hrs+PIPS sample.

As can be seen in Figure 73a and b, the large bimodal spherical particles were still noted to be present in the 8090/AMB+PIPS TEM foils, but they weren't as

prevalent as the examples in Figure 71. Average particle diameter (for the native  $\delta'$  precipitates) was measured to be 22.1 nm, indicating that the significant reduction in average particle diameter observed in the 8090/53.1°C/36hrs+PIPS was not present in the 8090/AMB+PIPS sample.

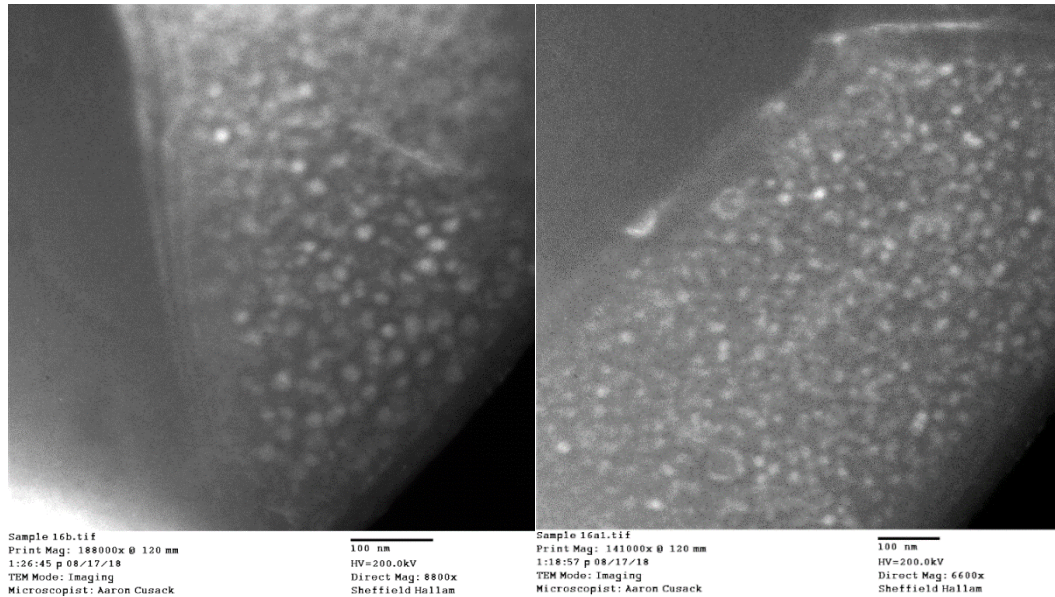


**Figure 73: TEM micrograph showing an 8090/AMB+PIPS Al-Li main lift frame sample.**

#### 5.3.4 Summary of using PIPS to remove oxide layers on Al-Li TEM samples.

Noting that the  $\delta'$  particle diameters have been measured to be different for both the 8090/AMB+PIPS and the 8090/53.1°C/36hrs+PIPS samples, by comparison with the non-PIPS samples, it is concluded that the PIPS process had an deleterious effect on the Al-Li main lift frame foils, which is consistent with the effect predicted from the TTT diagram (Figure 70). Therefore the +PIPS samples were disregarded from this project and PIPS was not employed for future work.

## 6 Differential scanning calorimetry of al-li 8090 main lift frame material.

### 6.1 Introduction to differential scanning calorimetry.

Differential Scanning Calorimetry (DSC) is an established technique for studying precipitate transformations in metallic materials. DSC fundamentally works by monitoring the change in heat flow in a sample. This can be shown as either an endothermic or exothermic reaction, which is related to the Gibbs Free Energy ( $\Delta G$ ).  $\Delta G$  is defined as:

$$\Delta G = \Delta H - T\Delta S \quad (8)$$

Where:

$\Delta H$  = Change in enthalpy.

$\Delta S$  = Change in entropy.

$T$  = Temperature.

In an exothermic reaction, the change in enthalpy has a negative value ( $\Delta H < 0$ ). Ordering of structures, such as crystalline solidification from a liquid, results in energy being released as heat, giving a negative change in enthalpy which is an exothermic reaction. The melting of a solid structure into a liquid requires an input of energy. This results in absorption of heat and requires a positive change in enthalpy ( $\Delta H > 0$ ), which is an endothermic reaction. In DSC, the change in heat flow is measured by using two aluminium pans (one containing sample material and the other a blank reference) that are heated at a constant rate in a controlled atmosphere. If the sample material undergoes an

exothermic reaction, the released heat will increase the temperature of the pan and therefore less heat is required to maintain the sample pan at the same temperature as the reference pan. The reduction in heat flow is equal to the heat flow from the exothermic process. Conversely, during an endothermic reaction as heat is absorbed, the sample pan requires heat input to maintain the same temperature as the reference pan. The increase in heat required during an endothermic reaction is equal to the amount of heat absorbed by the endothermic process.

DSC has been used to study Al-Li alloys 8090 across a range of heat treatments, including the solution heat treated (530°C for 30 mins) and T81 (solution heat treated, cold worked and artificially aged for 24 hours at 150°C) conditions, (Starink, Hobson, Sinclair and Gregson (2000)<sup>124,xxxviii,125</sup>. These workers produced the DSC curve shown in Figure 74 (heat flow versus temperature) for solution heat treated Al-Li 8090 sheet and the curve in Figure 75 for the T81 temper. Six possible reactions were proposed by Starink and Gregson (1996)<sup>126</sup>, that would be likely to evolve or consume heat and result in the observed peaks and troughs on the DSC curve. The latter are designated 'A' to 'F'. The six reactions are:

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*xxxviii This work, undertaken by the Materials Research Group at the University of Southampton, was encountered as a result of the progression of the findings of this project and from discussions with Sir Peter Gregson<sup>125</sup>. Given that the work correlates with this part of the project so closely, the author has chosen to comment on it at this point and not earlier in the literature review as to maintain relevance with the chronological development of this project.*

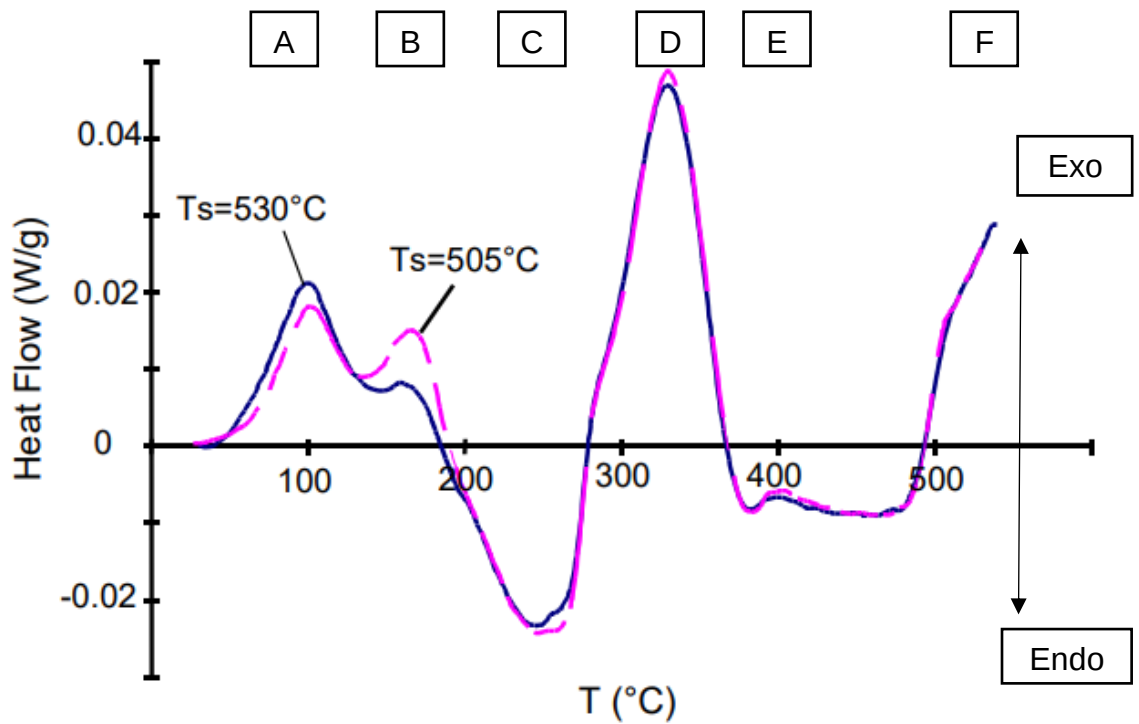


**Figure 74:** Showing the DSC curve for solution heat treated Al-Li 8090 sheet. Starink et al. (2000)<sup>124</sup>.

- A. GP zone formation (exothermic).
- B.  $\delta'$  precipitation (exothermic).
- C. GP zone and  $\delta'$  dissolution (endothermic).
- D. S precipitates precipitation (exothermic).
- E. S precipitates dissolution (endothermic).
- F. Oxidation reactions.

The DSC curve for peak aged Al-Li 8090 sheet material in the T81 temper state (as shown in Figure 75, Starink et al., 2000<sup>124</sup>), does not exhibit exothermic peaks for the formation of GP zones or  $\delta'$  precipitates (reactions A and B, Figure 74). The reason for this that the material has been artificially aged, and therefore all of the available solute<sup>xxxix</sup> for the formation of GP zones and  $\delta'$  precipitates has already been utilised, and therefore no further exothermic reactions will occur between 20-175°C on the DSC curve for the artificially aged material.

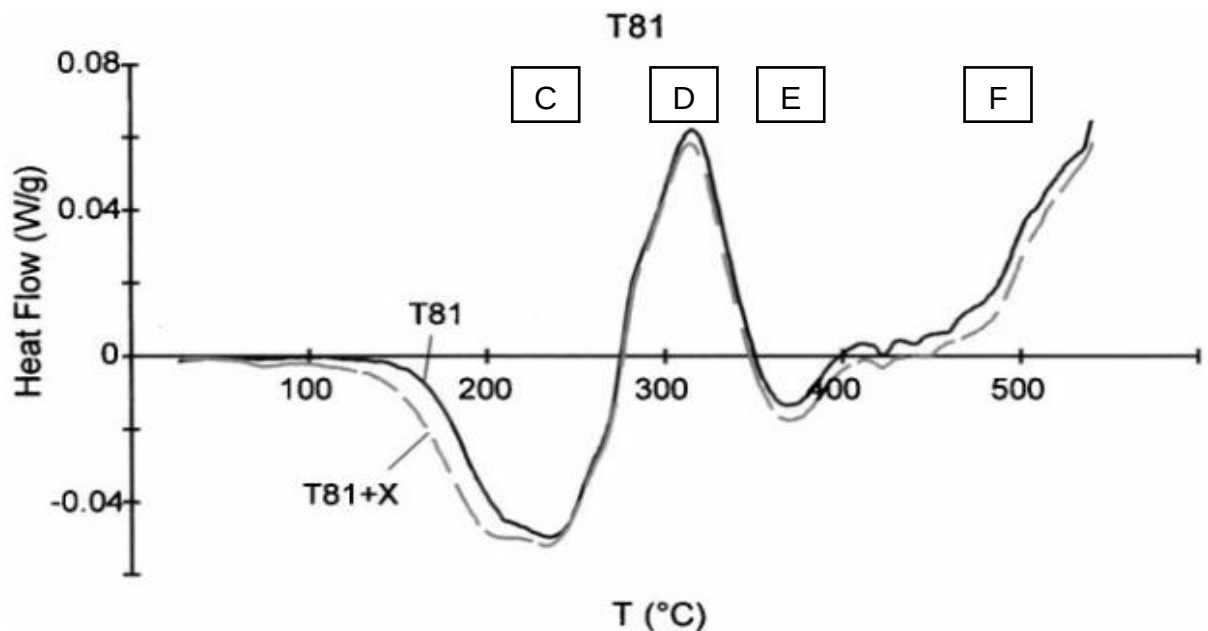


**Figure 75:** Showing the DSC curve for 8090 sheet material in the T81 temper state. Note the absence of reactions A and B Starink et al. (2000)<sup>124</sup>.

<sup>xxxix</sup> The term 'solute' in this thesis is used to describe an atom (of an alloying element). For example, Li dissolves into the Al matrix to form a solid solution. For the formation of  $\delta'$  this would adopt a specific position in the lattice and form a compound. However the term 'Al-Li compound' (or Al-Cu as Cu is also present) has not been used as this would infer a degree of covalent bonding, where the bonding of  $\delta'$  is metallic bonding.

## **6.2 Sample preparation of Al-Li 8090 main lift frame material for DSC analysis.**

Sample material was cut from a main lift frame section and was filed to form an approximate rod-shaped profile, the diameter of which was able to fit within a DSC sample pan. From this rod, ~0.8 mm thick discs were cut using a Struers Accutom. The samples were lightly abraded with 800 grit sandpaper to remove any burrs and ensure good contact with the sample pan.

The decision to section the rod and prepare the samples prior to any heat treatment was instigated by findings of Starink, Hobson and Gregson (1995)<sup>127</sup> who determined that grinding and punching of DSC samples post heat treatment was significant enough to alter the precipitation behaviour in single crystal Al-Li alloys. This was mainly attributed to the formation of additional dislocations during the grinding and punching processes. Starink, Hobson and Gregson (1995)<sup>127</sup> therefore concluded, that as precipitation reactions are influenced by dislocations in all Al-based alloys, post-mechanical processing of heat treated samples could result in misleading DSC curves. Initial DSC testing for this work was undertaken at SHU using a Perkin Elmer DSC 8000, however further comparative testing was also undertaken at 1710 NAS using a Perkin Elmer Diamond DSC.

### **6.2.1 DSC methodology for Al-Li main lift frame material.**

The temperature scale of both DSCs was calibrated using pure indium reference samples. Baseline DSC testing was undertaken with empty aluminium pans. The SHU testing did reveal an increase in heat flow as the



temperature increased, resulting in a curved baseline. This is not uncommon<sup>128</sup> when analysing metals. The temperature range of the detectors is often being operated far beyond that required for the more common assessment of non-metallic materials. Comparison of the data from both DSCs gave consistent baselines results, giving confidence in both sets of data. However, the baseline subtraction for the Perkin Elmer Diamond DSC machine gave more comparable DSC curves with the curves presented in the established literature, (Starink et al. (2000)<sup>124</sup> and Starink and Gregson (1996)<sup>126</sup>). The methodology used for the DSC analysis (on both pieces of equipment) was adopted from the process detailed by Starink and Gregson (1996)<sup>126</sup> and was as follows:

- 1) Placement of the Al-Li sample in the DSC sample chamber and an empty aluminium pan in the DSC reference chamber to be used as the reference sample.
- 2) Cooling of the sample(s) to 0°C.
- 3) Once the Heat Flow had stabilised at 0°C, the following heat run method was initiated:
  - a. No. of continuous runs: 3
  - b. Start temperature: 0°C.
  - c. Maximum temperature: 545°C.
  - d. Ramp up rate: 10°C / min.
  - e. Cooling rate between each run: free cooling rate for the 1<sup>st</sup> run.
  - f. Cooling rate for 2<sup>nd</sup> and 3<sup>rd</sup> runs: free cooling to 300°C, followed by controlled cooling of 1°C / min.

This method required three subsequent heating runs beginning at 0° and heating to 545°C at a ramp up rate of 10°C/min. However, unlike the free cooling rate used by Starink and Gregson (1996)<sup>126</sup>, a controlled 10°C/min cooling rate was used for each cycle. As each run required a heating and cooling cycle, each three-run session on the same sample required in excess of 5 hours to complete. As the 2<sup>nd</sup> and 3<sup>rd</sup> runs did not appear to provide any further benefit to this work, mainly as the various precipitates were taken into solution (530°C) during the 1<sup>st</sup> run, the decision was taken to discontinue the 2<sup>nd</sup> and 3<sup>rd</sup> runs.

DSC sample runs were undertaken on the following material conditions:

- i. Solution heat treated main lift frame material (8090/SHT).
- ii. Ambient temperature main lift frame material (8090/AMB).
- iii. Main lift frame material that had been heat treated at 53.1°C for 24 hours (8090/53.1°C/24hrs).
- iv. Main lift frame material that had been heat treated at 53.1°C for 36 hours (8090/53.1°C/36hrs).

All of the heat treated DSC sample runs (8090/SHT, 8090/53.1°C/24hrs, 8090/53.1°C/36hrs) were started within 5 minutes of completion of their specific heat treatments.

## 6.2.2 DSC results for Al-Li main lift frame material.

### 6.2.2.1 Solution heat treated material (8090/SHT).

The DSC curve for 8090/SHT material is shown in Figure 76. As can be seen in Figure 76, the majority of the reactions proposed by Starink and Gregson(1996)<sup>126</sup> (Figure 74), are present in the data curve for the 8090/SHT material. reactions; A (GP zone formation), C (GP zone and  $\delta'$  dissolution), D (S precipitation) and E (S dissolution) were clearly defined on the DSC curve. reaction B ( $\delta'$  precipitation) was not clearly defined but could still be discerned.

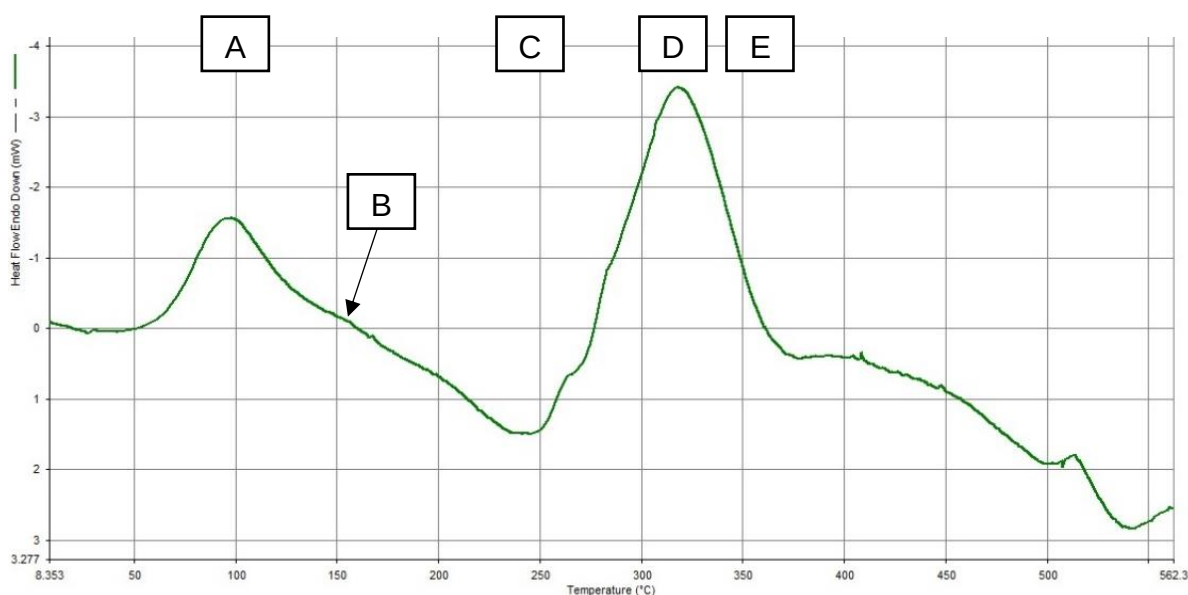


**Figure 76: Showing the DSC curve for the 8090/SHT material.<sup>x1</sup>**

The exothermic reaction B that is attributed in the current work to  $\delta'$  precipitation was not found to be as distinctive as shown in the literature, however a slight exothermic peak was present at  $\sim 160^{\circ}\text{C}$  which is consistent

<sup>x1</sup> Note all DSC curves in this work are presented 'endo down'. (Exothermic reactions are shown up in the y axis and endothermic reactions are down).

with the DSC curve shown in Figure 74. Reaction F (attributed to oxidation reactions), gave an endothermic behaviour as opposed to exothermic behaviour in Figure 74. However, as the main focus of this work was centred on the thermal behaviour of the material considering reactions A to D, these data points (reaction F) on the DSC curves were not deemed to be relevant and were not considered in this work<sup>xli</sup>.

The DSC curve for the 8090/SHT material displayed slightly less heat flow for both the exothermic and endothermic reactions when compared against the literature (Starink et al. 2000)<sup>124</sup>. This was revealed in Figure 76 and the DSC curve in Figure 74, with a difference of approximately 0.5 mW for the exothermic peak in the data for reaction A, 1 mW for the endothermic peak in reaction C and 1 mW for the exothermic peak in reaction D.

However, apart from the reduced reaction B peak, the 8090/SHT DSC curve gave favourable results with Starink et al.'s (2000)<sup>124</sup> solution heat treated DSC curve, Figure 74.

#### 6.2.2.2 Ambient temperature material (8090/AMB).

The DSC curve for the 8090/AMB material is shown in Figure 77. There was no significant indication of reactions A or B. This was expected, as the 8090/AMB material was already in the artificially aged condition, and therefore

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<sup>xli</sup> Consultation with Perkin-Elmer<sup>128</sup>, provided confidence to support this decision.

maximum GP zone and  $\delta'$  formation and growth should have already been achieved through the dual heat treatment process undertaken at manufacture.

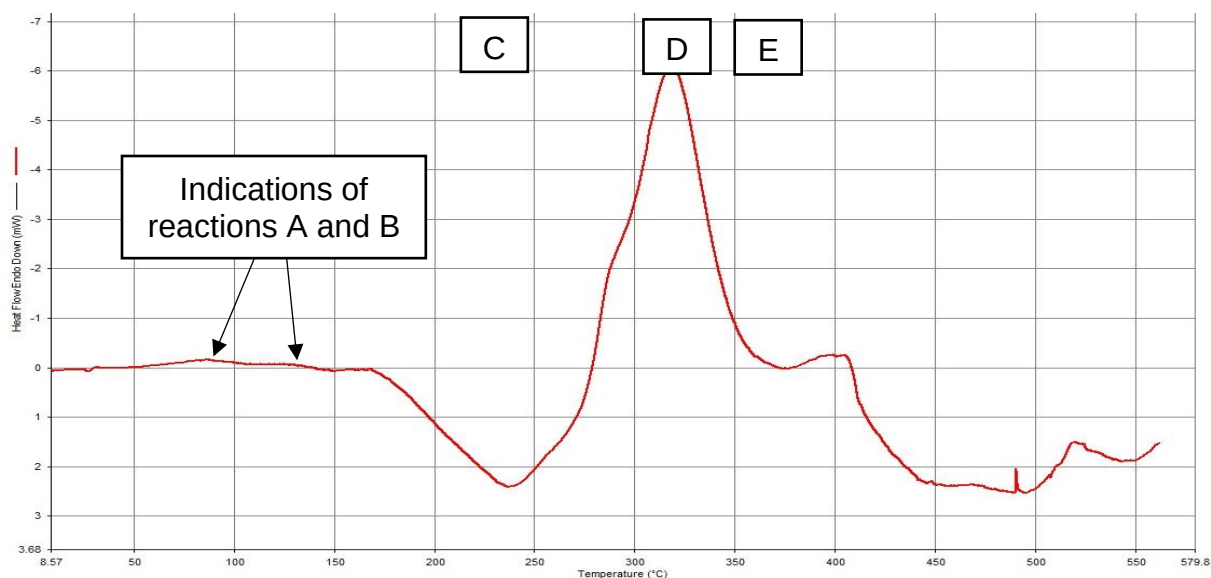


**Figure 77:** Showing the DSC curve for the 8090/AMB material, with regions labelled as being consistent with reactions C, D and E.

These findings were consistent with 8090 material in the T81 temper state Starink et al. (2000)<sup>124</sup>, Figure 75. However, in the DSC curve for the 8090/AMB material, Figure 77 there is a slight indication of an exothermic reaction occurring between 50 to 150°C which could be indicative of either a precipitation or coarsening mechanism. Close inspection of this region did reveal indications of two exothermic peaks that may be consistent with reactions A and B (Figure 77). This is discussed further in Section 7.

The main endothermic event for the 8090/AMB material (likely reaction C, Figure 77) commenced at around 170°C, as opposed to the material in the T81 temper state where it began at approximately 140°C, Figure 75. There

was no evidence of the dual endothermic peaks at reaction C for the 8090/AMB material, Figure 77 that are evident in the material in the T81 temper state, but the maximum heat loss for an endothermic reaction was at  $\sim 230^{\circ}\text{C}$  for both materials.

#### **6.2.2.3 8090/53.1°C/24hrs material and 8090/53.1°C/36hrs material**

The DSC curves for the 8090/53.1/24hrs and 8090/53.1°C/36hrs materials are shown Figure 78 and Figure 79, respectively. Both DSC curves gave similar results as the 8090/AMB material (Figure 77) with evidence of reactions C, D and E occurring at the same temperatures as those obtained for the 8090/AMB material. Furthermore, both the 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials gave similar indications of exothermic peaks for reactions A and B between the temperatures of 50 to  $150^{\circ}\text{C}$ , which is also consistent with the 8090/AMB material. These minor exothermic peaks are discussed further in Section 7.

#### **6.2.3 Summary of DSC analysis for Al-Li 8090 main lift frame material.**

The DSC analysis has shown that the precipitation events that occurred in the Al-Li main lift frame material are broadly consistent with findings reported in the literature for the same alloy, across a number of different heat treatment conditions. The 8090/SHT material (Figure 76), was used to provide a comparison with Starink et al.'s (2000)<sup>124</sup> solution heat treated Al-Li 8090 DSC curve and associated endothermic and exothermic reactions, (Figure 74).

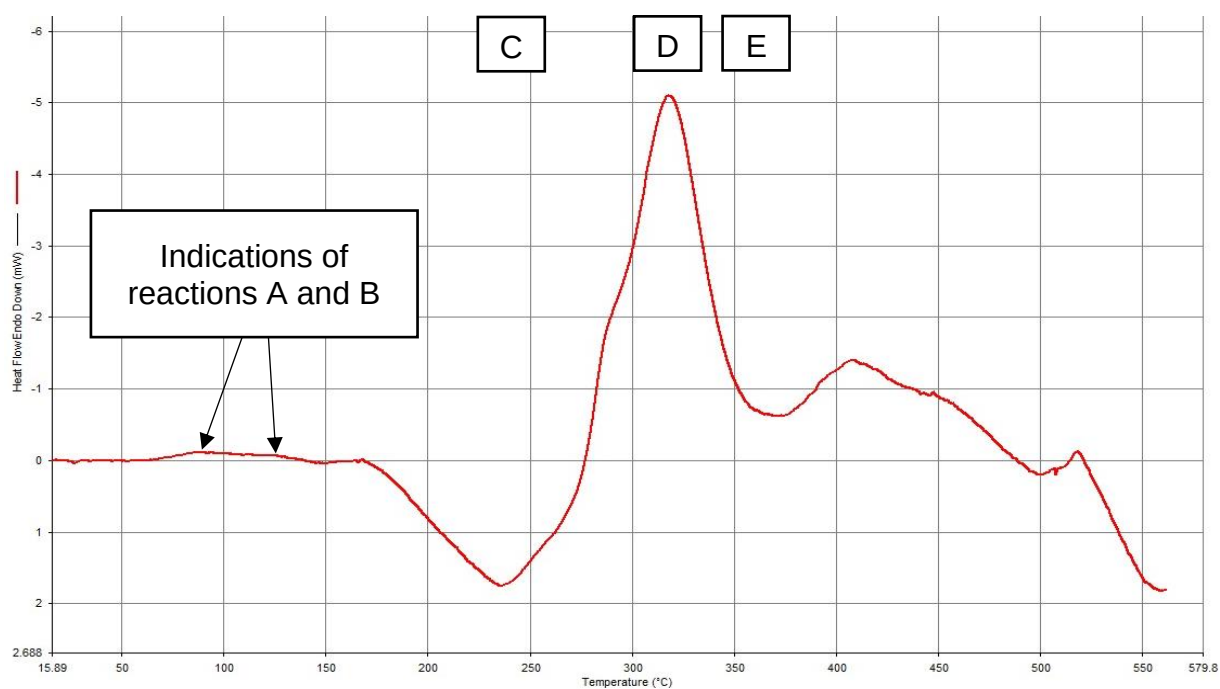


**Figure 78:** Showing the DSC curve the 8090/53.1°C/24hrs material.

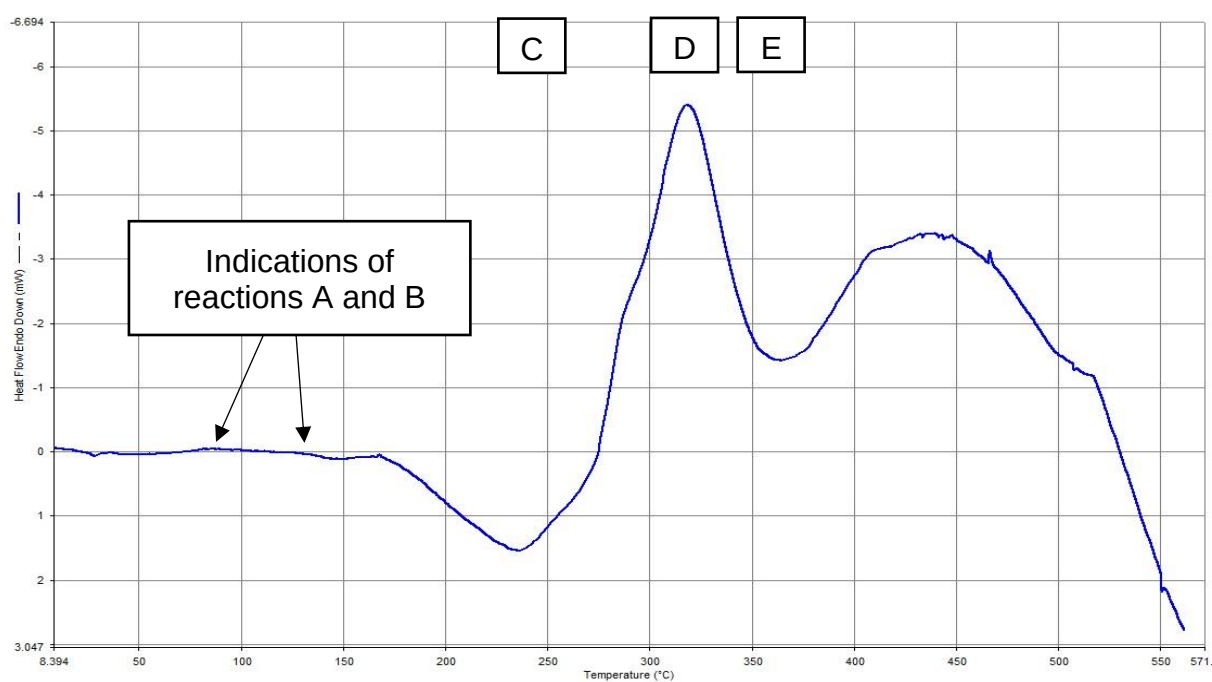


**Figure 79:** Showing the DSC curve for 8090/53.1°C/36hrs material.

This 8090/SHT DSC curve provided evidence to support;

1. The formation and growth of GP zones (reaction A).
2. An indication of the formation and growth of  $\delta'$  precipitates (reaction B).
3. The dissolution of GP zones and  $\delta'$  precipitates (reaction C).
4. The precipitation of S precipitates (reaction D).
5. The dissolution of S precipitates (reaction E).

DSC analysis of the 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials gave consistent thermal behaviour for reactions C, D and E. Being heat treated to an artificially aged temper state, the DSC curves for reactions A and B for all three aforementioned material conditions (Figure 77, Figure 78 and Figure 79, respectively) gave results that were more consistent with the DSC results reported artificially aged for T81 temper material (Figure 75), which was as to be expected. However, where there was no indication of any exothermic behaviour for reactions A and B in Starink et al.'s (2000)<sup>124</sup> T81 temper material, the 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials all exhibited indications of exothermic behaviour for reactions A and B between 50 and 150°C. As reactions A and B are attributed to the growth and/or formation of GP zones and  $\delta'$  precipitates, the indications of exothermic behaviour suggest that these formations are occurring in the Al-Li 8090 main lift frame material that has been heat treated at 53.1°C. Further investigation and discussion of these findings and the potential effect on the mechanical properties of the Al-Li main lift frame material is discussed in Section 7.



## **7 Discussion.**

### **7.1 Comparison of as-received Al-Li 8090 main lift frames and historical Al-Li 8090 data.**

Characterisation testing of 8090/AMB material has shown that the elemental chemical and mechanical properties of the in-service material were consistent with data provided from a number of sources (Rioja and Liu (2012)<sup>10</sup>, Smith (1988)<sup>13</sup>, Abdel-Latif, et al. (1988)<sup>14</sup>, Roth and Bright (1985)<sup>98</sup> Edgar, Starke and Quist(1985)<sup>99</sup> Prasad, Gokhale and Wanhill (2013)<sup>100</sup>). Although there are a number of other properties that could also be characterised (fracture toughness, fatigue strength, etc) the aforementioned properties were deemed to be the most relevant for this project.

Interestingly, it appears that the concerns raised regarding the mechanical properties of Al-Li 8090 (QETE 1988)<sup>14</sup> which were also documented by Smith (1988)<sup>13</sup> do not seem to have been rectified with an increase in magnesium over the original material specification, as was proposed in both aforementioned references. This addition of Mg was also anticipated by Roth and Bright (1985)<sup>98</sup> and Edgar, Starke and Quist (1985)<sup>99</sup>. However, as is shown in Table 10, an increase in the Mg content was not implemented for the 8090/AMB material used during this investigation, and is not the case for Al-Li 8090 T852 main lift frame material that is currently in-service. Therefore, there is no indication of the 'superior ductility properties' as proposed by Smith (1988)<sup>13</sup>.

Microstructural examination of the Al-Li main lift frame revealed a partially recrystallised microstructure. This was consistent with what would be expected for a dual stage heat treatment Al-Li alloy with small additions of Zr and 2-5% compression which is used to inhibit recrystallisation (Palmer et al. (1985))<sup>54</sup>. The primary source of historic data regarding the mechanical properties of Al-Li 8090 which most closely matches the alloy investigated in this work has been taken from Smith (1988)<sup>13</sup>, (Figure 24). The data (UTS and 0.2% proof stress vs elongation) in Smith's results is displayed in such a manner that only the range of the results is presented, giving solely minimum and maximum values. It should be noted that the actual positioning of data points is not given. An estimated value for the minimum and maximum results taken from Smith's graphs is given in Table 17.

Therefore, this does not completely portray the weighting of the test results towards either the minimum or maximum values of the aforementioned range. As an example, it is entirely possible (although unlikely) that the majority of Smith's longitudinal elongation results fall below the specified minimum value for elongation.

It is the opinion of the author that the way in which the data is presented is uncommon and the style has not been found to be repeated anywhere else in the references used in this project. However, the consistencies between the range of scatter in Smiths' data (Figure 24), and the scatter in the ambient temperature (8090/AMB) tensile test results undertaken in this work (Figure

34 and Figure 35) requires discussion. The scatter, particularly in the elongation results for the longitudinal orientation (graph C, Figure 34) was such a notable feature that additional testing (Group H, Figure 43) was undertaken to determine whether sample preparation and/or changes in cross-sectional area were variables that were having an effect on the scatter. Additional testing has shown that extra care in preparation and consideration of changes in cross-sectional area (depending on the location of the test piece in relation to the main lift frame) had no effect in reducing the scatter. The average 8090/AMB tensile test results, (without the Group H results as this Group was only tested the longitudinal orientation) can be seen in Table 16.

Therefore, the variation in the mechanical testing results was postulated to be directly related to the manufacturing process (forging) and the partially recrystallised nature of the microstructure of the Al-Li main lift frame material and not test piece preparation. In addition, the main lift frames have operated for a minimum of ten years in a myriad of climates. The latter includes exposure to temperatures which, following the findings of this work have the potential to change the mechanical properties of the material. This is a metastable change and long-term exposure to ambient temperature has the potential to revert the changed properties (Pitcher, (1993)<sup>81</sup>.

Therefore, combining this with the manufacture and resulting variable structure of the Al-Li main lift frames, it is understandable why some scatter would be expected in the results. Ultimately, it would be anticipated that a more

uniformly controlled anisotropic microstructure such as that provided in rolled sheet, would provide a more repeatable set of results. Procurement and testing of Al-Li sheet material was not within the scope of this project – however Al-Li 8090 sheet is used in the construction of the skin of the EH101 helicopter and could be consideration for further work.

Comparison of the mechanical property results for both orientations against the estimated findings of Smith's work can be seen in Table 17. With regard to meeting minimum specification requirements, it can be seen that the main-lift frame material meets the UTS and 0.2% proof stress requirements and falls short in the elongation requirement. However, the failure to meet the minimum specification requirement is subject to a number of interpretations.

**Table 16: Showing the complete average mechanical properties of the 8090/AMB material tensile test pieces from this work.**

| Longitudinal Orientation |        |                   |            |
|--------------------------|--------|-------------------|------------|
| Mechanical Properties    | UTS    | 0.2% proof stress | Elongation |
|                          | (MPa)  | (MPa)             | (%)        |
| Specification            | 425    | 340               | 6.0        |
| Experimental Value       | 496    | 394               | 5.1        |
| % Change                 | ↑16.7% | ↑15.9%            | ↓15.0%     |

| Long-Transverse Orientation |        |                   |            |
|-----------------------------|--------|-------------------|------------|
| Mechanical Properties       | UTS    | 0.2% proof stress | Elongation |
|                             | (MPa)  | (MPa)             | (%)        |
| Specification               | 405    | 325               | 3.0        |
| Experimental Value          | 506    | 402               | 4.4        |
| % Change                    | ↑24.9% | ↑23.7%            | ↑46.7%     |

**Table 17: Comparison of the minimum and maximum mechanical property results for Smith (1988)<sup>13</sup> and the 8090/AMB material.**

|              | Longitudinal Orientation    |     |      |                |     |      |           |      |      |
|--------------|-----------------------------|-----|------|----------------|-----|------|-----------|------|------|
|              | UTS (MPa)                   |     |      | 0.2% P.S (MPa) |     |      | Elong (%) |      |      |
|              | Min                         | Max | Spec | Min            | Max | Spec | Min       | Max  | Spec |
| Smith (1988) | 468                         | 535 | 425  | 380            | 465 | 340  | 3.0       | 12.0 | 6    |
| Current Work | 449                         | 568 |      | 354            | 479 |      | 2.5       | 7.0  |      |
|              | Long-Transverse Orientation |     |      |                |     |      |           |      |      |
|              | UTS (MPa)                   |     |      | 0.2% P.S (MPa) |     |      | Elong (%) |      |      |
|              | Min                         | Max | Spec | Min            | Max | Spec | Min       | Max  | Spec |
| Smith (1988) | 435                         | 530 | 405  | 340            | 465 | 325  | 2.5       | 11.5 | 3    |
| Current Work | 474                         | 544 |      | 352            | 450 |      | 2.5       | 7.3  |      |

The LT elongation results, Table 17 (minimum 2.5% for both sets of data) would commonly be interpreted as a pass in a manufacturing environment. It is not uncommon for elongation percentages to be rounded to the nearest whole number and the results in Table 17 would likely be presented as 3% and therefore meeting minimum requirements.

Futhermore, there is a scatter factor associated with all tensile data results. For elongation, this scatter is dependent on material thickness and data specifically attributed to an individual tensile testing machine. At the time of testing the scatter range for elongation for the testing in this project was  $\pm 0.4\%$ . By taking this scatter range into account (as shown in Figure 35c) if only the results were considered, then two samples failed to meet the specification. If values at the top end of the error bars were employed, only one sample falls

below the specification but if the lower end of the error bars was considered, six test pieces would fall outside of the specification requirement.

Analysis of the current 8090/AMB L elongation results (using Figure 43 as it provided a greater data set of tensile test results) gives a 43% pass rate. Therefore, there is a notable reduction in ductility across the range of test pieces. The minimum elongation value is similar to the findings of Smith. Analysis of the maximum UTS results for the current work revealed an increase by comparison with Smith's results (the 0.2% proof stress gives an increase for L but a slight decrease in comparison with the LT) indicating that an age hardening process may have occurred over time.

This is further reflected in the maximum elongation results for both orientations for the current work as they are 42% and 37% (L and LT respectively) less than what was reported by Smith. With an overall increase in strength by age hardening, a reduction in ductility would be anticipated. It is known that long-term natural aging of aluminium alloys does typically cause a slight increase in strength, Cochard, Zhu, Joulie, Douin, Huez, et al. (2017)<sup>129</sup>. This may not be noticeable on the more ductile alloys in the aluminium family. However, a slight increase in strength through natural age hardening is likely to have a noticeable effect on the elongation values of a material with an already low elongation specification such as the Al-Li main lift frame material.

In summary, characterisation testing of the 8090/AMB material has given comparable results with the historic data although indications of long-term age hardening are evident. In support of this, an increase in maximum ultimate tensile strength is associated with a loss of elongation. Given the in-service life of the material where it is fitted in current platforms, this work is likely to be the most accurate example of the condition of the real-world application that is currently available.

## **7.2 Discussion of low temperature testing findings.**

### **7.2.1 The effect of low temperature heat treatment on Al-Li 8090 main lift frame material.**

As is shown in the mechanical property testing results for the 80.0°C tensile test group (Figure 38), low temperature exposure (<100°C) of the in-service Al-Li main lift frames, specifically 1000 hours exposure at 80.0°C was sufficient to increase the strength and reduce the ductility of the material (graphs A and C, Figure 38). When compared against the average 8090/AMB data, the strength increase of 1.9% UTS and 7.2% 0.2% proof stress, are relatively minor in comparison to the 36.1% reduction in elongation. Using data provided by the UK MoD deployable Met Office<sup>107</sup> and DSTL<sup>109</sup> (Figure 37), an accurate temperature of an internal airframe structure operating in a hot environment has been determined (53.1°C). This representative temperature has also been shown to be sufficient to affect all three mechanical properties as discussed below.

### **7.2.2 Effect of exposure to 53.1°C on the mechanical properties of the Al-Li 8090 main lift frames.**

For the initial 1000 hours at 53.1°C exposure testing (Group A), the results gave an increase in strength compared with the average 8090/AMB data. These findings are consistent with the results of the 80.0°C testing group – albeit with a 8.1% and 16.4% increase for UTS and 0.2% proof stress, respectively and a 40.3% reduction in elongation for Group A. Combined with the results of the longer-term testing regime (Group C +1500 hours), it is clear that continual exposure to 53.1°C is capable of producing this strength increase in the Al-Li main lift frame material. It is clear from these results that there are fluctuations in tensile test performance. For example, the Group C, 0 hours test piece result was uncharacteristically high. But when the overall 8090/AMB data is averaged and compared with Groups A, B and C (Table 16), then the increase in strength and subsequent reduction in ductility becomes apparent.

Although this conclusion cannot be reached if only looking at the results for Group C as the 0 hours test piece result was uncharacteristically high. The increase in strength (and subsequent reduction in ductility) as a repeatable feature can only be seen when the average data values, (Table 16) are compared across the complete set of 8090/AMB results and compared with the data for groups A, B and C, Table 18. Further to this, the Group B testing results (1000 hours soaking at 53.1°C and a further 1000 hours exposure at ambient temperature) gave an increase in UTS over the 8090/AMB data, but the average result was lower than that determined for Group A which were



tested immediately after the 1000 hour soaking at 53.1°C. Given that the Group B test pieces were manufactured from the same lift frame as Group A and subjected to the same heat treatment process in the same oven, the reduction in value for the Group B results can only have occurred as a direct result of the additional 1000 hours at ambient temperature. These findings correlate with both Pitcher (1993)<sup>81</sup> and McDarmaid (1993)<sup>84</sup> in that the main lift frame material composing the Group B test pieces have partially 'recovered' or 'reverted back' towards their ambient temperature condition.

The mechanical property results for Group C (+1500 hours at 53.1°C) were consistent with the findings of the previous testing groups, giving a 2.4% and 10.4% increase for UTS and 0.2% proof stress respectively, and a 43.2% reduction in elongation. The lower average UTS result for the Group C data is due to a lower than average result for the final test piece (C2-9). It is unknown however, whether this was an indication of a further change in mechanical properties post 1512 hours soaking at 53.1°C. A summary of the change in the average data value for the mechanical properties of the 80.0°C, A, B and C testing groups is presented in Table 18. As can be seen in Table 18, a decrease in elongation is shown for all four groups. Whilst there is a 7% difference in elongation between the results for the 80.0°C, Group A and Group C results, this is equivalent to 0.4% elongation which is well within the scatter of the data, (Figure 46). This data provides evidence to support the hypothesis that the reduction in elongation resulting from low temperature

exposure to the Al-Li main lift frame material is a repeatable mechanism that can be shown in laboratory testing.

**Table 18: Showing the percentage change in average mechanical properties for four testing groups (80.0°C and Groups A, B and C) compared against the 8090/AMB tensile test results.**

| Test Group | UTS   | 0.2% P.S. | % Elong. | Comments                                                                                    |
|------------|-------|-----------|----------|---------------------------------------------------------------------------------------------|
| 80.0°C     | ↑1.9% | ↑7.2%     | ↓36.1%   | 1000 hours exposure to 80.0°C.                                                              |
| Group A    | ↑8.1% | ↑16.4%    | ↓40.3%   | 1000 hours exposure to 53.1°C.                                                              |
| Group B    | ↑4.9% | ↑10.6%    | ↓15.8%   | 1000 hours exposure to 53.1°C and an additional 1000 hours exposure at ambient temperature. |
| Group C    | ↑2.4% | ↑10.4%    | ↓43.2%   | 1512 hours exposure to 53.1°C.                                                              |

The results of the Group C testing indicated that the reduction in elongation, as a result of exposure to 53.1°C occurred within the first 72 hours, (Figure 45). The evidence in support of this was the mechanical property results for the first test tensile test piece (C2-4) that was destructively tested after 72 hours. Therefore, to further explore effect of exposure to 53.1°C on the mechanical properties of the Al-Li main lift frame material within the 0 to 72 hour period, a tensile testing regime with Groups E1, F1 and G1 was undertaken. The mechanical property test results for Groups E1, F1 and G1 (tested in the longitudinal grain orientation) are shown in Figure 80.

The UTS results for all three groups clearly show a pattern of increasing strength after 24 hours (Group E1 reached peak strength after 12 hours), with a sharp reduction after 36 hours and a partial recovery after 48 hours. For all

three groups, the partial recovery in strength following 48 hours at 53.1°C did not equal the peak strength that had been recorded prior to the 36 hours testing.

The 0.2% proof stress results for the three groups show an increase in scatter with by comparison with the UTS results, with Groups F1 and G1 showing a similar increase/decrease/partial increase in strength after the same time periods as the UTS results. The Group E1 results gave a slight shift in results, with an almost 12 hour delay in the anticipated change in strength. In addition, the curve followed a steady decrease in strength until after 48 hours, where the partial increase occurred. Although it was noted that the 0 and 72 hour results for the three groups gave very similar results.

The elongation results for the three groups gave interesting results, with a general initial increase after 12 hours, followed by a decrease after 24 hours (notably where the UTS reaches peak strength), and an increase at 36 hours for Groups F1 and G1 and at 48 hours for Group E1. For all three Groups, the pattern in the elongation curves approximately mimic the results for the UTS and 0.2% proof stress, giving reductions in strength where elongation increases and vice versa. Apart from Group E1, which gave an uncharacteristic UTS curve, the behaviour appears to be more strongly related to the 0.2% proof stress curve.

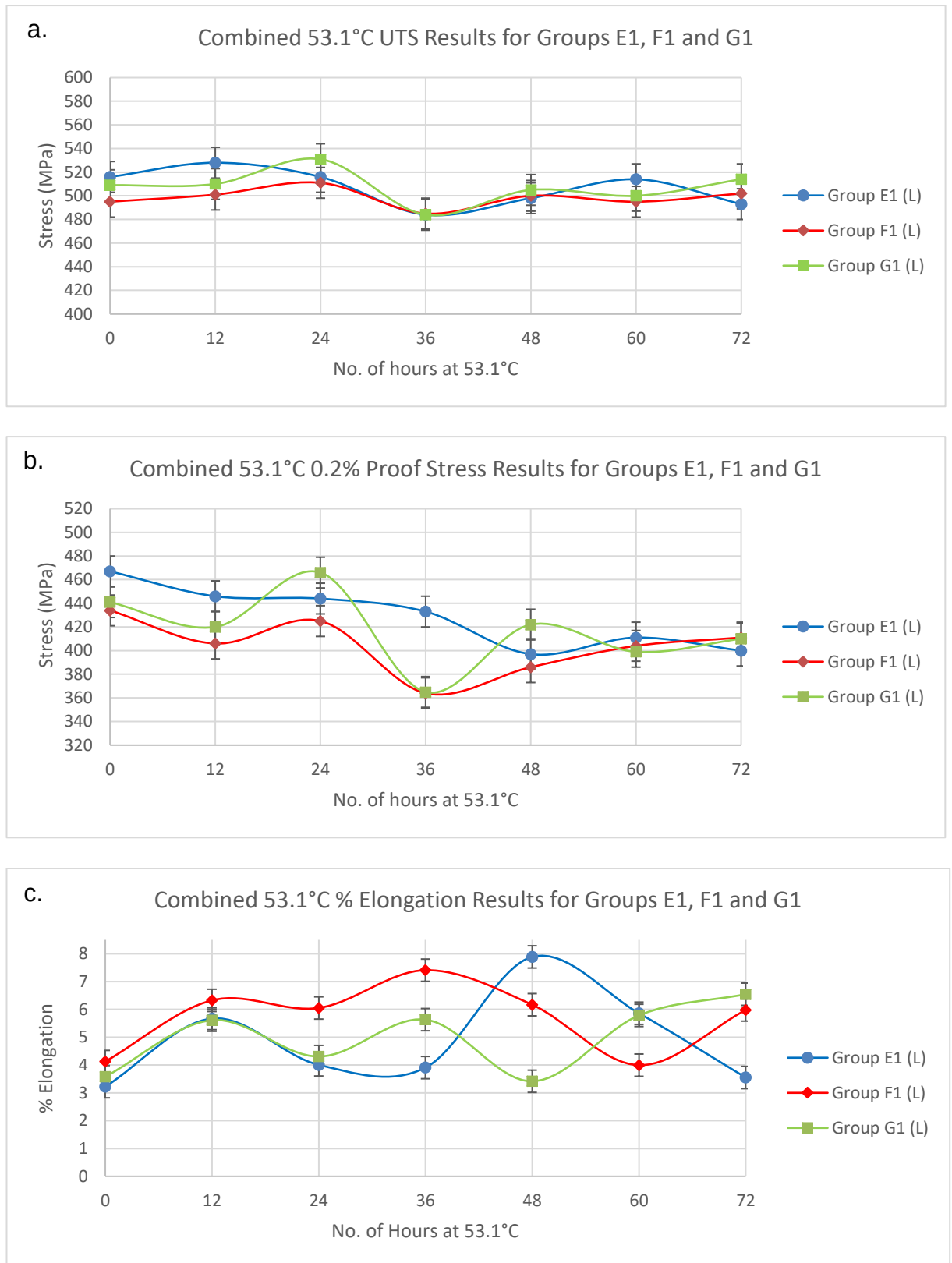


**Figure 80: Comparing the mechanical property results for tensile test samples from groups E1, F1 and G1. a) UTS, b) 0.2% proof stress, c) elongation.**

However, the mechanical property behaviour for all three groups becomes clearer when observing the average data curve as shown in Figure 81. As can be seen in Figure 81, the defined increase in strength following 24 hours exposure at 53.1°C is accompanied by a reduction in the elongation. Notably the loss in strength after 36 hours does not appear to have had a similar deleterious effect on the elongation as the increase after 24 hours. It should be noted, that in contrast to the 8090/AMB results, interrogation of the data for all three testing Groups revealed sub-average elongation results for their respective 0 hours test pieces. These results were less than 82% of the 8090/AMB test results, including testing Group H, Figure 43.

### **7.2.3 Discussion of the potential strengthening mechanism affecting the mechanical properties of the Al-Li main lift frame material.**

As has been previously mentioned, the analysis of the average mechanical property testing data of the Al-Li main lift frame material as shown in Figure 81, reveals a pattern of increase/decrease/partial recovery of strength. Therefore, to determine what phenomena was responsible for the change in strength, consideration of the normal strengthening mechanisms will be considered here;

- 1.) *Work hardening.* There is no work hardening of the material during the 53.1°C soaking process and therefore it is not considered to be a factor.

2.) *Radiation hardening*. Radiation is not considered to be a factor in this work.

3.) *Grain boundary strengthening*. The relative heat input of 53.1°C is considered to be far too low to cause any change in grain size and therefore is not considered to be relevant in this analysis.

4.) *Dispersion hardening*. Although the principle of dispersion hardening is the same as precipitation hardening (i.e. impedance of dislocations by the introduction of particles in the matrix), the process by which it is achieved (i.e. introduction of insoluble particles by powder metallurgy) is not relevant to this work. However, *precipitation hardening*, will be discussed further.

#### 7.2.3.1 **The potential for strengthening of Al-Li main lift frame material by precipitation hardening.**

Precipitation hardening is achieved by taking precipitates and the elements forming them into a solid solution at a high temperature, quenching and aging at a lower temperature. Ambient temperature aging is also known as *natural aging*. A second heat treatment at a temperature lower than the solid solution temperature but higher than ambient, is termed *artificial aging*.

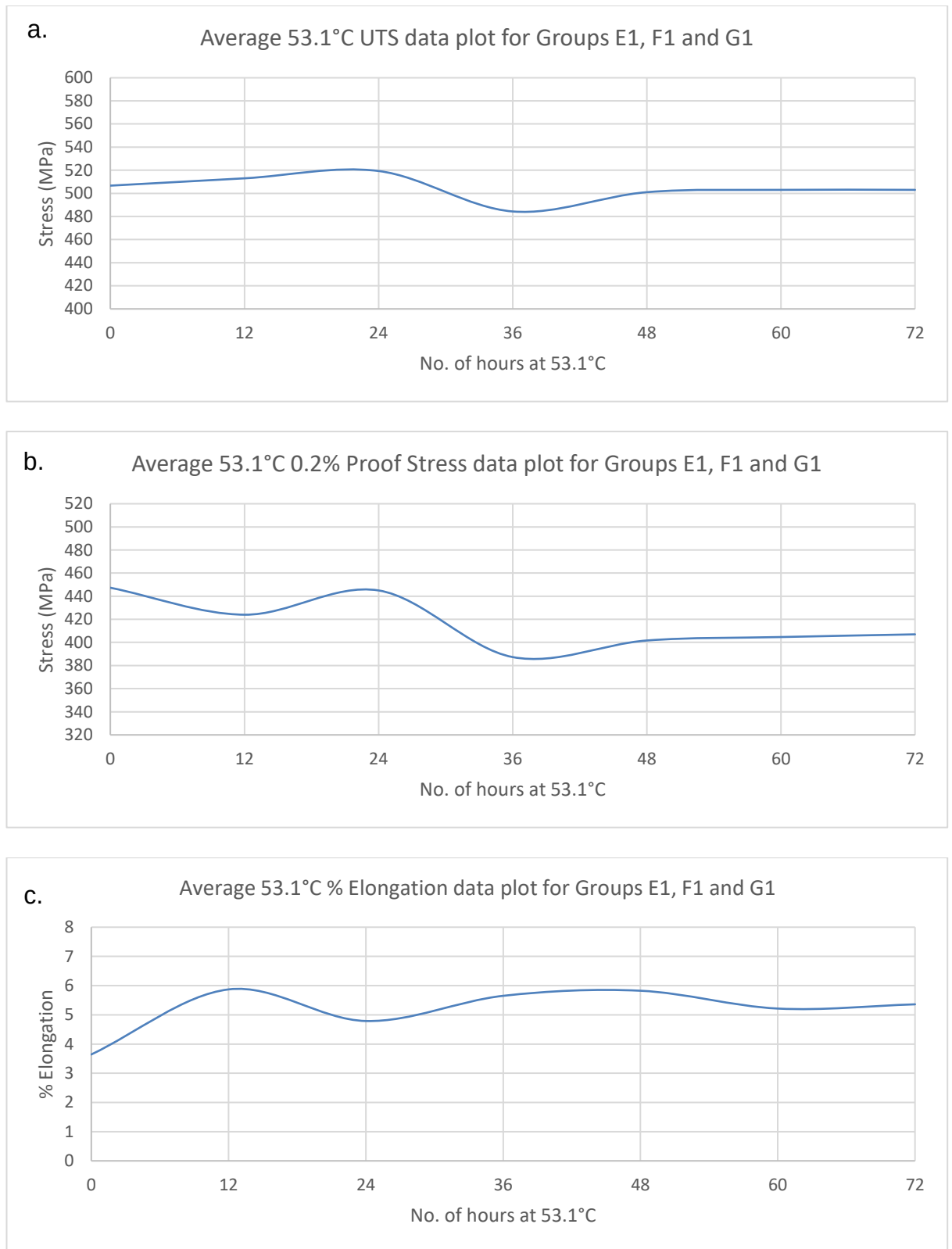


**Figure 81:** Showing the average mechanical property results for Groups E1, F1 and G1. a) UTS, b) 0.2% proof stress, c) elongation.

Precipitation hardening is a standard strengthening process for a range of heat-treatable Al alloys, with the 2xxx and 7xxx series relying on this process for their peak strength. This is also the primary method used to obtain the desired mechanical properties in Al-Li 8090, although cold working in between heat treatments is used to further enable the formation of heterogeneously distributed S precipitates during artificial aging.

As detailed in Section 1.7, the solution heat treatment (SHT) temperature for Al-L 8090 main lift frames is specified as 525-535°C for two hours followed by a water quench and the artificial aging heat treatment to obtain the T852 temper state (175°C for 20-50 hours), Figure 6. With regards to the formation of new precipitates, exposure to 53.1°C would initially, seem to be far too low a temperature for any notable precipitation effect to occur in Al-Li 8090. Additional data in support of this is given in the DSC curve for Al-Li 8090 material in the T81 temper condition, presented by Starink et al. (2000)<sup>124</sup> (Figure 75), which gave no indication of an exothermic event in the 20-200°C temperature range that incorporates the exothermic events for the formation and growth of GP zones and  $\delta'$  precipitates (reactions A and B, Figure 74). Conversely, the T81 DSC curve gave an indication of an endothermic dissolution event initiating after ~ 50°C.

As was highlighted in Section 6.1, the absence of any exothermic events between the 20-200°C temperature range is explained by the fact that in the peak aged T81 temper condition, the material is intended to have utilised all



of the available solute from the solid solution to maximise the formation of  $\delta'$  precipitates for optimal strength in the material. Therefore, there should not be any available solute available for additional exothermic reactions to take place.

However, it is clear that this is not the case for the 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials, (Figure 77, Figure 78 and Figure 79, respectively) which all gave indications of exothermic reactions between 20-175°C in their respective DSC curves. As the main lift frame material is in an artificially aged condition (T852 temper state), it would be anticipated that DSC analysis of newly heat treated main lift frame material should have maximised the formation of  $\delta'$  precipitates and hence, would exhibit a DSC curve with no indications of exothermic reactions between the 20-175°C temperature range, as per the newly heat treated T81 temper material given by Starink et al. (2000)<sup>124</sup>, Figure 75.

Therefore, the major difference between the DSC curves for the material in the T81 temper condition presented by Starink et al. (2000)<sup>124</sup> and the 8090/AMB material investigated in this work is the length of time between completion of the artificial aging heat treatment and initiation of the DSC analysis, which was 5 minutes for the T81 temper material and a minimum of a decade for the 8090/AMB material. This suggests that the in-service timeframe for the 8090/AMB material has provided suitable conditions to enable solute to become available in the 8090/AMB material during operational usage. For this

available solute to be present in the 8090/AMB material, one of the following would have had to occurred:

1. The solute had been present in the material from manufacture. This would indicate that the heat treatment process had not maximised the formation of the  $\delta'$  precipitates. As has been discussed, the DSC curve for Starink et al.'s (2000)<sup>124</sup> T81 temper material would indicate otherwise. It could be argued that artificially aging sheet material presents a much less complex geometry and reduced thickness for heat treatment in comparison to a forged main lift frame. However, the artificial aging time for the main lift frame is 20-50 hours and combined with the aforementioned thermal conductivity of Al, is sufficient to ensure suitable heat treatment of the thickest parts of the main lift frame.
2. In-service usage of Al-Li main lift frame material, has enabled dissolution of solute into solid solution. Whilst it is not anticipated that the entire length of time in service is be responsible for the dissolution event, it could be surmised that the potential exists for multiple opportunities for low temperature exposure of the main lift frame material to occur, which over time, could have an accumulative effect on the available amount of solute in the 8090/AMB material.

### 7.2.3.2 Indications of endothermic dissolution events occurring in the DSC results for Al-Li alloys in the 20-200°C temperature range.

Starink et al. (2000)<sup>124</sup>, bringing together the efforts of a number of workers (Gomerio, (1991)<sup>34</sup>, Pitcher, Stewart and Gupta, (1991)<sup>58</sup>, Starink, Hobson and Gregson, (1995)<sup>127</sup>, Starink and Gregson, 1996)<sup>126</sup>), determined that low temperature dissolution events for Al-Li alloys can potentially occur during long term low exposure temperature at 70°C. The primary reason for agreement with the literature by Starink et al. (2000)<sup>124</sup> was the DSC data curve for Al-Li 8090 material in the T3 and T3+X temper conditions (X = 1000 hours at 70.0°C), in which both respective DSC curves gave evidence of dual endothermic reactions occurring between 50 and 270°C, Figure 82. This event was further split into a 'primary' dissolution event being defined as occurring between 200 to 270°C and a 'precursor' dissolution event occurring between 50 and 175°C.

The primary dissolution event which was consistent with reaction C, Figure 74 was found to be present for all of the of Al-Li 8090 main lift frame materials that have been investigated in this work (8090/SHT, 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs), and was clearly represented in the results for the solution heat treated 8090 material (Starink et al., 2000)<sup>124</sup>, Figure 74. Starink et al. (2000)<sup>124</sup> proposed that the reason for the earlier 'precursor' dissolution event (between 50 to 175°C) was the result of a combination of the following six DSC reactions (i – vi), that were originally

determined by Starink and Gregson (1996)<sup>127</sup> (not to be confused with the 5 A-E reactions in Figure 74) .

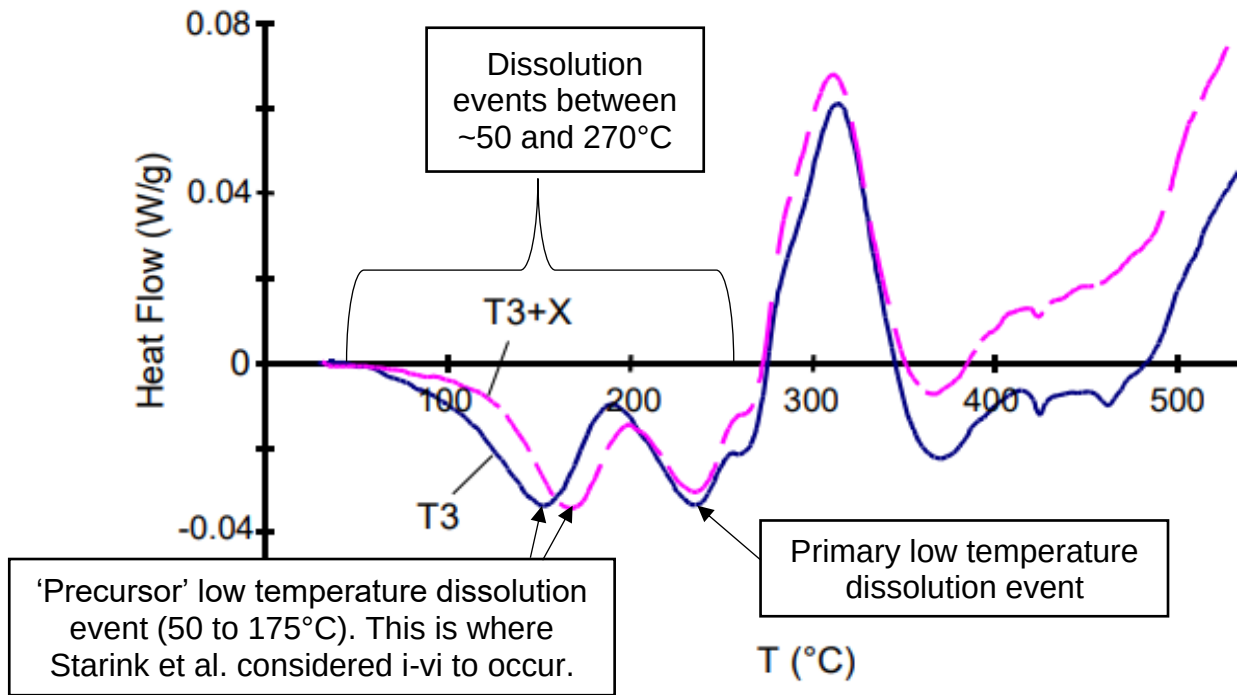


**Figure 82: Showing a DSC for 8090 T3 and T3+X materials Starink et al. (2000)<sup>124</sup>.**

- i) Dissolution of precursors to  $\delta'$  (an endothermic event).
- ii) Dissolution of very fine  $\delta'$  precipitates that become unstable on increase of temperature (an endothermic event).
- iii) Further ordering of  $L1_2$  ordered domains (an exothermic event).
- iv) Changes in the initially diffuse interface layer between  $\delta'$  and the matrix leading to a sharper interface (an exothermic event).
- v) Coarsening of the  $\delta'$  precipitates (an exothermic event).
- vi) Dissolution of GP zones/clusters (an endothermic event).

In this work, Starink et al. (2000)<sup>124</sup> disregarded the  $\delta'$  precipitate precursors (as these have not yet been observed, only hypothesised) and coarsening of the  $\delta'$  precipitates as the exothermic energy produced by the coarsening effect was considered to have a negligible impact on the DSC curve. Therefore, it was deemed that the 'precursor' dissolution effect was the result of a combination of reactions ii), iii), iv) and vi). It should be noted that Starink et al. (2000)<sup>124</sup> attribute the exothermic peak (clearly visible between the precursor and primary endothermic dissolution events) to the formation of  $\delta'$  precipitates.

DSC analysis reported upon in the literature of artificially aged Al-Li 8090 T81 and T81+X materials (X = 1000 hours at 70°C, Figure 83), did not provide evidence of distinct separate dual endothermic events as was evident for the T3 and T3+X materials. As can be seen in Figure 83, the T81 material does not appear to initiate the endothermic event until temperatures higher than 100°C, although a very slight endothermic dip does appear on the curve between 50 and 100°C for the Al-Li 8090 T81+X temper, (Figure 84). The absence of the exothermic event can be explained the material being heat treated to an artificially aged condition, whereas the T3 material has not, therefore allowing for precipitation of  $\delta'$  precipitates to occur.

The primary observation of Starink et al. (2000)<sup>124</sup> with regard to the T81 and T81+X curves was the formation of two endothermic peaks (Figure 83), without any indication of an exothermic event between these peaks, as was

evident in the T3 and T3+X material, Figure 82. This inferred that low temperature exposure of artificially aged Al-Li 8090 T81 temper material did not result in the formation of any new precipitates which led Starink et al., (2000)<sup>124</sup> to conclude that the reported changes in UTS in the Al-Li 8090 T81 alloy was a result of changes to the volume fractions of GP zones and  $\delta'$  precipitates in the T81 material.

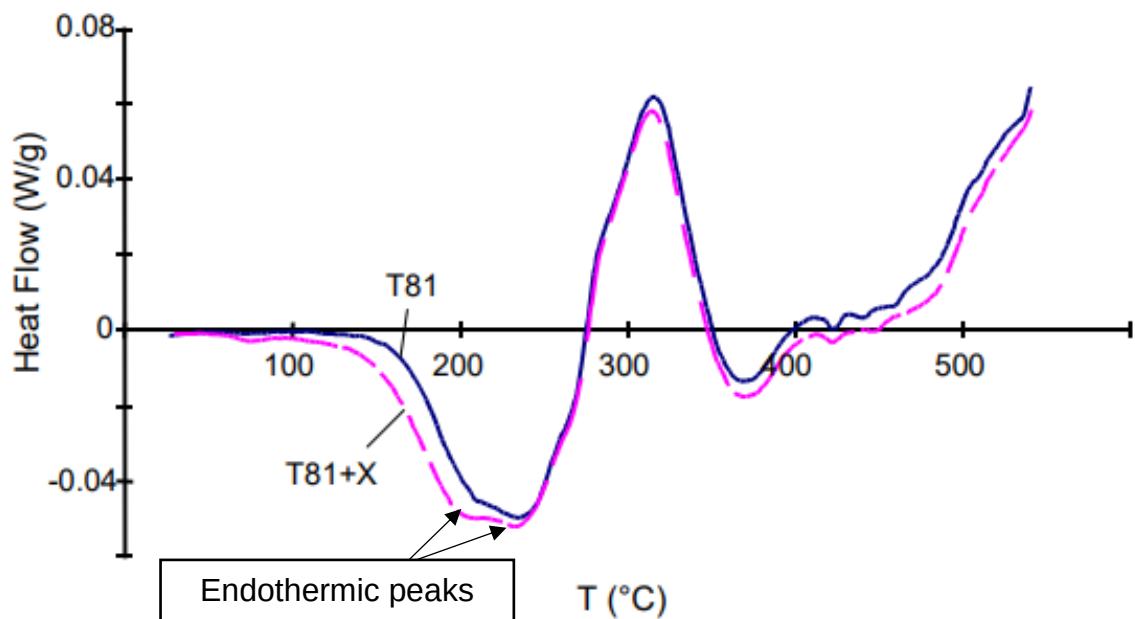


**Figure 83:** Showing DSC curves for age hardened Al-Li 8090 sheet (X = 1000 hours at 70°C) Starink et al. (2000)<sup>124</sup>.

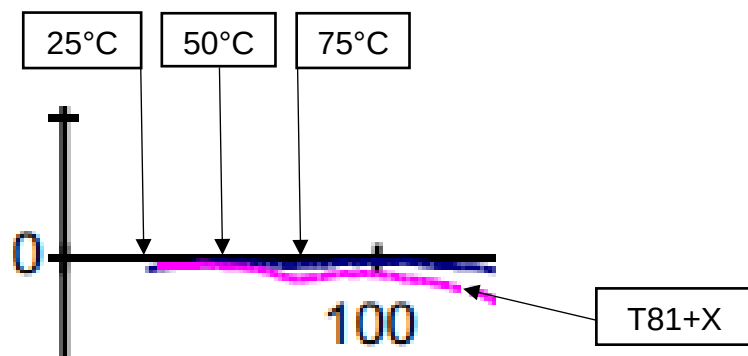


**Figure 84:** Showing an enhanced view of the Al-Li 8090 T81+X DSC curve.

**7.2.4 Evidence in support of exothermic reactions (A and B) occurring in the 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials.**

The DSC curves for the 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials (Figure 77, Figure 78 and Figure 79, respectively) have shown repeatable data in support of exothermic reactions (A and B, Figure 74) having occurred prior to the main dissolution event of reaction C, which initiates at 175°C. However, the exothermic reactions for the 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials are better appreciated when all three DSC curves are compared together. A composite image showing the DSC curves for all four main lift frame material conditions investigated in this work, is shown in Figure 85. As can be seen in Figure 85, the four DSC curves have been aligned with two minor, but repeatable endothermic events that occur before reaction A at 25°C and after reaction B at 165°C, but prior to the beginning of the main dissolution event at reaction C at 175°C. An enhanced image of this region of interest is shown in Figure 86.

As can be seen in Figure 86, comparison of 8090/SHT DSC curve against the 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs DSC curves clearly shows the reduction in the exothermic energy generated as a result of artificial aging. As has been previously mentioned, reaction A (GP zone formation), and reaction B ( $\delta'$  precipitation), both require available solute atoms for the formation of the GP clusters and growth of the  $\delta'$  precipitates.

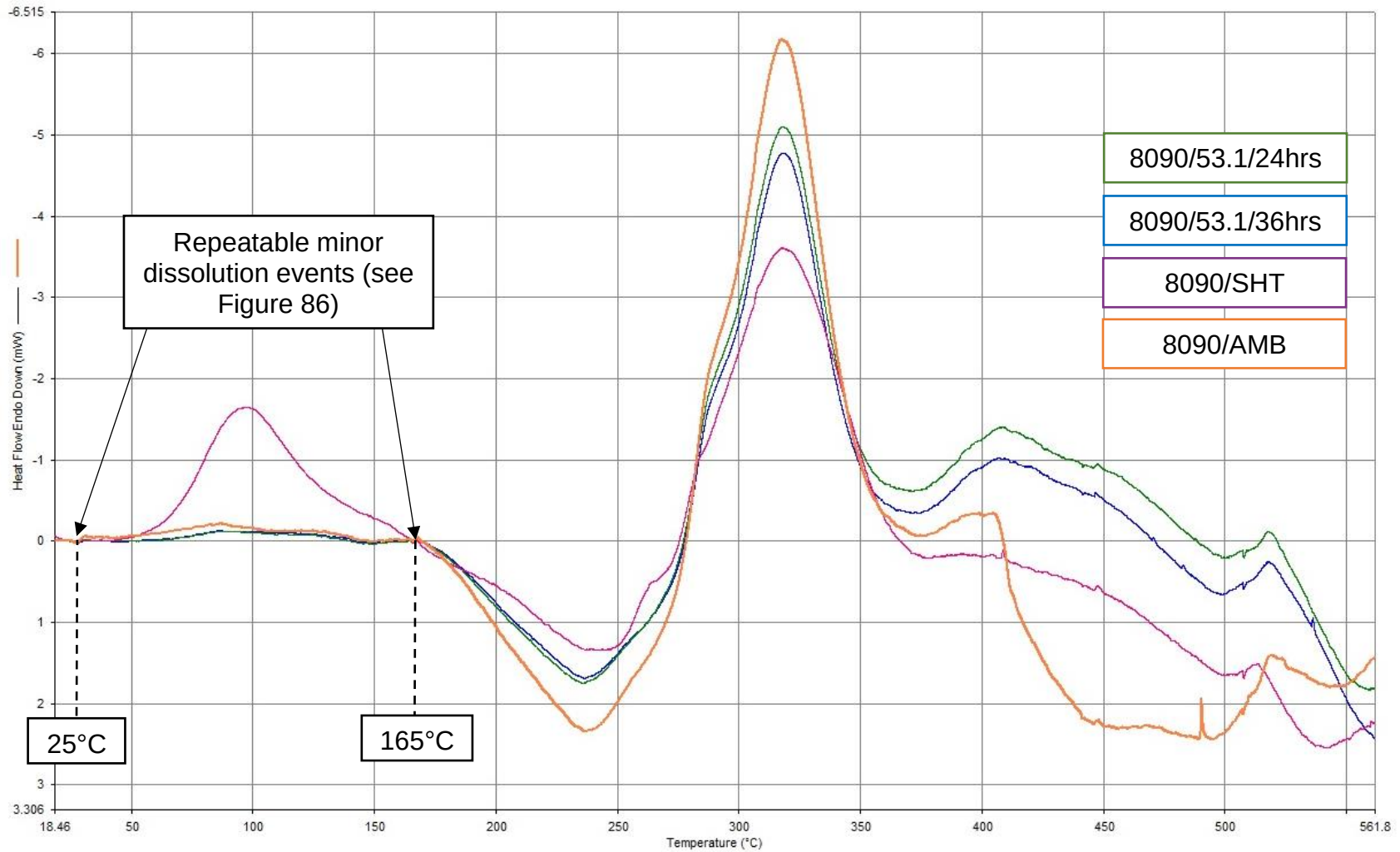


**Figure 85:** Showing the DSC curves for the 8090/SHT, 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials.



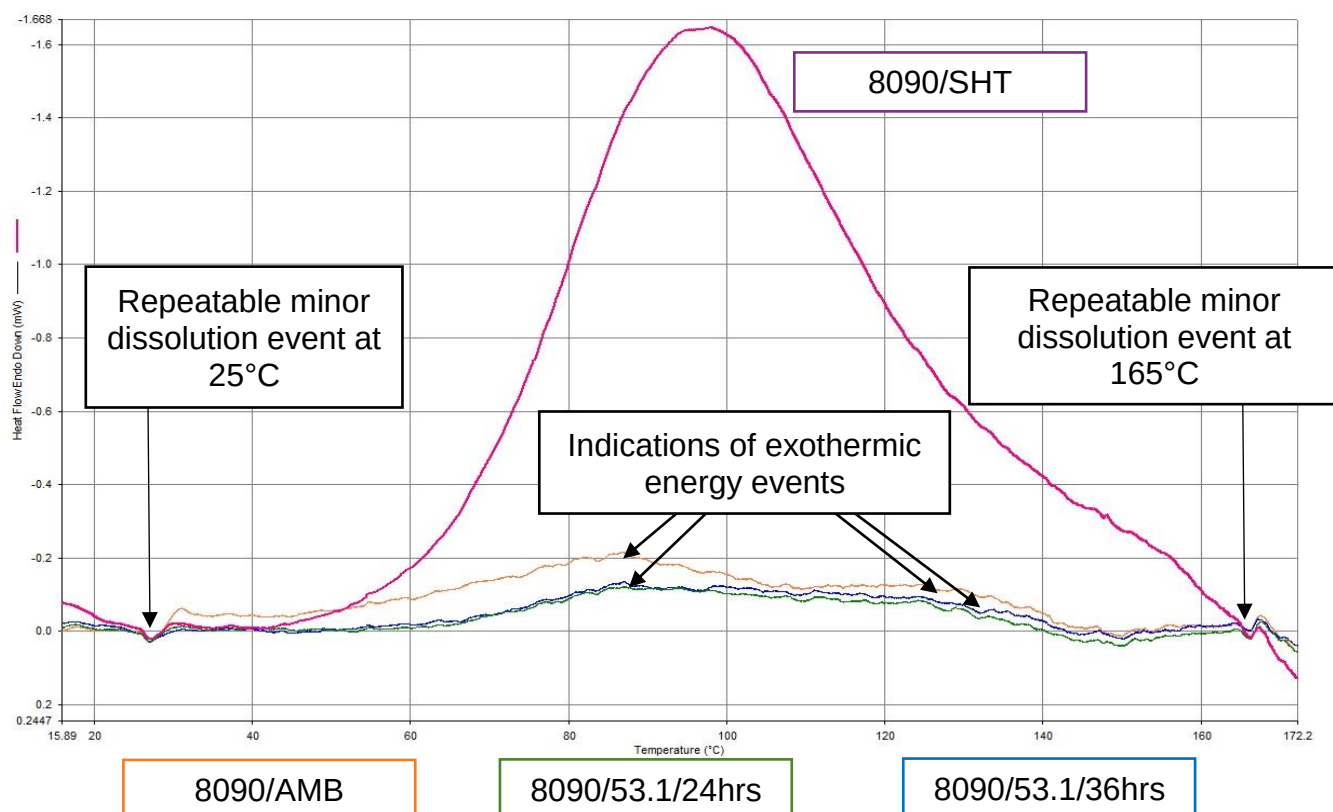


**Figure 86:** Showing a magnified view of the DSC curves for the 8090/SHT, 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials.

Further investigation of the exothermic events that occur for the 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials show that a reduction in the exothermic heat flow for the 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials when compared against the 8090/AMB material, Figure 87, i.e. there is a greater exothermic event recorded for the 8090/AMB samples than for the 8090/53.1°C/24hrs and 8090/53.1°C/36hrs samples.

This reduction in the heat flow for the exothermic events for the 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials indicate that the amount of the available solute that has been shown to be present in the 8090/AMB material had been utilised during the

time the samples have been heat treated at 53.1°C. Therefore, there is a reduction in heat flow for the exothermic events in the DSC curves for the 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials, Figure 87. Therefore, the low temperature heat treated samples have less solute available for exothermic precipitation because GP zones have already formed during the low temperature heat treatment.

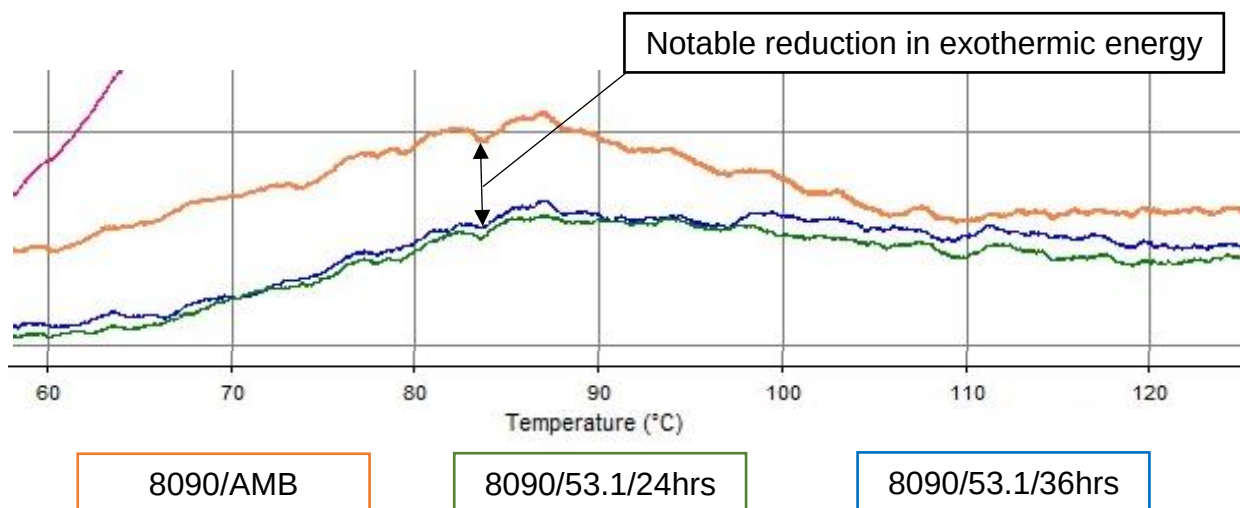


**Figure 87:** Showing an enhanced view for the DSC curves of the exothermic reactions for the 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials.

Additional explanation may be provided by Zakharov (2003)<sup>62</sup>. He has suggested that a retained residual supersaturation of alloying elements after quenching and aging, coupled with the low thermal stability of Al-Li alloys (below 100°C), enables the decomposition of the solid solution and the subsequent formation of additional  $\delta'$  precipitates. This in turn results in an '*undesirable change in mechanical properties*'. The DSC results for the 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials, for the temperature range between 25 and 165°C (reactions A and B, Figure 74) on the respective DSC curves, (Figure 86 and Figure 87) provides evidence to support the notion that decomposition of the solid solution has occurred. This solute

has to be available to enable the formation of GP zones and  $\delta'$  precipitates which is evident by the respective exothermic reactions. As this decomposition is stated to occur after quenching and aging and noting that all of the material in the current work has been in service for a minimum of a decade, this supports the theory that decomposition of the Al-Li main lift frame material has occurred during operational usage.

### **7.2.5 Consideration of the effect of the exothermic reactions on the 8090/AMB microstructure and mechanical properties.**

A number of authors Stewart and Gupta (1991)<sup>58</sup>, Nobel, Harris and Dinsdale (1996)<sup>28</sup> Zakharov (1999)<sup>64</sup> and (2003)<sup>62</sup>, Pitcher (1993)<sup>81</sup> Starink et al. (2000)<sup>124</sup>, have attributed the changes in the mechanical properties of Al-Li alloys at relatively low temperatures (between 70-150°C) to be either the result of coarsening or dissolution of the existing  $\delta'$  precipitates or the formation of new  $\delta'$  precipitates. Consideration of each mechanism is given below.

#### **7.2.5.1 Coarsening of existing $\delta'$ precipitates.**

Coarsening of the  $\delta'$  precipitates in Al-Li 8090 requires sufficient time at temperature which, as shown for the binary Al-Li alloys in the Time Temperature Transformation diagram, (Figure 70) 50% transformation of  $\delta'$  precipitates cannot be achieved in the time and at the temperatures employed in this work. Work by Nobel, Harris and Dinsdale (1996)<sup>28</sup> also supports this determination. Measurement of  $\delta'$  precipitate diameters for binary Al-Li alloys soaked at 70°C for 1000 hours revealed no increase in the  $\delta'$  precipitate diameters for both 1.7% and 2.6% wt Li alloys. These findings correlate with the  $\delta'$  precipitate diameter measurements undertaken in this work, using TEM, which revealed there was no change in  $\delta'$  precipitate diameters for the

8090/AMB and 8090/53.1°C/36hrs materials. Therefore, it is evident that, coarsening of the  $\delta'$  precipitates may not be attributed to the change in mechanical properties that has been identified for in Al-Li main lift frame material investigated in this work.

#### 7.2.5.2 Dissolution of existing $\delta'$ precipitates.

Dissolution of GP zones and  $\delta'$  precipitates the primary dissolution event (reaction C, Figure 74) has been shown to repeatably occur for the 8090/SHT, 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials investigated in this work, (Figure 85). However, for all four of the Al-Li main lift frame material conditions investigated, the primary dissolution event was shown to initiate at  $\sim 165^\circ\text{C}$ , which was at a higher temperature than the  $53.1^\circ\text{C}$  exposure temperature investigated in this work. However, the principle methodology of reversion age heat treatment of Al-Li 8090 in the T81 temper is a 5-10 minute heat treatment at  $190^\circ\text{C}$ , Pitcher (1993)<sup>81</sup>, McDairmaid (1992)<sup>83</sup> and (1993)<sup>84</sup>. This temperature places the reversion heat treatment within the primary dissolution event on the DSC curve for T81 temper material as shown by Starink et al. (2000)<sup>124</sup>, Figure 88. This would indicate that for the 5-10 minute heat treatment at  $190^\circ\text{C}$ , dissolution of the  $\delta'$  precipitates would occur. Therefore, it is likely that the smaller  $\delta'$  precipitates would dissolve into solution, increasing the average  $\delta'$  precipitate diameter, increasing the volume fraction of larger  $\delta'$  precipitates and reducing the volume fraction of the smaller  $\delta'$  precipitates which would inherently increase the resistance of the material to planar slip. This would reduce the UTS of the material, by allowing for more dislocation movement. However there would also be an increase in ductility as a result of the increase in dislocation movement, which would justify the reported toughness increases in reversion age heat treated Al-Li alloys (as detailed in Sections 2.3.4 and 2.3.5).

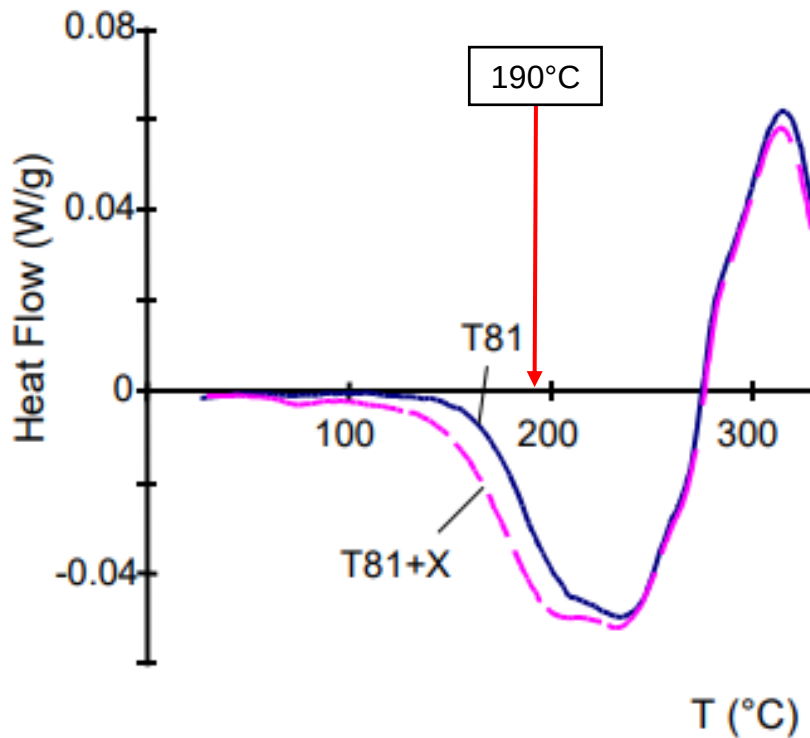


**Figure 88: Showing the location of the reversion heat treatment temperature (190°C) in relation to the GP zone and  $\delta'$  precipitate dissolution event (reaction C) for Al-Li T81 material, Starink et al. (2000)<sup>124</sup>.**

In the case of low temperature dissolution ( $< 100^\circ\text{C}$ ) whilst a 'precursor' dissolution event has been shown to initiate at  $\sim 50^\circ\text{C}$  in Al-Li 8090 material that has been heat treated to the T3 temper condition and subsequently exposed to 1000 hours at  $70^\circ\text{C}$  (T3+X) Figure 82, there was no indication of a similar such 'precursor' even in the T81 and T81+X DSC curves, (Starink and Gregson (1996)<sup>127</sup>). However, there was a slight endothermic 'dip' reported for the T81+X material at  $\sim 75^\circ\text{C}$ , Figure 83. Starink and Gregson's DSC curves (1996)<sup>127</sup>, show heat flow for materials subjected to  $70^\circ\text{C}$  for 1000 hours and indicated that a small, or partial dissolution event may occur at exposure to temperatures  $< 100^\circ\text{C}$ . For newly heat treated material, this minor dissolution event would likely have little to no noticeable effect on the mechanical properties of an artificially aged Al-Li alloy. However, if the dissolution effect was

cumulative over the significantly longer exposure period, this could provide the solute required for the formation of new GP zones, which would have a notable effect on the mechanical properties of the aforementioned Al-Li alloy. Therefore, as a standalone mechanism, dissolution of the existing  $\delta'$  precipitates in Al-Li main lift frame material exposed to temperatures  $< 100^{\circ}\text{C}$  is unlikely to have a substantial effect on the mechanical properties of the main lift frame material. However, the dissolution would provide solute for the precipitation of new GP zones and  $\delta'$  precipitates.

#### 7.2.5.1 Formation of new GP zones and $\delta'$ precipitates.

As previously mentioned in Section 2.1,  $\delta'$  precipitates are considered to be the main strengthening precipitate in Al-Li alloys, Whitman (1990)<sup>23</sup>. Precipitation and growth of GP zones and  $\delta'$  precipitates (to peak strength) is directly responsible for the increase in strength of Al-Li 8090. Although the  $\delta'$  precipitate diameter of  $\sim 24$  nm measured in this work is less than the critical diameter for Orowan looping to occur ( $\sim 50$  nm, Edgar, Starke and Quist, (1988)<sup>3</sup>), dislocation impedance by the sheer volume fraction of  $\delta'$  precipitates is considered by multiple authors to be an significant factor that provides the increase in strength and reduction in ductility, (Noble Harris and Dinsdale (1996)<sup>28</sup>, El-Aty, Xu Guo, [et al.] (2017)<sup>27</sup>, Pitcher Stewart and Gupta (1991)<sup>58</sup>, Starink et al. (2000)<sup>124</sup>). Hence, an increase in strength (and reduction in ductility) is likely to be the result of new  $\delta'$  precipitate formation. As previously mentioned, in support of this Noble, Harris and Dinsdale (1996)<sup>28</sup> noted that precipitate diameters did not increase in 1.7% and 2.6% wt Li Al-Li alloys, following 1000 hours at  $70^{\circ}\text{C}$ , although an additional fine dispersion of new  $\delta'$  precipitates or ordered GP zones with a measured diameter of 2 nm were resolved in the 1.7% Li alloy. A similar dispersion was also

present in the 2.6% Li alloy, (again following 1000 hours at 70°C), but measurement of the new  $\delta'$  precipitate diameters in the TEM was difficult due to the volume fraction of the larger native  $\delta'$  precipitates resulting from a 150°C pre-age heat treatment. Starink et al. (2000)<sup>124</sup> also determined that the effect of exposure to 70°C on T81 Al-Li 8090 sheet was primarily due to an increase in volume fraction of GP zones and  $\delta'$  precipitates.

Starink and Gregson (1996)<sup>127</sup> postulated that the formation of GP zones and  $\delta'$  precipitates were responsible for the exothermic events (reactions A and B) in the DSC curve for solution heat treated Al-Li 8090 material, Figure 74. These exothermic events have been shown to occur in the DSC curves for the 8090/SHT, 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials investigated in this work. Hence, the literature and the evidence presented in this work supports the theory that the increase in strength observed in the Al-Li main lift frame material exposed to 53.1°C over a range of times for tensile test groups A, B, C, E, F and G, is attributable to the formation of GP zones and/or  $\delta'$  precipitates. This is as a result of the presence of available solute from low temperature exposure, that has been found to be present in the 8090/AMB material. This is suspected to be due to precipitate dissolution.

In summary, the change in mechanical properties in the Al-Li main lift frame material exposed to 53.1°C is considered to be the result of both; initial dissolution and subsequent precipitation of GP zones and  $\delta'$  precipitates in the main lift frame material.

### **7.2.6 Discussion of the metastable nature of the increase in strength in the main lift frame material over a 72 hour period.**

As detailed in Section 4.2 and 7.2, the low temperature exposure tensile test groups E, F and G, (exposed to 53.1°C for 72 hours), gave a notable repeatable pattern in the tensile testing results in the longitudinal grain orientation (Figure 81a). Specifically, the pattern for the UTS and 0.2% proof stress results was:

1. An increase in strength from 0 to 24 hours.
2. a significant reduction in strength after 36 hours.
3. a partial recovery from 48 to 72 hours.

The increase in strength is likely to be a direct result of the formation of either GP zones and/or  $\delta'$  precipitates. These species would have potentially formed from the solute that has been shown to be available in the DSC results (Sections 6.2 and 7.2). Consequently, an increase in volume fraction of GP zones and  $\delta'$  precipitates would have the dual effect of impeding dislocation movement, and therefore increasing strength. Also, the relatively small additional  $\delta'$  precipitates would have been susceptible to shearing by dislocations, which would have increased the propensity for planar slip. The combination of these factors would have been sufficient to reduce the ductility of the material. This is evident in the reduction in average elongation of the 80.0°C, Group A, B and C results (Table 18).

In the current work, dissolution events in Al-Li 8090 are considered to be endothermic events in DSC analysis. Given the nature of this secondary  $\delta'$  precipitates (small dispersed precipitates measured to be ~ 2-4 nm in diameter), any endothermic event



caused by dissolution of these precipitates may not provide a significant endothermic heat flow on a DSC curve. In a similar manner, Starink et al. (2000)<sup>124</sup> discounted the exothermic heat flow resulting from coarsening of  $\delta'$  as minimal and not sufficient to have an effect on a DSC curve. However, when considering the 'precursor' temperature dissolution effect (Figure 82), Starink et al. (2000)<sup>124</sup> made reference to *'dissolution of very fine  $\delta'$  precipitates that become unstable on increase of temperature'* as being a potential contributory factor for the endothermic peak.

There is no indication of a significant endothermic event in the region of 53.1°C in the DSC analysis for the 8090/AMB material. An instability or change in the GP zone/ $\delta'$  precipitate characteristics as a result of time (as the temperature is a constant) may be responsible for the change in mechanical properties of the Al-Li 8090 material over the 72 hour period.

As previously mentioned, comparison of the DSC curves for 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials (Figure 85 and Figure 86) indicate evidence of reactions A and B (GP zone formation and  $\delta'$  growth, respectively) as proposed by Starink and Gregson (1996)<sup>127</sup>. As can be seen in Figure 89, 53.1°C is situated at the start of the exothermic event for reaction A. This indicates that GP zones formation is the more likely occurrence in the samples that were held at 53.1°C for 24 and 36 hours, respectively.

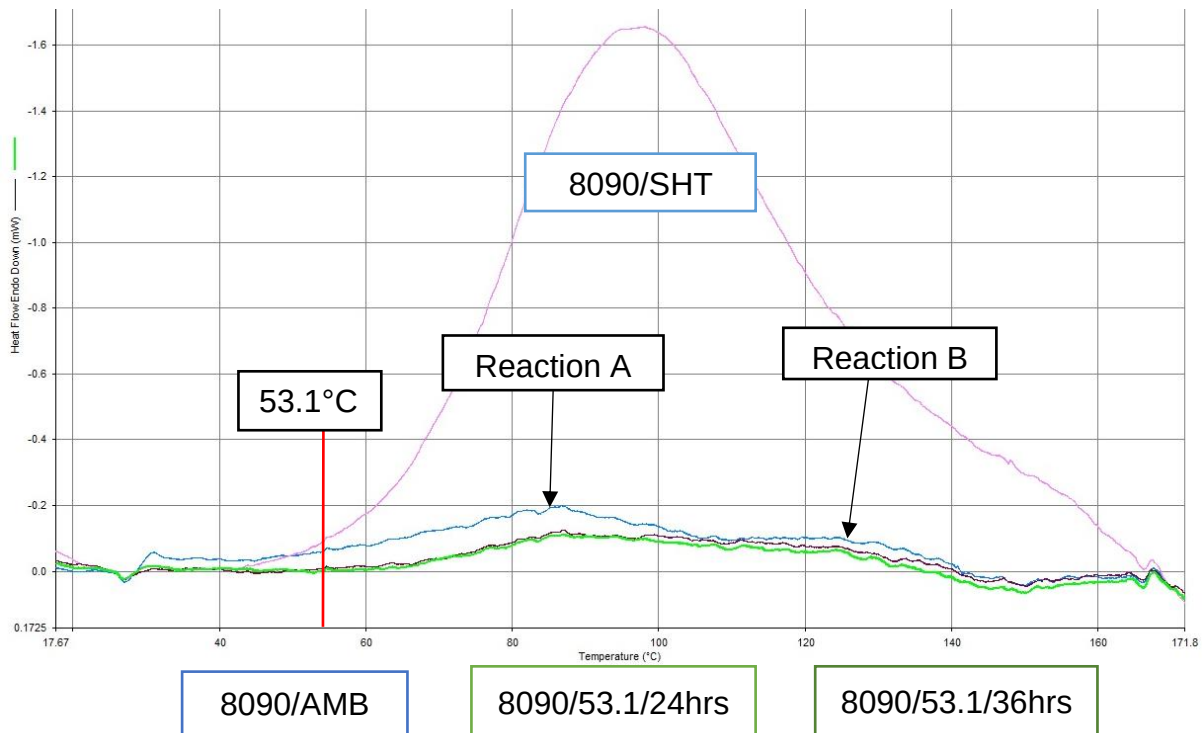


**Figure 89: Identifying reactions A and B in the DSC curves of the 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials.**

Given that the strengthening effect of GP zone comes from the coherency strains, for the reduction in strength in the 8090/53.1°C/36hrs material, a mechanism would be required to explain the loss of coherency strains.

For a precipitate to form, a viable nucleus of a critical radius is required<sup>130</sup>. Initial clustering and formation of the GP zones during the 0 to 24 hour period may not be sufficient to allow for nucleation to occur. This may result in the GP zones becoming unstable, dissolving back into solution after 36 hours at 53.1°C. However, if further exposure to 53.1°C enabled a second clustering and formation of GP zones to occur, as more solute is available this time around, the GP clusters may be able to attain critical radius for nucleation and the finer  $\delta'$  precipitates would be able to form and remain relatively stable in the microstructure.

It should be noted that in their work understanding age hardening of Al-Cu-Mg alloys, Ringer, Hono, Sakurai and Polmear (1996)<sup>131</sup> did not distinguish between GP and GBP zones. The findings of Ringer [et al.] (1996)<sup>131</sup> indicated that whilst GP zones do form in Al-Cu-Mg alloys, their detection in the TEM occurred after hardening had begun to take place in the material. The precursor to the hardening mechanism was attributed to *cluster hardening* which was the result of 'Cu-Mg co-clusters'.

In 2xxx series (Al-Cu) alloys, it is Al-Cu GP zones that form at ambient temperature. Noting that both Al and Cu are present in Al-Li 8090, it could be surmised that the GP zone formation as indicated in the 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials is formed from Al and Cu in the solid solution. Alternatively, these exothermic reactions also indicate the presence of a 'precursor' state prior to the formation of  $\delta'$  precipitates. As precursors are hypothesised to exist prior to the formation of  $\delta'$  precipitates, in Al-Li binary alloys these precursors could potentially be Al-Li GP zones. However as noted by a number of authors, including Starink et al. (2000)<sup>124</sup>, the existence of  $\delta'$  precipitate precursors have not yet been observed in the TEM.

Unfortunately, this work would require a significant amount of TEM investigation of significant number of foils at range of exposures to 53.1°C, and is far beyond the time constraints of this project and could be of interest for further work.

### **7.3 Real world implications of the change in mechanical properties of Al-Li main lift frames subjected to low temperature exposure.**

As mentioned in Section 3.1.1, to ensure safe operation, aircraft components are monitored using Safe Life or Damage Tolerance techniques. Structurally Significant Items are often operated under a Safe Life policy, where extensive modelling, combined with full scale fatigue testing is used to provide a measurable 'fatigue life', typically given in flying hours. Theoretically once this fatigue life has been met, the component is then replaced. In reality a number of factors can have an effect on when a component is replaced. A shortage of spares, or operational requirements (i.e. being embarked on a frigate in the middle of the ocean) can impact a maintenance schedule. In addition, new and emerging Vibration Health and Usage Monitoring Systems (VHUMS) which are fitted to the majority of UK MoD aircraft (in a range of forms) are being used to more accurately assess the in-service loading applied to components. This not only enables early detection of components that may be close to failure (such as increased resonance in a bearing) but also prevents under-used components from being replaced prematurely, maintaining capability and reducing costs.

However, many of the safety cases which allow these aircraft to fly are based on tests to determine the S-N curve (stress vs number. of cycles to failure) of a material in a particular manufactured condition and temper state. These conditions will ultimately be defined by the mechanical properties of the selected material. If the material is found to have altered its mechanical properties over time in service, such as the reduction in ductility in the 53.1°C testing groups in this work, a question should be asked as to what effect this change would have on the fatigue characteristics of the

material. Any improvements to the S-N curve could provide an increase in component life, but naturally any reduction would result in a penalty being applied, which would require component replacement to be made sooner (and therefore increase cost).

It is not uncommon for certain aircraft fleets to recognise ‘mini-fleets’ within their ranks. These mini-fleets often are made up of specific aircraft that have operated in an environment that is known to be harmful or detrimental to the life of the aircraft or the components. For example, aircraft that flew during the eruption of the Eyjafjallajökull volcano (also known as E11<sup>xlii</sup>) in 2010 would have had penalties applied to engines, gearboxes, main rotor heads and main rotor blades due to the conditions they flew in. However, it is accepted that some of these aircraft *had* to fly and therefore the penalties were accepted accordingly. Furthermore, the mechanical properties of the main lift frame of an aircraft directly influence how the aircraft will behave in the event of a crash. Whilst strong materials typically take more load to result in failure, they tend to fail in a brittle manner, which adds a myriad of additional complex issues.

During the research into reversion age heat treatments in the 1990’s by Pitcher, Stewart and Gupta (1991)<sup>58</sup>, McDarmaid and Starr (1995)<sup>59</sup>, McDarmaid (1995)<sup>60,61</sup> and Pitcher (1993)<sup>81</sup>, it was noted that reversion age heat treatments were able to alter the mechanical properties of Al-Li 8090 sheets by way of reducing the peak strength and regaining lost ductility from ‘low temperature embrittlement’. However ultimately, the material would ‘decay’ back to its original mechanical properties prior to the reversion age heat treatment. Pitcher (1993)<sup>81</sup> proposed that this ‘decay’ at 70°C

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<sup>xlii</sup> *For ease of reference in the volcanic ash community.*

would occur within weeks. This same process of a gain in ductility and subsequent 'decay' has been shown to occur in the Group B test results (Figure 40) and is considered by the author to be more consistent with an actual 'reversion' or 'recovery' by way of a metastable mechanism. For all three mechanical properties tested in Group B, the average value of the data points is consistent with a recovery back towards the average value of the main lift frame ambient testing results when compared to the Group A results. The only variable between the testing of Group A and Group B was the additional 1000 hours exposure to ambient temperature, for Group B, following 1000 hours soaking at 53.1°C. Notably the data in the current work, combined with Pitchers' findings indicate that any low temperature effect on the Al-Li 8090 may not be permanent. Further work would be required to determine the rate of recovery and the cause of the effect over a range of temperatures. However, the current findings indicate that if required, ambient temperature exposure mitigation policies could be devised and implemented to recover the mechanical properties of aircraft where it is suspected that exposure to certain in-service/operational temperatures has resulted in alteration of the mechanical properties of Al-Li material.

This work has generated evidence in support of the theory that exposure of a structure manufactured from an Aluminium-Lithium alloy (specifically Al-Li 8090 in the T852 temper condition) to a temperature below the Low Temperature Exposure range of 70°C – 100 °C, is capable of reducing the ductility of Al-Li alloy by 40%.

This data provides a foundation of understanding the condition of in-service Al-Li main lift frames – operating in both ambient and 'hot' (53.1°C) temperature environments

from a microstructural and mechanical property aspect. However, the fundamentals of Structural Integrity and Air Safety are built upon fatigue and toughness characteristics. Therefore, this data supports the following hypothesis:

***Due to the reduction in ductility, current in-service Al-Li main lift frame structures exposed to 53.1°C will exhibit a reduced fatigue life and will subsequently require a re-evaluation in terms of Structural Integrity when compared against newly manufactured material.***

#### **7.4 Limitations of the findings presented in this work.**

From a “real world” perspective, there are limitations to the work presented and these limitations should be acknowledged.

- 1) The soaking of material at a single temperature is not representative of true day/night cycling in a hot operating environment. Whilst the temperature of 53.1°C has been determined from accurate in-region data, in reality, the temperature at night can drop to 18°C during the summer<sup>107</sup>. As has been shown in the Group B results as well as work by (Pitcher (1993)<sup>81</sup>, exposure of Al-Li 8090 main lift frame material to ambient temperature has shown that recovery of mechanical properties can occur.

Therefore, it may be possible that the ambient exposure at night is capable of mitigating any changes in mechanical properties caused by elevated temperature exposure during the day.

- 2) There is notable scatter in the tensile test results for the partially recrystallised main lift frame material. Although the material was the most representative option, and provided a unique insight into the current condition of the main lift frame structure, it is likely that a more microstructurally controlled test sample, such as Al-Li 8090 T81 sheet would provide more reproducible data for further testing.
- 3) TEM examination was undertaken on the ambient temperature (8090/AMB) material and material soaked at 53.1° for 36 hours (8090/53.1°C/36hrs), predominantly to determine if there was any difference between the notable mechanical property results. However, TEM examination of the 24 and 48 hour periods would have provided additional data to help understand the complex microstructural changes which influence the mechanical performance of this material. Unfortunately, time constraints, meant further work in this area was not an option and will need to be the subject of further work.
- 4) Whilst exposure to 53.1°C has been shown to alter the mechanical properties of the Al-Li main lift frame material, the DSC results (Figure 86) indicates that the exothermic reaction A (Starink and Gregson (1996)<sup>126</sup>) initiates around 30°C. This infers that the minimum temperature required to promote formation of GP zones in the main lift frame material and therefore alter the mechanical properties could be a lower temperature than the one investigated in this work.



## **8 Conclusion of the findings from the investigation into the effect of ambient and low temperature exposure of Al-Li main lift frames.**

- 1) Characterisation of the elemental chemical composition, microstructure and mechanical properties for as-received Al-Li 8090 main lift frame material in the T852 temper condition (8090/AMB material) has been shown to be consistent with previous work and existing literature, Riojia and Liu (2012)<sup>10</sup>, Smith (1988)<sup>13</sup>, Prasad, Gokhale and Wanhill (2013)<sup>93,100</sup>, Roth and Bright (1985)<sup>98</sup> and Edgar, Starke and Quist (1985)<sup>99</sup>. This is the first reporting of investigative work that has assessed the current condition of in-service Al-Li 8090 main lift frames.
- 2) The tensile test results for the 8090/AMB material in the longitudinal grain orientation (Figure 34, and Figure 43), gave an average increase of 86 MPa for UTS, an increase of 74 MPa for 0.2% proof stress and a reduction of 1% for elongation when compared against the original material specification (ASM Handbook Vol 2, 1992)<sup>97</sup>. This equates to an increase of 20% for UTS, a 22% increase for 0.2% proof stress and a 17% reduction in elongation (Table 16).
- 3) Using data provided by DSTL (2016)<sup>109</sup>, a representative temperature for in-service exposure of 8090/AMB material operating in a 'hot' environment has been determined to be 53.1°C (Figure 37).
- 4) The experimental results for the tensile test group exposed to 53.1°C for 1000 hours, (Group A, Figure 39), gave an average increase of 40.1 MPa for UTS,

an increase of 64.6 MPa for 0.2% proof stress and a reduction of 2.1% for elongation when compared to the 8090/AMB tensile test results. This equates to an increase of 8.1% for UTS, a 16.4% increase for 0.2% proof stress and a 40.3% reduction in elongation (Table 18). This indicates that consistent exposure of the Al-Li main lift frame material to the representative temperature of 53.1°C is capable of altering the mechanical properties of the Al-Li main lift frames.

- 5) The tensile test results for Group B (1000 hours at 53.1°C and 1000 hours at ambient temperature, Figure 40), gave an average increase of 24.2 MPa for UTS, an increase of 41.7 MPa for 0.2% proof stress and a reduction of 0.8% for elongation when compared to the 8090/AMB tensile test results. This equates to an increase of 4.9% for UTS, a 10.6% increase for 0.2% proof stress and a 15.8% reduction in elongation (Table 18). This data agrees with the work of Pitcher (1993)<sup>81</sup>, in that following 1000 hours at ambient temperature, the material properties of the Group B test specimens began to revert back to those of the 8090/AMB material.
- 6) Six tensile test Groups; E1, (L) E2 (LT), F1 (L), F2 (LT), G1 (L) and G2 (LT), (Figure 47 to Figure 51, respectively) were exposed to 53.1°C for 72 hours and tensile test samples for each group were tested at 12 hour intervals. For the three longitudinal grain orientation groups (E1, F1 and G1), the UTS results gave a repeatable loss of strength after 36 hours exposure to 53.1°C, (Figure 80a).

- 7) TEM examination of the 8090/AMB and 8090/53.1°C/36hrs materials did not reveal any changes to precipitate diameters (i.e. evidence of coarsening) of the  $\delta'$  precipitates in the 8090/53.1°C/36hrs material when compared against the average measured diameter of the  $\delta'$  precipitates in the 8090/AMB material. This is consistent with findings reported in the literature for low temperature exposure of Al-Li alloys below 100°C (Nobel, Harris and Dinsdale, (1996)<sup>28</sup>).
- 8) DSC analysis of the 8090/AMB material (Figure 77), gave indications of exothermic heat flow between 20-175°C, which is the temperature range attributed to the formation and growth of GP zones and  $\delta'$  precipitates (reactions A and B, Figure 74). Further examination of the DSC curves for the 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials (Figure 86 and Figure 87), show that in addition to evidence of exothermic heat flow for reactions A and B for the 8090/AMB material, the same reactions were found to present in the 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials.
- 9) The indications of reactions A and B for the 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials were noted to exhibit less exothermic heat flow than the 8090/AMB material. This indicated that:
- i. There is available solute in the 8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs in order for the detection of exothermic heat flow between 20 and 175°C to be possible.

- ii. The reduction of the exothermic heat flow for the 8090/53.1°C/24hrs and 8090/53.1°C/36hrs materials when compared against the 8090/AMB material indicates that the solute has been utilised in the 8090/53.1°C/24hrs and 8090/53.1°C/36hrs for the formation and growth of GP zones and/or  $\delta'$  precipitates.

Therefore, the change in mechanical properties in the Al-Li main lift frame material exposed to 53.1°C is considered to be the result of both: initial dissolution and subsequent precipitation of GP zones and  $\delta'$  precipitates in the main lift frame material.

## **9 Summary of the work into the effect of ambient temperature aging of Al-Li main lift frames.**

In summary, a representative temperature of internal aircraft main lift frame structure operating in hot environments has been determined. This temperature (53.1°C) has been shown to alter the mechanical properties of Al-Li main lift frame material which are constructed from Al-Li 8090 forgings in the T852 temper state. This material has been taken from aircraft that at a minimum, have experienced a decade of operational service. This material has provided a unique insight in to the current condition of the UK MoD Merlin aircraft fleet.

Characterisation of the in-service main lift frame material by way of elemental chemical analysis, microstructural examination and mechanical testing, has shown the current in-service material to be comparable with the original design specification, albeit with some potential long-term temperature exposure effects.

A mechanical testing regime has shown that 1000 hours exposure to 53.1°C resulted in a modest increase in ultimate tensile strength (7.8%) and 0.2% proof stress (13.2%) and a significant reduction in ductility (36%) when compared to the average mechanical property data of the ambient temperature main lift frame material. These changes in mechanical properties, were found to be consistently repeatable.

Further scrutiny of the mechanical testing regime indicated that the change in mechanical properties for the main lift frame material occurred within the first 72 hours of soaking at 53.1°C. Therefore, a second mechanical testing regime, focusing on the

0 to 72 hour period time was undertaken. The results of this testing regime revealed that a change in mechanical properties occurs, with an increase to peak strength from 0 to 24 hours, a sharp decrease in strength after 36 hours and a subsequent partial recovery after 48 hours, followed by a relatively stable results up to 72 hours. The ductility of the material followed the opposite behaviour, as would be expected.

Complementary analytical techniques of Transmission Electron Microscopy (TEM) and Differential Scanning Calorimetry (DSC) have shown that on comparing the ambient temperature (8090/AMB) and material soaked at 53.1°C for 36 hours (8090/53.1°C/36hrs), the main strengthening precipitate  $\delta'$  ( $\text{Al}_3\text{Li}$ ) does not show evidence of coarsening. In addition, DSC curves for a range of conditions of Al-Li main lift frame material (8090/AMB, 8090/53.1°C/24hrs and 8090/53.1°C/36hrs) have given exothermic heat flow occurring in the region of 20-175°C. This is indicative of; i) available solute in the material, likely to have been present as a result of long term low temperature exposure ii) the formation of Guinier-Preston/Guinier-Preston Bagaryatsky (GP/GPB) zones, which are likely to be responsible for the increase in strength and reduction in ductility in the main lift frame material iii) formation of additional  $\delta'$  precipitates.

These findings indicate that the 8090/AMB material, which is artificially aged to the T852 temper state has, when exposed to 53.1°C, sufficient available solute to enable the growth and formation of GP zones and/or  $\delta'$  precipitates. The formation and dissolution of these GP zones and/or  $\delta'$  precipitates is postulated as being the

explanation for the change in mechanical properties of the Al-Li main lift frame material that has been subjected to tensile testing in this work.

As previously mentioned, the availability of the solute in the 8090/AMB material is not supported by the DSC curve for the 8090 T81 temper material reported by Starink et al. (2000)<sup>124</sup>. The major difference between the two artificially aged materials (whilst acknowledging the T81 was sheet and the main lift frame is a forging), is the length of time between the DSC analysis and the completion of the heat treatment process (5 minutes for the T81 material and a decade of in-service operation for the 8090/AMB material).

These findings indicate that exposure of Al-Li main lift frames to 53.1°C is sufficient to utilise solute available in the solid solution. This is likely to be the result of decomposition of the precipitates over an extended period of time during in-service operation. As no evidence for coarsening of the established  $\delta'$  has been identified, the alternative option for the reported change in mechanical properties is the formation of GP zones and/or a new  $\delta'$  precipitates, albeit far smaller (2 to 4 nm) than the diameter of the  $\delta'$  formed in the 8090/AMB material (measured to be 24 nm in this work). It should be mentioned that a second smaller dispersion of  $\delta'$  precipitates was not identified during this work. However, it has been noted by other authors (Nobel, Harris and Dinsdale (1996)<sup>28</sup> that the small size of the precipitates combined with the sheer volume fraction of the established  $\delta'$  precipitates, makes it very difficult to resolve the small precipitates in the TEM.

In short, these findings indicate that the 8090/AMB material has been subjected to low temperature exposure throughout its in-service operational life, which has resulted in the dissolution of strengthening precipitates into a solid solution. This has subsequently altered the mechanical properties of the main lift frame when exposed to a representative temperature of a 'hot' operating environment.

### 9.1 Suggestions for further work.

Taking into account the findings of the current work and the limitations detailed in the previous section, the following is considered for future work:

- 1) TEM examination of the Al-Li main lift frame material that has been soaked at 53.1°C for 12, 24, 48, 60 and 72 hours, may provide a much clearer picture as to the mechanism affecting the mechanical properties within the first 72 hours period of exposure to 53.1°C.
- 2) Comparison of the change in mechanical properties of the Al-Li main lift frame material in the current work against a more microstructurally controlled material (such as 8090 T81 sheet). It is anticipated that this material would reduce the scatter range in the results and help provide a clearer understanding on the pattern of change in the mechanical properties in the first 72 hour period. However, caution would need to be exercised as freshly heat treated material is likely to not contain the decomposition of the solid solution as proposed by Zakharov (2003)<sup>62</sup> combined with the potential effect of long-term age hardening.



- 3) Determine the day/night temperature cycle in a hot operating environment and assess whether the 'complete thermal cycle' has a detrimental effect on the mechanical properties of the Al-Li main lift frame material.
- 4) Analysis of Al-Li main lift frame material at temperatures lower than 53.1°C. It is highly likely that 53.1°C is not the minimum required temperature to affect the mechanical properties of the Al-Li main lift frame material. Therefore, a study looking into the effect of 50°C down to at least 25°C is recommended. This could have further impact on a number of factors where temperature and exposure of main lift frames may require consideration.

In terms of the availability of material for future projects, a significant number of the main lift frames provided for this project have been utilised. There are only a limited selection of tensile test pieces remaining. There is however, a significant amount of sectioned material that could be used for SEM, TEM, DSC and other analytical techniques that require small samples.

## Annex A: Tensile test results.

**Table A-1: Showing the tensile test results for the ambient temperature tensile test pieces (Frame H2).**

| Test Piece Number | Test Piece Orientation | UTS (MPa) | 0.2% proof stress (MPa) | Elongation (%) |                |
|-------------------|------------------------|-----------|-------------------------|----------------|----------------|
| H2-1              | Longitudinal           | 498.38    | 401.96                  | 5.80           | Test Section 1 |
| H2-2              | Longitudinal           | 495.00    | 389.19                  | 6.70           |                |
| H2-3              | Longitudinal           | 488.51    | 416.50                  | 3.30           |                |
| H2-4              | Longitudinal           | N/A       | N/A                     | N/A            |                |
| H2-5              | Longitudinal           | 508.84    | 404.42                  | 5.10           |                |
| H2-6              | Longitudinal           | 511.88    | 431.96                  | 5.60           |                |
| H2-7              | Longitudinal           | 504.59    | 403.36                  | 5.80           |                |
| H2-8              | Longitudinal           | 498.51    | 426.40                  | 7.00           |                |
| H2-9              | Longitudinal           | 488.41    | 378.30                  | 5.30           |                |
| H2-10             | Long-Transverse        | 510.87    | 395.86                  | 5.10           |                |
| H2-11             | Long-Transverse        | 499.92    | 368.43                  | 5.90           | Test Section 2 |
| H2-12             | Long-Transverse        | 505.02    | 392.29                  | 4.30           |                |
| H2-13             | Long-Transverse        | 493.86    | 391.60                  | 3.90           |                |
| H2-14             | Long-Transverse        | 495.30    | 389.47                  | 3.00           |                |
| H2-15             | Long-Transverse        | 473.81    | 404.68                  | 2.50           |                |
| H2-16             | Long-Transverse        | 500.93    | 422.89                  | 2.70           |                |
| H2-17             | Long-Transverse        | 502.81    | 406.08                  | 3.00           |                |
| H2-18             | Long-Transverse        | 506.45    | 403.39                  | 4.20           |                |
| H2-19             | Long-Transverse        | 500.57    | 380.40                  | 6.90           |                |
| H2-20             | Long-Transverse        | 508.76    | 387.94                  | 4.80           |                |
| H2-21             | Long-Transverse        | 515.58    | 423.42                  | 3.91           | Test Section 4 |
| H2-22             | Longitudinal           | 492.05    | 408.99                  | 2.83           |                |
| H2-23             | Longitudinal           | N/A       | N/A                     | N/A            |                |
| H2-24             | Longitudinal           | 449.15    | 381.93                  | 2.67           |                |
| H2-25             | Longitudinal           | 479.21    | 368.11                  | 4.63           |                |
| H2-26             | Long-Transverse        | 525.97    | 450.03                  | 3.16           |                |
| H2-27             | Longitudinal           | 491.58    | 406.11                  | 3.70           |                |
| H2-28             | Longitudinal           | 491.58    | 383.08                  | 6.04           |                |
| H2-29             | Long-Transverse        | 502.77    | 397.90                  | 4.51           | Section 3      |
| H2-30             | Long-Transverse        | 525.66    | 406.76                  | 5.35           |                |
| H2-31             | Longitudinal           | 505.36    | 394.14                  | 4.70           |                |
| H2-32             | Longitudinal           | 479.25    | 370.62                  | 5.12           |                |
| H2-33             | Longitudinal           | 485.07    | 371.58                  | 4.73           |                |
| H2-34             | Longitudinal           | 486.46    | 353.93                  | 6.76           |                |
| H2-35             | Longitudinal           | 498.79    | 386.69                  | 6.18           |                |

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|-------|-----------------|--------|--------|------|--|
| H2-36 | Longitudinal    | 531.02 | 411.39 | 4.89 |  |
| H2-37 | Long-Transverse | 482.03 | 351.94 | 7.28 |  |
| H2-38 | Long-Transverse | 543.63 | 445.95 | 4.72 |  |
| H2-39 | Long-Transverse | 511.31 | 405.59 | 3.11 |  |

**Table A-2: Showing the tensile test results for the 80°C 1000 hour low temperature exposure reduced size tensile test pieces (Frame B2).**

| Test Piece Number | Test Piece Orientation | UTS (MPa)                      | 0.2% Proof Stress (MPa) | Elongation (%) |
|-------------------|------------------------|--------------------------------|-------------------------|----------------|
| B2-1              | Longitudinal           | 505.00                         | 429.00                  | 2.94           |
| B2-2              | Longitudinal           | 495.00                         | 462.00                  | 0.87           |
| B2-3              | Longitudinal           | 400.00                         | 416.00                  | 3.06           |
| B2-4              | Longitudinal           | 520.00                         | 423.00                  | 4.09           |
| B2-9              | Long-Transverse        | 520.00                         | 459.00                  | 1.00           |
| B2-11             | Long-Transverse        | 517.00                         | 446.00                  | 2.00           |
| B2-12             | Long-Transverse        | 507.00                         | 414.00                  | 3.00           |
| B2-18             | Long-Transverse        | 503.00                         | 364.00                  | 3.50           |
| B2-20             | Longitudinal           | 503.00                         | 405.00                  | 3.01           |
| B2-21             | Longitudinal           | Used for 'cold ductility test' |                         |                |
| B2-23             | Longitudinal           | 508.00                         | 417.00                  | 4.08           |
| B2-24             | Longitudinal           | 506.00                         | 402.00                  | 4.91           |
| B2-25             | Long-Transverse        | 510.00                         | 425.00                  | 3.00           |
| B2-26             | Longitudinal           | Used for 'cold ductility test' |                         |                |
| B2-27             | Long-Transverse        | 524.00                         | 427.00                  | 4.50           |

**Table A-3: Showing the tensile test results for the 53.1°C 1000 hour low temperature exposure tensile test pieces (Frame A2).**

| Test Piece Number | Test Piece Orientation | UTS (MPa)                      | 0.2% proof stress (MPa) | Elongation (%) | Group |
|-------------------|------------------------|--------------------------------|-------------------------|----------------|-------|
| A2-1              | Long-transverse        | 505.00                         | 381.00                  | 5.26           | A     |
| A2-2              | Longitudinal           | Not used due to geometry issue |                         |                | N/A   |
| A2-3              | Longitudinal           | 535.00                         | 455.00                  | 3.83           | A     |
| A2-4              | Long-transverse        | 524.00                         | 435.00                  | 3.66           | A     |
| A2-5              | Longitudinal           | 525.00                         | 423.00                  | 4.86           | A     |
| A2-6              | Longitudinal           | 574.00                         | 468.00                  | 1.92           | A     |
| A2-7              | Longitudinal           | 538.00                         | 490.00                  | 2.35           | A     |

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|       |                 |        |        |      |   |
|-------|-----------------|--------|--------|------|---|
| A2-8  | Longitudinal    | 529.00 | 444.00 | 3.57 | A |
| A2-9  | Longitudinal    | 525.00 | 443.00 | 3.12 | A |
| A2-10 | Longitudinal    | 496.00 | 420.00 | 2.52 | B |
| A2-11 | Long-transverse | 488.00 | 373.00 | 4.12 | A |
| A2-12 | Long-transverse | 477.00 | 384.00 | 2.47 | A |
| A2-13 | Long-transverse | 492.00 | 391.00 | 3.72 | A |
| A2-14 | Long-transverse | 495.00 | 394.00 | 3.06 | A |
| A2-15 | Long-transverse | 500.00 | 398.00 | 3.05 | A |
| A2-16 | Long-transverse | 490.00 | 389.00 | 2.80 | B |
| A2-17 | Long-transverse | 490.00 | 402.00 | 2.37 | B |
| A2-18 | Long-transverse | 512.00 | 430.00 | 2.79 | B |
| A2-19 | Long-transverse | 500.00 | 410.00 | 3.16 | B |
| A2-20 | Long-transverse | 498.00 | 433.00 | 2.63 | B |
| A2-21 | Longitudinal    | 520.00 | 431.00 | 4.30 | B |
| A2-22 | Longitudinal    | 513.00 | 408.00 | 5.08 | B |
| A2-23 | Longitudinal    | 500.00 | 402.00 | 3.91 | B |
| A2-24 | Longitudinal    | 529.00 | 445.00 | 3.32 | B |
| A2-25 | Longitudinal    | 539.00 | 477.00 | 4.78 | B |
| A2-26 | Longitudinal    | 547.00 | 465.00 | 6.28 | B |
| A2-27 | Longitudinal    | 529.00 | 485.00 | 1.79 | A |

**Table A-3: Showing the tensile test results for Groups C and D (Frame C2).**

| Test Piece Number | Test Piece Orientation | UTS (MPa) | 0.2% Proof Stress (MPa) | Elongation (%) | Group |
|-------------------|------------------------|-----------|-------------------------|----------------|-------|
| C2-2              | Longitudinal           | 523       | 420                     | 4.957          | C     |
| C2-4              | Longitudinal           | 514       | 445                     | 2.9            | C     |
| C2-5              | Longitudinal           | 511       | 427                     | 3.266          | C     |
| C2-6              | Longitudinal           | 518       | 445                     | 3.054          | C     |
| C2-7              | Longitudinal           | 508       | 438                     | 2.554          | C     |
| C2-8              | Longitudinal           | 513       | 432                     | 4.337          | C     |
| C2-9              | Longitudinal           | 487       | 420                     | 2.8            |       |
| C2-23             | Longitudinal           | 505       | 409                     | 4.018          | D     |
| C2-24             | Longitudinal           | 508       | 402                     | 4.675          | D     |
| C2-25             | Longitudinal           | 515       | 438                     | 3.975          | D     |
| C2-26             | Longitudinal           | 495       | 446                     | 2.751          | D     |
| C2-27             | Longitudinal           | 508       | 410                     | 4.978          | D     |
| C2-28             | Longitudinal           | 507       | 454                     | 2.781          | D     |

**Table A-4: Showing the tensile test results for Groups E, E2, G and G2 (Frame F2).**

| Test Piece Number | Test Piece Orientation | UTS (MPa) | 0.2% Proof Stress (MPa) | Elongation (%) | Group |
|-------------------|------------------------|-----------|-------------------------|----------------|-------|
| F2-1              | Longitudinal           | 516       | 467                     | 3.221          | E     |
| F2-2              | Longitudinal           | 528       | 446                     | 5.672          | E     |
| F2-3              | Longitudinal           | 516       | 444                     | 5.672          | E     |
| F2-4              | Longitudinal           | 484       | 433                     | 3.909          | E     |
| F2-5              | Longitudinal           | 498       | 397                     | 7.888          | E     |
| F2-6              | Longitudinal           | 514       | 411                     | 5.862          | E     |
| F2-7              | Long-transverse        | 443       | 411                     | 0.7129         | E2    |
| F2-8              | Long-transverse        | 516       | 445                     | 2.395          | E2    |
| F2-9              | Long-transverse        | 485       | 443                     | 1.907          | E2    |
| F2-10             | Long-transverse        | 397       | 0                       | 0.072          | E2    |
| F2-11             | Long-transverse        | 474       | 398                     | 3.064          | E2    |
| F2-12             | Long-transverse        | 505       | 420                     | 4.826          | E2    |
| F2-13             | Long-transverse        | 320       | 434                     | 4.266          | E2    |
| F2-14             | Long-transverse        | 503       | 385                     | 5.16           | G2    |
| F2-15             | Long-transverse        | 504       | 390                     | 5.614          | G2    |
| F2-16             | Long-transverse        | 512       | 394                     | 7.305          | G2    |
| F2-17             | Longitudinal           | 493       | 400                     | 3.552          | E     |
| F2-18             | Longitudinal           | 509       | 441                     | 3.582          | G     |
| F2-19             | Longitudinal           | 510       | 420                     | 5.613          | G     |
| F2-20             | Longitudinal           | 531       | 466                     | 4.306          | G     |
| F2-21             | Longitudinal           | 484       | 365                     | 5.633          | G     |
| F2-22             | Longitudinal           | 505       | 422                     | 3.417          | G     |
| F2-23             | Long-transverse        | 490       | 434                     | 1.794          | G2    |
| F2-24             | Long-transverse        | 523       | 413                     | 6.294          | G2    |
| F2-25             | Longitudinal           | 500       | 399                     | 5.786          | G     |
| F2-26             | Longitudinal           | 514       | 410                     | 6.546          | G     |
| F2-27             | Long-transverse        | 506       | 386                     | 6.459          | G2    |

**Table A-5: Showing the tensile test results for Groups F, F2 and G2 (Frame G1).**

| Test Piece Number | Test Piece Orientation | UTS (MPa)               | 0.2% Proof Stress (MPa) | Elongation (%) | Group |
|-------------------|------------------------|-------------------------|-------------------------|----------------|-------|
| G1-27             | Longitudinal           | 495                     | 434                     | 4.126          | F     |
| G1-28             | Longitudinal           | 501                     | 406                     | 6.326          | F     |
| G1-29             | Longitudinal           | 511                     | 425                     | 6.052          | F     |
| G1-30             | Longitudinal           | 485                     | 364                     | 7.41           | F     |
| G1-31             | Longitudinal           | Unacceptable test piece |                         |                |       |
| G1-32             | Longitudinal           | 500                     | 386                     | 6.169          | F     |
| G1-33             | Longitudinal           | 495                     | 404                     | 3.996          | F     |
| G1-34             | Long-transverse        | 484                     | 374                     | 3.761          | F2    |
| G1-35             | Long-transverse        | 502                     | 407                     | 3.338          | F2    |
| G1-36             | Long-transverse        | Unacceptable test piece |                         |                |       |
| G1-37             | Long-transverse        | 490                     | 399                     | 3.717          | F2    |
| G1-38             | Long-transverse        | 501                     | 393                     | 5.354          | F2    |
| G1-39             | Long-transverse        | 497                     | 392                     | 5.981          | F2    |
| G1-40             | Long-transverse        | 494                     | 388                     | 5.87           | F2    |
| G1-41             | Long-transverse        | 499                     | 389                     | 6.235          | F2    |
| G1-42             | Long-transverse        | 493                     | 361                     | 6.783          | G2    |
| G1-44             | Longitudinal           | 502                     | 411                     | 5.979          | F     |

**Table A-6: Showing the tensile test results for Group H (Frame E1).**

| Test Piece Number | Test Piece Orientation | UTS (MPa) | 0.2% Proof Stress (MPa) | Elongation (%) | Group |
|-------------------|------------------------|-----------|-------------------------|----------------|-------|
| E1-5              | Longitudinal           | 533       | 412                     | 6              | H     |
| E1-6              | Longitudinal           | 484       | 371                     | 6              | H     |
| E1-7              | Longitudinal           | 502       | 414                     | 5.6            | H     |
| E1-18             | Longitudinal           | 568       | 479                     | 4.5            | H     |
| E1-19             | Longitudinal           | 513       | 421                     | 5.1            | H     |
| E1-20             | Longitudinal           | 511       | 418                     | 4.4            | H     |
| E1-21             | Longitudinal           | 499       | 403                     | 5.2            | H     |
| E1-22             | Longitudinal           | 485       | 408                     | 2.9            | H     |
| E1-25             | Longitudinal           | 517       | 424                     | 5.7            | H     |

## **Annex B: Inductively coupled plasma atomic emission spectroscopy methodology and results.**

### 1710 NAS Internal ICP Al Li Method

Prepared samples need to be approximately 1mg of metal in 10ml of 10% Aqua Regia in order to match the concentrations of the standard.

### Safety

All preparation work requires fume cupboard, gloves, goggles and lab-coat.

Analysis requires gloves, goggles and lab-coat.

### Equipment

20ml Aqua Regia, make as:

1 Part Nitric acid at ca 63%. (5ml)

3 Parts Hydrochloric acid at ca 37%. (15ml)

200ml distilled water.

Pipettes to suit.

5mg each sample alloy as filings (or similar sample).

30ml Custom multi-element standard.

1 x 50ml beaker.

4 x 50ml volumetric flasks.

16 x 10ml disposable test tubes.

Method

Add 5mg of alloy to three of the volumetric flasks. The fourth is used for the base solution.

Aqua Regia to be prepared immediately before use. Nitric acid must be carefully added to the hydrochloric acid in the 50ml beaker.

Add 5ml to of Aqua Regia to each flask.

Leave to react and cool.

Make up to volume with distilled water.

Allow to cool.

Decant each sample solution to 4 x 10ml disposable test tubes for analysis. Two tubes are filled with Aqua Regia base and custom standard for instrument calibration and two further tubes are filled with custom standard as check samples. Analyse on Perkin Elmer Optima 3000 RL ICP using a standard aluminium method and aqueous sample introduction system. Each element was analysed using two wavelengths, which are detailed in Table B-1.

The wavelengths used were selected for their high sensitivity and lack of interference from other elements, typical limits of detection of these wavelengths are better than 10 parts per billion. Since the concentrations being analysed are low, the instrument calibration is proportional using the Aqua Regia base and a purchased custom



standard. The custom standard is NIST<sup>xliii</sup> traceable. Four tubes for each sample are used and each tube is analysed using five replicates, this means that the final value that is given for each wavelength is the average of twenty determinations.

**Table B-1: Showing the analytical wavelengths used for ICP-OES analysis.**

| Element | Wavelength 1<br>(nm) | Wavelength 2<br>(nm) |
|---------|----------------------|----------------------|
| Al      | 308.215              | 396.152              |
| Cu      | 324.754              | 224.700              |
| Fe      | 238.204              | 239.562              |
| Li      | 670.781              | 610.364              |
| Mg      | 279.079              | 280.270              |
| Mn      | 257.610              | 260.569              |
| Si      | 251.611              | 212.412              |
| Ti      | 334.941              | 336.121              |
| Zr      | 343.823              | 339.198              |

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<sup>xliii</sup> *National Institute of Standards and Technology.*

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**Table B-2: Showing the ICP results for the main lift frame material**

| Run                  | Sample | Elemental Composition (ppm) |         |         |         |         |         |         |         |         |         |         |         |
|----------------------|--------|-----------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
|                      |        | Al                          |         | Cu      |         | Fe      |         | Li      |         | Mg      |         | Zr      |         |
|                      |        | 396.153                     | 308.215 | 327.393 | 324.752 | 238.204 | 239.562 | 670.784 | 610.362 | 285.213 | 279.077 | 343.823 | 339.197 |
| 1                    | 1-1    | 62.610                      | 63.570  | 0.862   | 0.876   | 0.038   | 0.035   | 1.501   | 1.281   | 0.497   | 0.440   | 0.080   | 0.081   |
|                      | 1-2    | 83.680                      | 80.300  | 1.149   | 1.154   | 0.033   | 0.035   | 2.023   | 1.643   | 0.648   | 0.575   | 0.100   | 0.101   |
|                      | 1-3    | 81.030                      | 76.800  | 1.118   | 1.126   | 0.031   | 0.025   | 1.964   | 0.657   | 0.635   | 0.660   | 0.102   | 0.095   |
|                      | 1-4    | 84.890                      | 81.710  | 1.141   | 1.155   | 0.035   | 0.026   | 2.099   | 1.689   | 0.645   | 0.616   | 0.101   | 0.100   |
| 2                    | 2-1    | 64.150                      | 63.540  | 0.885   | 0.872   | 0.055   | 0.040   | 1.577   | 0.787   | 0.532   | 0.484   | 0.059   | 0.072   |
|                      | 2-2    | 82.480                      | 81.090  | 1.100   | 1.110   | 0.046   | 0.039   | 1.987   | 1.583   | 0.621   | 0.663   | 0.093   | 0.096   |
|                      | 2-3    | 82.970                      | 83.050  | 1.152   | 1.128   | 0.047   | 0.034   | 2.007   | 1.758   | 0.650   | 0.616   | 0.092   | 0.084   |
|                      | 2-4    | 81.120                      | 80.960  | 1.103   | 1.096   | 0.047   | 0.045   | 1.199   | 1.648   | 0.631   | 0.613   | 0.090   | 0.094   |
| 3<br>(Recalc)        | 3-1    | 64.150                      | 63.540  | 0.885   | 0.872   | 0.052   | 0.035   | 1.577   | 0.787   | 0.530   | 0.520   | 0.060   | 0.072   |
|                      | 3-2    | 82.480                      | 81.090  | 1.100   | 1.110   | 0.046   | 0.031   | 1.987   | 1.583   | 0.619   | 0.661   | 0.095   | 0.096   |
|                      | 3-3    | 82.970                      | 83.050  | 1.152   | 1.128   | 0.045   | 0.030   | 2.007   | 1.758   | 0.645   | 0.670   | 0.100   | 0.084   |
|                      | 3-4    | 81.120                      | 80.960  | 1.103   | 1.096   | 0.048   | 0.035   | 1.991   | 1.648   | 0.628   | 0.623   | 0.095   | 0.094   |
| Mean                 |        | 77.804                      | 76.638  | 1.063   | 1.060   | 0.044   | 0.034   | 1.743   | 1.402   | 0.607   | 0.595   | 0.089   | 0.089   |
| Mean (Lines)         |        | 77.221                      |         | 1.061   |         | 0.039   |         | 1.573   |         | 0.601   |         | 0.089   |         |
| St Deviation         |        | 8.623                       | 8.047   | 0.114   | 0.114   | 0.008   | 0.006   | 0.528   | 0.416   | 0.054   | 0.076   | 0.015   | 0.010   |
| St Deviation (Lines) |        | 0.408                       |         | 0.000   |         | 0.001   |         | 0.079   |         | 0.015   |         | 0.003   |         |

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Table B-2: continued.

|                         |     |         |         |       |       |       |       |       |       |       |       |       |       |
|-------------------------|-----|---------|---------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| 4                       | 4-1 | 110.700 | 105.800 | 1.593 | 1.602 | 0.064 | 0.052 | 2.831 | 2.276 | 0.887 | 0.821 | 0.134 | 0.134 |
|                         | 4-2 | 126.900 | 121.400 | 1.812 | 1.817 | 0.064 | 0.053 | 3.206 | 2.378 | 0.998 | 0.975 | 0.162 | 0.152 |
|                         | 4-3 | 126.900 | 121.100 | 1.793 | 1.798 | 0.052 | 0.045 | 3.208 | 3.343 | 0.989 | 0.944 | 0.152 | 0.147 |
|                         | 4-4 | 122.400 | 116.800 | 1.786 | 1.780 | 0.039 | 0.045 | 3.116 | 2.984 | 0.989 | 1.011 | 0.149 | 0.149 |
| 5                       | 5-1 | 109.000 | 107.000 | 1.536 | 1.527 | 0.068 | 0.067 | 2.807 | 2.958 | 0.849 | 0.867 | 0.114 | 0.124 |
|                         | 5-2 | 123.100 | 122.800 | 1.739 | 1.732 | 0.067 | 0.075 | 3.091 | 2.679 | 1.018 | 0.932 | 0.150 | 0.129 |
|                         | 5-3 | 115.400 | 116.300 | 1.660 | 1.623 | 0.046 | 0.055 | 3.029 | 2.845 | 0.903 | 0.829 | 0.141 | 0.125 |
|                         | 5-4 | 119.200 | 116.600 | 1.626 | 1.662 | 0.061 | 0.052 | 2.983 | 2.595 | 0.910 | 0.923 | 0.133 | 0.132 |
| 6<br>(Recalc)           | 6-1 | 109.000 | 107.000 | 1.536 | 1.527 | 0.062 | 0.054 | 2.807 | 2.958 | 0.843 | 0.887 | 0.111 | 0.124 |
|                         | 6-2 | 123.100 | 122.800 | 1.739 | 1.732 | 0.069 | 0.063 | 3.091 | 2.679 | 1.011 | 0.958 | 0.148 | 0.129 |
|                         | 6-3 | 115.400 | 116.300 | 1.660 | 1.623 | 0.051 | 0.044 | 3.029 | 2.845 | 0.895 | 0.868 | 0.147 | 0.125 |
|                         | 6-4 | 119.200 | 116.600 | 1.626 | 1.662 | 0.059 | 0.046 | 2.983 | 2.595 | 0.902 | 0.942 | 0.135 | 0.132 |
| Mean                    |     | 118.358 | 115.875 | 1.676 | 1.674 | 0.059 | 0.054 | 3.015 | 2.761 | 0.933 | 0.913 | 0.140 | 0.134 |
| Mean (Lines)            |     | 117.117 |         | 1.675 |       | 0.056 |       | 2.888 |       | 0.923 |       | 0.137 |       |
| St Deviation            |     | 6.466   | 6.143   | 0.097 | 0.099 | 0.009 | 0.010 | 0.141 | 0.291 | 0.064 | 0.059 | 0.015 | 0.010 |
| St Deviation<br>(Lines) |     | 0.229   |         | 0.001 |       | 0.000 |       | 0.106 |       | 0.004 |       | 0.004 |       |

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Table B-2: continued.

|                      |     |         |         |       |       |       |       |       |       |       |       |       |       |
|----------------------|-----|---------|---------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| 7                    | 7-1 | 83.510  | 79.220  | 1.186 | 1.193 | 0.040 | 0.031 | 2.061 | 0.863 | 0.665 | 0.658 | 0.103 | 0.095 |
|                      | 7-2 | 113.800 | 109.000 | 1.605 | 1.615 | 0.048 | 0.041 | 2.867 | 2.767 | 0.890 | 0.841 | 0.135 | 0.130 |
|                      | 7-3 | 117.700 | 112.800 | 1.627 | 1.634 | 0.046 | 0.051 | 3.025 | 2.209 | 0.905 | 0.892 | 0.139 | 0.132 |
|                      | 7-4 | 108.500 | 103.800 | 1.516 | 1.526 | 0.039 | 0.032 | 2.726 | 2.332 | 0.842 | 0.766 | 0.136 | 0.131 |
| 8                    | 8-1 | 80.390  | 77.940  | 1.090 | 1.096 | 0.041 | 0.040 | 2.052 | 1.800 | 0.631 | 0.559 | 0.089 | 0.088 |
|                      | 8-2 | 115.100 | 114.000 | 1.578 | 1.598 | 0.060 | 0.059 | 2.905 | 1.702 | 0.885 | 0.916 | 0.125 | 0.129 |
|                      | 8-3 | 116.700 | 115.000 | 1.592 | 1.619 | 0.063 | 0.046 | 2.911 | 2.164 | 0.902 | 0.872 | 0.131 | 0.124 |
|                      | 8-4 | 110.100 | 108.100 | 1.502 | 1.497 | 0.046 | 0.041 | 2.736 | 2.680 | 0.849 | 0.808 | 0.121 | 0.128 |
| 9<br>(Recalc)        | 9-1 | 80.390  | 77.940  | 1.090 | 1.096 | 0.044 | 0.037 | 2.052 | 1.800 | 0.625 | 0.605 | 0.094 | 0.088 |
|                      | 9-2 | 115.100 | 114.000 | 1.578 | 1.598 | 0.063 | 0.053 | 2.905 | 1.702 | 0.875 | 0.901 | 0.128 | 0.129 |
|                      | 9-3 | 116.700 | 115.000 | 1.592 | 1.619 | 0.063 | 0.042 | 2.911 | 2.164 | 0.895 | 0.857 | 0.136 | 0.124 |
|                      | 9-4 | 110.100 | 108.100 | 1.502 | 1.497 | 0.049 | 0.030 | 2.736 | 2.680 | 0.841 | 0.812 | 0.128 | 0.128 |
| Mean                 |     | 105.674 | 102.908 | 1.455 | 1.466 | 0.050 | 0.042 | 2.657 | 2.072 | 0.817 | 0.791 | 0.122 | 0.119 |
| Mean (Lines)         |     | 104.291 |         | 1.460 |       | 0.046 |       | 2.365 |       | 0.804 |       | 0.120 |       |
| St Deviation         |     | 14.919  | 15.184  | 0.206 | 0.210 | 0.009 | 0.009 | 0.374 | 0.540 | 0.109 | 0.120 | 0.017 | 0.017 |
| St Deviation (Lines) |     | 0.187   |         | 0.003 |       | 0.000 |       | 0.118 |       | 0.008 |       | 0.000 |       |

| Chemical Proportion |      |      |      |      |      |      |
|---------------------|------|------|------|------|------|------|
| Sample              | Cu   | Fe   | Li   | Mg   | Zr   | Al   |
| 1                   | 0.86 | 0.03 | 1.27 | 0.48 | 0.07 | Bal. |
| 2                   | 1.35 | 0.07 | 3.55 | 1.13 | 0.17 | Bal. |
| 3                   | 1.59 | 0.05 | 2.58 | 0.88 | 0.13 | Bal. |
| Mean                | 1.27 | 0.05 | 2.46 | 0.83 | 0.12 |      |

### Annex C: Tensile test piece frame locations.

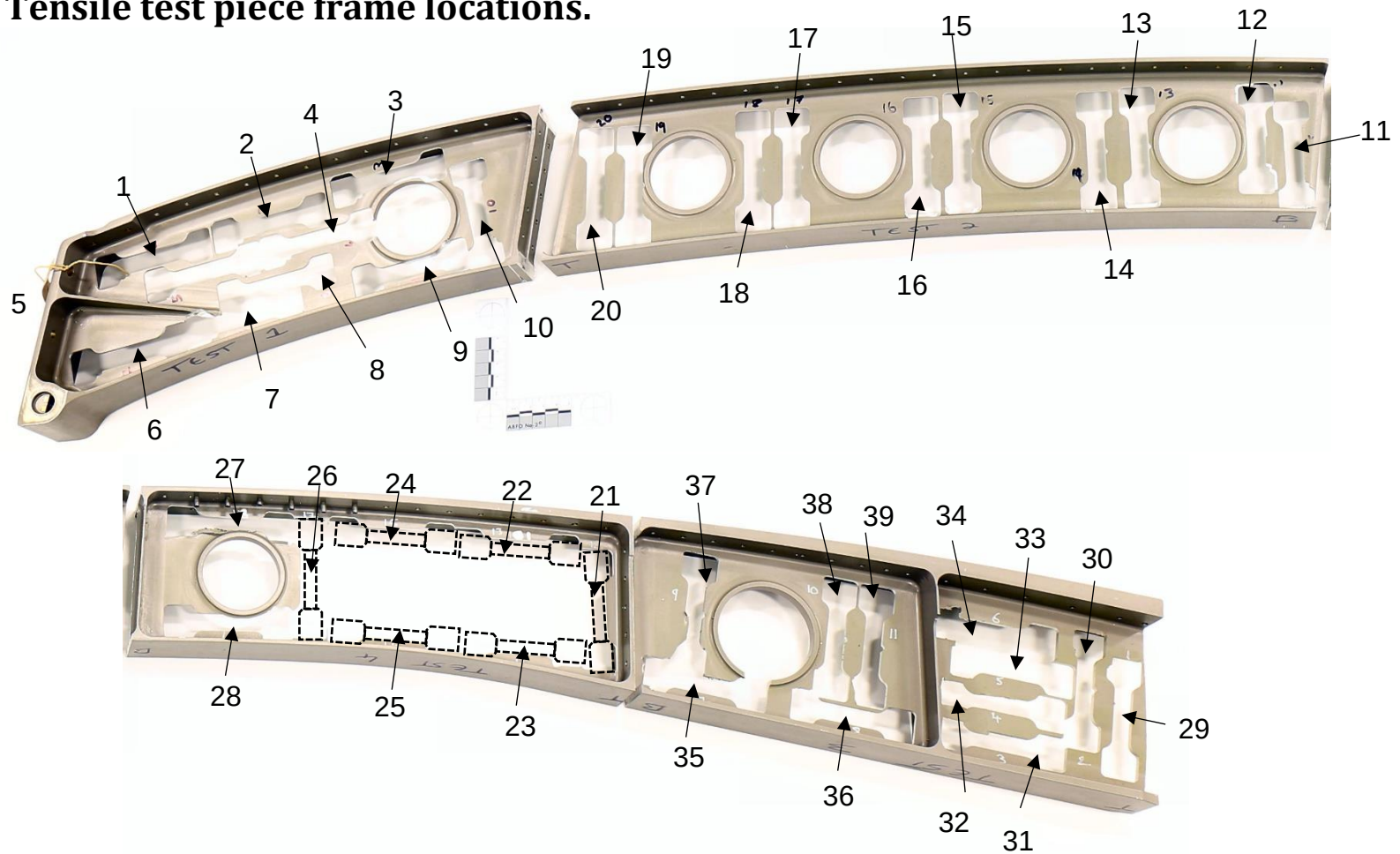


**Figure C1. Showing the locations of the ambient tensile test specimens taken from Frame H2.**

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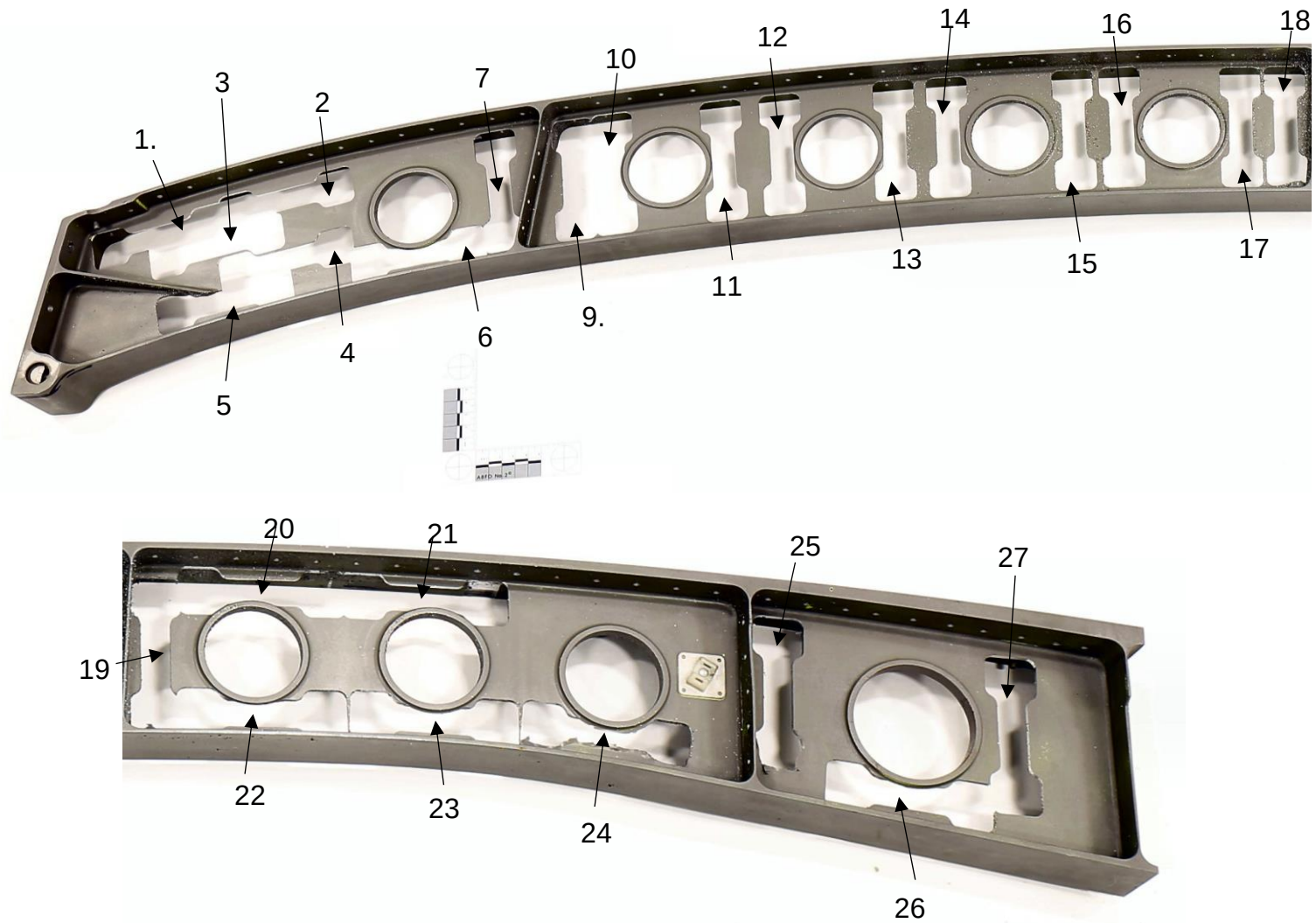


**Figure C2. Showing the locations of the 80.0°C tensile test specimens taken from Frame B2 (note the absence of number 8).**

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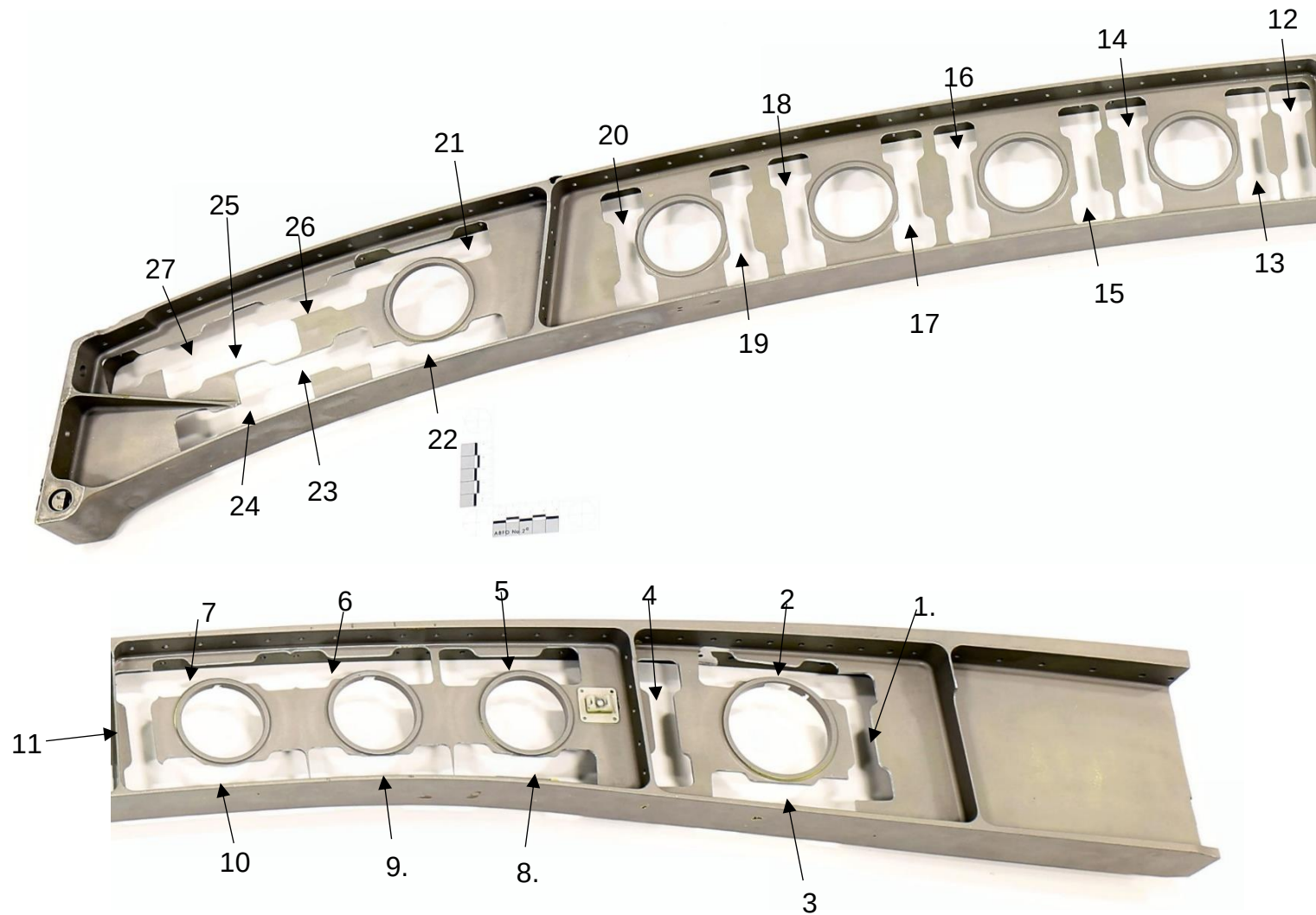


**Figure C3. Showing the location of the 53.1°C tensile test specimens taken from Frame A2.**

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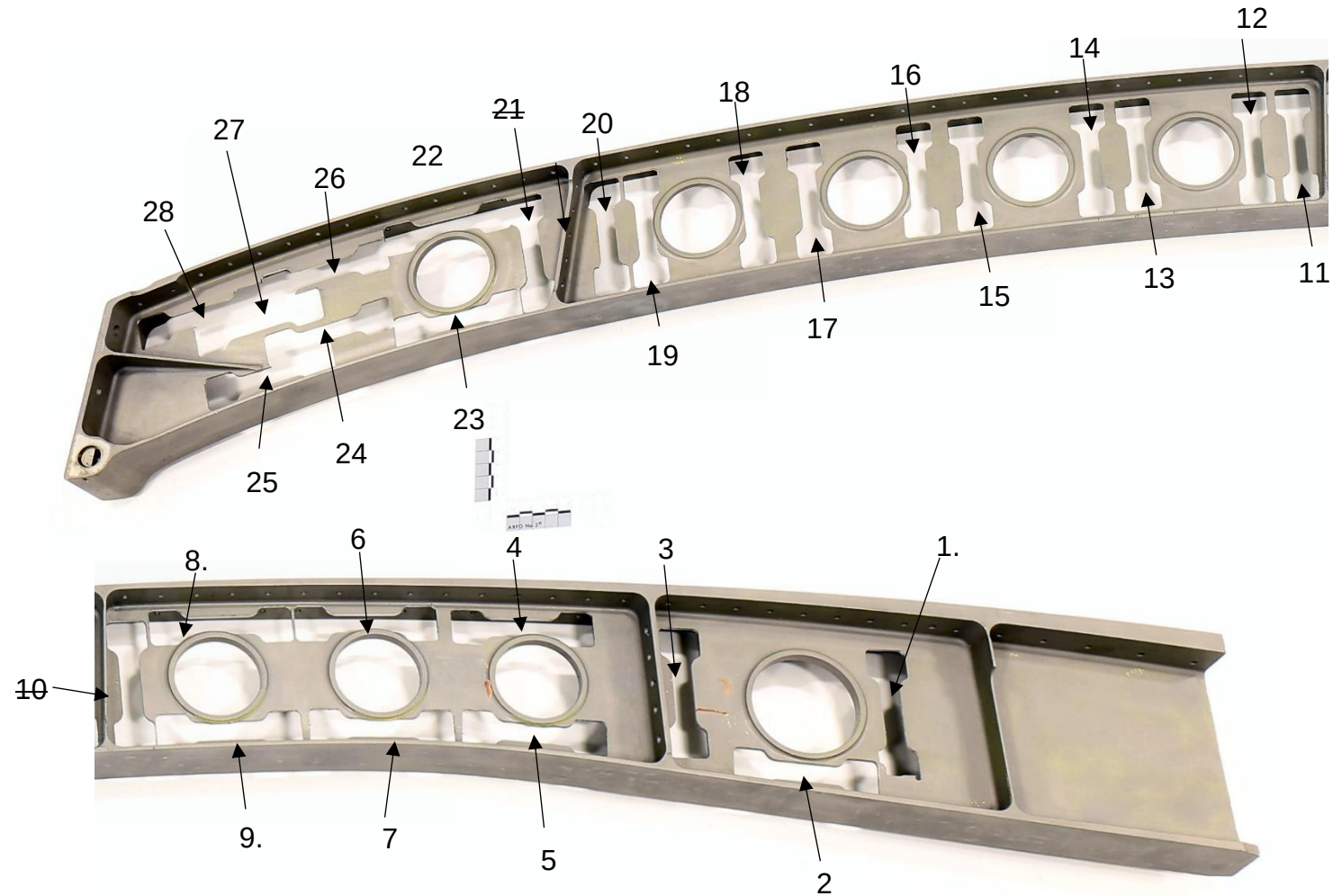


**Figure C4. Showing the location of the 53.1°C tensile test specimens taken from Frame C2.**

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**Annex D: Hardness testing results.****Table D-1: Showing the hardness test results for sample C2-7.**

| Indent No. | Sample C2-7            |       |       |       |       |
|------------|------------------------|-------|-------|-------|-------|
|            | No. of hours at 53.1°C |       |       |       |       |
|            | 0                      | 12    | 24    | 36    | 48    |
| 1          | 159                    | 159   | 161   | 159   | 155   |
| 2          | 158                    | 158   | 159   | 160   | 155   |
| 3          | 160                    | 160   | 159   | 160   | 156   |
| 4          | 158                    | 158   | 159   | 156   | 155   |
| 5          | 158                    | 158   | 158   | 157   | 155   |
| 6          | 161                    | 161   | 160   | 154   | 154   |
| 7          | 159                    | 159   | 159   | 158   | 155   |
| 8          | 159                    | 158   | 159   | 158   | 154   |
| 9          | 160                    | 160   | 160   | 158   | 155   |
| 10         | 157                    | 157   | 159   | 159   | 155   |
| 11         | 161                    | 161   | 159   | 159   | 157   |
| 12         | 158                    | 158   | 159   | 158   | 156   |
| 13         | 162                    | 162   | 159   | 158   | 156   |
| 14         | 161                    | 161   | 160   | 160   | 155   |
| 15         | 166                    | 160   | 160   | 158   | 157   |
| 16         | 160                    | 160   | 159   | 159   | 155   |
| 17         | 156                    | 156   | 158   | 157   | 155   |
| 18         | 159                    | 159   | 160   | 157   | 153   |
| 19         | 160                    | 160   | 158   | 158   | 156   |
| 20         | 157                    | 157   | 159   | 160   | 154   |
| Average    | 159.5                  | 159.1 | 159.2 | 158.2 | 155.2 |

**Table D-1: Showing the hardness test results for sample C2-10.**

| Indent No. | Sample C2-10           |     |     |     |     |
|------------|------------------------|-----|-----|-----|-----|
|            | No. of hours at 53.1°C |     |     |     |     |
|            | 0                      | 12  | 24  | 36  | 48  |
| 1          | 154                    | 154 | 154 | 152 | 158 |
| 2          | 151                    | 151 | 154 | 151 | 160 |
| 3          | 151                    | 151 | 156 | 152 | 158 |
| 4          | 155                    | 155 | 153 | 154 | 158 |
| 5          | 151                    | 151 | 155 | 154 | 158 |
| 6          | 152                    | 152 | 155 | 152 | 158 |
| 7          | 153                    | 153 | 155 | 154 | 158 |

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|         |       |       |        |        |       |
|---------|-------|-------|--------|--------|-------|
| 8       | 152   | 152   | 156    | 155    | 159   |
| 9       | 155   | 155   | 155    | 155    | 156   |
| 10      | 153   | 153   | 156    | 156    | 158   |
| 11      | 155   | 153   | 156    | 157    | 160   |
| 12      | 156   | 156   | 156    | 155    | 159   |
| 13      | 154   | 154   | 154    | 156    | 159   |
| 14      | 154   | 154   | 157    | 159    | 159   |
| 15      | 156   | 156   | 156    | 155    | 159   |
| 16      | 155   | 155   | 156    | 155    | 158   |
| 17      | 154   | 154   | 155    | 154    | 159   |
| 18      | 156   | 156   | 156    | 153    | 158   |
| 19      | 154   | 154   | 156    | 153    | 159   |
| 20      | 155   | 155   | 154    | 153    | 161   |
| Average | 153.8 | 153.7 | 155.25 | 154.25 | 158.6 |

**Table D-1: Showing the hardness test results for sample C2-Section.**

| Indent No. | Sample C2-Section      |       |       |        |       |
|------------|------------------------|-------|-------|--------|-------|
|            | No. of hours at 53.1°C |       |       |        |       |
|            | 0                      | 12    | 24    | 36     | 48    |
| 1          | 154                    | 154   | 157   | 152    | 158   |
| 2          | 151                    | 151   | 154   | 151    | 160   |
| 3          | 151                    | 151   | 156   | 152    | 158   |
| 4          | 155                    | 155   | 153   | 154    | 158   |
| 5          | 151                    | 151   | 155   | 154    | 158   |
| 6          | 152                    | 152   | 155   | 152    | 158   |
| 7          | 153                    | 153   | 155   | 154    | 158   |
| 8          | 152                    | 152   | 156   | 155    | 159   |
| 9          | 155                    | 155   | 155   | 155    | 156   |
| 10         | 153                    | 153   | 156   | 156    | 158   |
| 11         | 153                    | 153   | 156   | 157    | 160   |
| 12         | 156                    | 156   | 156   | 155    | 159   |
| 13         | 154                    | 154   | 154   | 156    | 159   |
| 14         | 154                    | 154   | 157   | 159    | 159   |
| 15         | 156                    | 156   | 156   | 155    | 158   |
| 16         | 155                    | 155   | 156   | 155    | 159   |
| 17         | 154                    | 154   | 155   | 154    | 158   |
| 18         | 156                    | 156   | 156   | 153    | 159   |
| 19         | 154                    | 154   | 156   | 153    | 161   |
| 20         | 155                    | 155   | 154   | 153    | 159   |
| Average    | 153.7                  | 153.7 | 155.4 | 154.25 | 158.6 |

## Annex E: Transmission electron microscopy.

### E.1 An introduction to electron microscopy.

For more than a century, the wavelength of visible (blue) light was considered to be one of the major limiting factors for the resolution of an optical (light) microscope. Ernst Abbe is accredited with defining the minimum resolution (diffraction limit) of an optical microscope as approximately half the wavelength of light (Abbe, 1872)<sup>132</sup> and presented the following formula<sup>133</sup>:

$$d_{min} = \frac{\lambda}{2n \sin \alpha}$$

**or**

$$d_{min} = \frac{0.612 \lambda}{n \sin \alpha}$$

|        |           |   |                                                                                |
|--------|-----------|---|--------------------------------------------------------------------------------|
| Where: | $d_{min}$ | = | the minimal resolvable distance,                                               |
|        | $\lambda$ | = | the wavelength of light, (0.612 $\lambda$ is the wavelength of visible light). |
|        | $n$       | = | refractive index of a medium.                                                  |
|        | $\alpha$  | = | half the angle of the cone of light from a specimen plane.                     |

In 1924 Louis de Broglie proposed in his PhD thesis (De Broglie, 1942)<sup>134</sup> the theory that akin to Einstein's theory of special relativity, along with light quanta (photons), other particles such as electrons neutrons and protons also exhibit wave-like properties. This was later supported by the Davisson-Germer experiment (Davisson and Germer, 1927)<sup>135</sup>.

The De Broglie wavelength is defined by:

$$\lambda = \frac{h}{p} = \frac{h}{mv}$$

Where a wavelength  $\lambda$  is associated with a particle such as an electron which is related to its momentum  $p$  (*mass x velocity*), through Planck's constant  $h$  ( $6.626 \times 10^{-34}$  J s). As electrons are accelerated to almost the speed of light (through increasing voltages) their effective mass increases, which is referred to as *relativistic mass*<sup>136</sup>. One expression of relativistic momentum is electron volts<sup>137</sup>. Therefore, as electrons are passed through different voltages, the relativistic momentum varies and so does the wavelength of the electrons. By determining the momentum in electron volts:

$$\lambda = \frac{h}{\sqrt{2meV}}$$

Where:

|     |   |                                                             |
|-----|---|-------------------------------------------------------------|
| $m$ | = | the rest mass of an electron ( $9.109 \times 10^{-31}$ kg), |
| $e$ | = | the charge of an electron ( $1.60 \times 10^{-19}$ C),      |
| $V$ | = | the accelerating voltage.                                   |

This can be approximated to<sup>138</sup>:

$$\lambda = \sqrt{\frac{1.5}{V}} \text{ nm}$$

The velocity of the electrons that are used for TEM work are sufficiently close to the speed of light that relativistic effects must be taken into account.

Therefore, acceleration voltages for TEM are also calculated with a relativistic correction (Goodhew, Humphreys and Beanland, 2001)<sup>139</sup>:

$$\lambda = \left[ \frac{1.5}{(V + 10^{-6} V^2)} \right]^{1/2}$$

As can be seen in Table 19, increasing the accelerating voltage reduces the electron wavelength, resulting in a far superior resolvable distances in comparison with an optical microscope.

The first electron microscope was constructed in 1931 by Ruska and Knoll (Ruska, 1986)<sup>140</sup>. However, it was Ruska and von Borries in 1932<sup>141</sup> (who invented the short-focal-length magnetic pole-piece lens) who are often credited as being the fathers of Transmission Electron Microscopy (TEM), as their contribution enabled Ruska to develop the first electron microscope that surpassed the resolution of light microscopes. Manfred von Ardenne published a review of the history of electron microscopy (Ardenne, 1985)<sup>142</sup> which highlights the individuals and groups of researchers that were involved in the development of electron microscopes in the 1930-40's and lists their notable achievements in a single table (Ardenne, 1985)<sup>143</sup>.

From the Abbe formula, the diffraction limit of an optical microscope is reported as being between  $0.2^{144}$ -  $0.5^{145}$   $\mu\text{m}$  whereas the diffraction limit of a typical electron microscope is  $0.004$  nm ( $0.000004$   $\mu\text{m}$ )<sup>xliv</sup>.

**Table 19: Showing calculated De Broglie wavelengths for various accelerating voltages.**

| Accelerating Voltage ( $V$ ) in kV | DeBroglie Wavelength $\lambda$ (nm) | Relativistic Wavelength $\lambda$ (nm) |
|------------------------------------|-------------------------------------|----------------------------------------|
| 5                                  | 0.0173                              | 0.0173                                 |
| 15                                 | 0.0100                              | 0.0099                                 |
| 20                                 | 0.0087                              | 0.0086                                 |
| 100                                | 0.0039                              | 0.0037                                 |
| 160                                | 0.0031                              | 0.0028                                 |
| 200                                | 0.0027                              | 0.0025                                 |
| 300                                | 0.0022                              | 0.0020                                 |

## E.2 The transmission electron microscope.

Whilst the theoretical advantages of electron wavelength can be shown by calculations, the practical application provides further challenges. Electrons are more susceptible to being impeded by air molecules than light, therefore all electron microscopy is undertaken in a vacuum which is required to be greater than  $10^{-10}$  Pa

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<sup>xliv</sup> It should be noted that Eric Betzig, Stefan W. Hell and William E Moerner were awarded the 2014 Nobel Prize in Chemistry for using the fluorescence of molecules to surpass the then-perceived minimum optical microscope resolution of  $0.2\mu\text{m}$ .

which is roughly equivalent to  $10^{-7}$  atmospheres<sup>146</sup>. As previously shown in Table 1, the relativistic electron wavelength at 100 kV is  $\sim 0.0037$  nm. This wavelength value is far less than the diameter of an atom (0.01-0.05 nm)<sup>147</sup>. However, in practice this is not achievable due to aberration of the electron beam. However, a TEM is still capable of magnifying over 10,000,000 time or more<sup>148</sup>

### **E2.1 The electron gun.**

An electron 'gun' provides the source of the electrons. Typically, this is either a tungsten filament (this is the cheapest method) or more expensive crystals, such as lanthanum hexaboride ( $\text{LaB}_6$ ) emitter (which is used on the Phillips CM20 TEM at SHU) are used to generate the electrons. The emitter is enclosed within a Wehnelt cylinder which is held at a negative charge which condenses a cloud of free electrons at the tip of the emitter. The Wehnelt cylinder contains a small hole at the bottom, beneath which an anode accelerates electrons from the emitter towards the anode. The electrons that contact the anode are returned to the power supply and the electrons that pass through the anode become the electron beam<sup>149</sup>. Where the beam converges (i.e. the point of smallest cross section of the beam) is called the *gun crossover*. The *gun crossover* provides the source of illumination in electron gun systems. A schematic of a TEM can be seen in Figure 90.

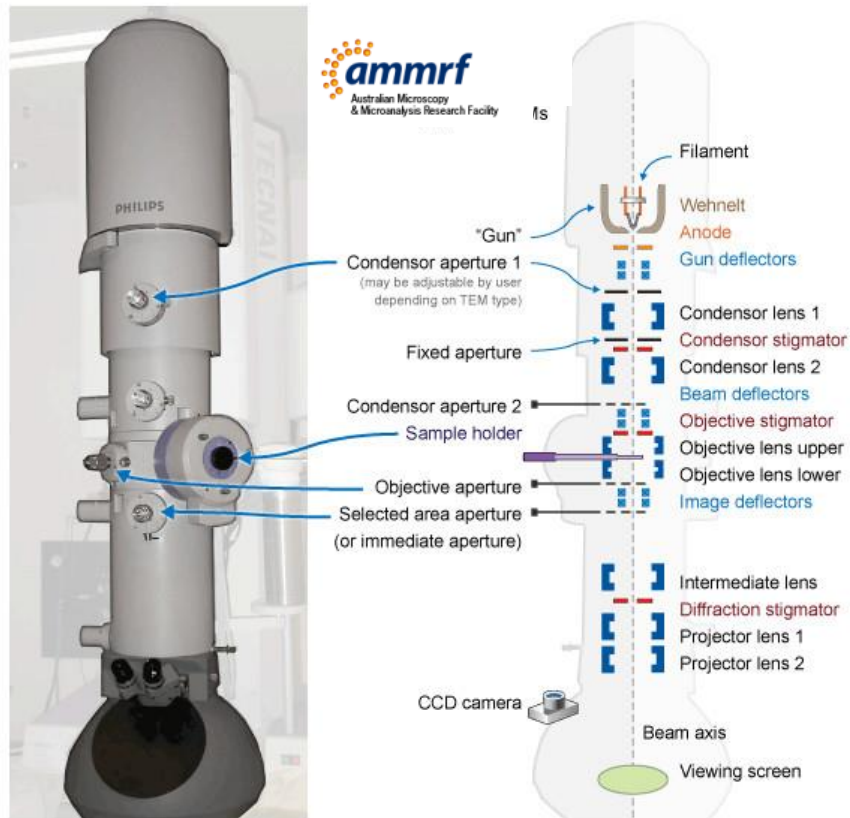


**Figure 90:** Showing a schematic of an example Transmission Electron Microscope<sup>150</sup>.

## E2.2 Lenses in the TEM.

The beam of electrons in an electron microscope is focussed using lenses in the same manner as light microscope. However, whereas light microscopes use glass lenses, an electron microscope uses electromagnetic fields to shape the beam. The electromagnetic field is provided by electromagnetic coils and the strength of the magnetic field is controlled by the current which passes through the coil. The effect of the electromagnetic field on an incident electron is such that the electron will be subjected to a radial effect. This applies a velocity to the electron and it will travel in a helix with a consistently reducing radius until the beam converges to a point – akin to the focusing of light by a glass lens. By altering the current of the lens, the focal point



of the beam can be altered. A visual representation of the helical effect can be seen in Figure 91.

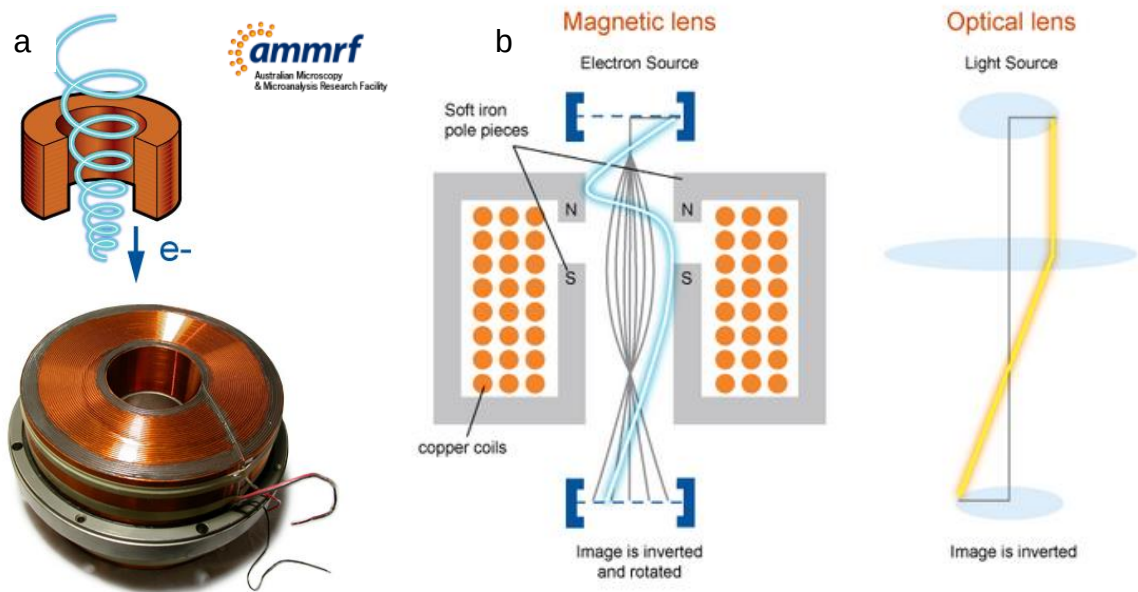


**Figure 91: Showing (a) the shape of the beam as it passes through a lens and (b) the rotation of the electron beam in contrast to a light microscope<sup>151,152</sup>.**

One point of note is that spiralling of the electrons through a series of lenses does result in a rotation of the image as seen by the operator. Whilst there is no distortion of the image that is produced, it can be disorientating at higher magnifications.

### **E2.3 The condenser system.**

Situated below the electron gun are a set of condenser lenses. A typical arrangement is two lens (designated C1 and C2 respectively) and situated between them is a condenser aperture. The C1 lens sets the demagnification of the gun crossover and the minimum achievable *Spot Size* for the remainder of the microscope. The condenser aperture limits the portion of the electron beam that illuminates the specimen by setting the convergence angle. The C2 lens focusses the convergence

angle of the beam. This is commonly referred to as the *Intensity* and depending on whether the beam is under focussed, focussed or over focussed the intensity will illuminate varying areas of the specimen ((i), (ii) and (iii) Figure 92 respectively).

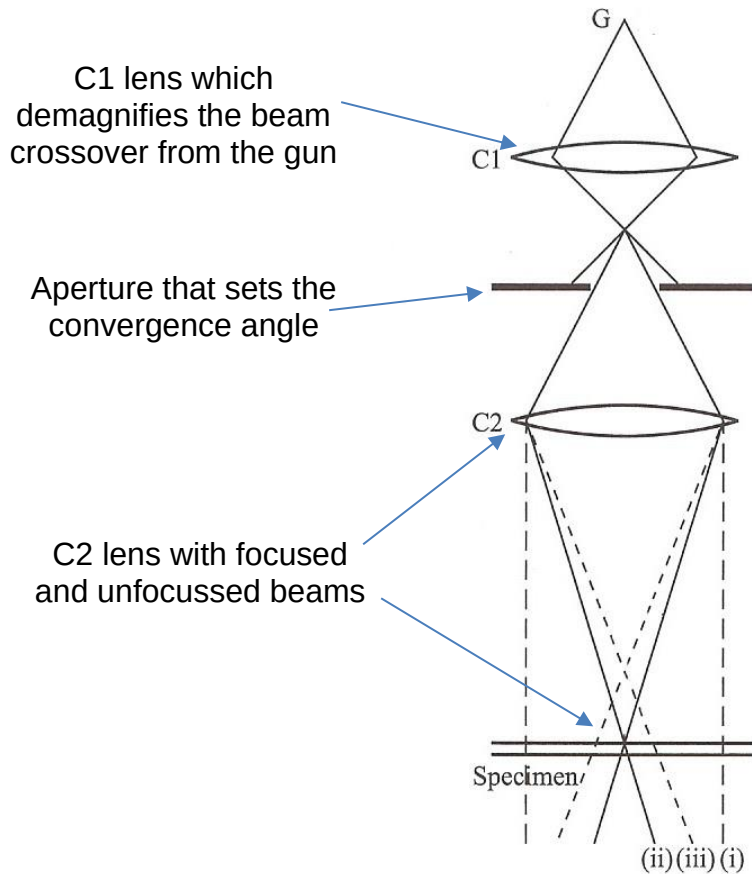


**Figure 92: Showing a typical condenser setup in a TEM.**<sup>153</sup>

Depending on the type of image required, the illumination provided by the condenser system can be adjusted to improve image quality for the operator. For general imaging, a larger spot size, a parallel (wider) beam and a standard condenser aperture provide a greater illuminated area for viewing with sufficient intensity to exam a specimen.

#### **E2.4 The specimen chamber and specimen holder.**

Unlike other electron microscopes, to allow for transmission to occur, TEM specimens are held in the middle of the column to allow the portion of the beam that transmits through and forms the image to be further refined and adjusted. The specimen holder is a long rod that is withdrawn from the column and contains a 3 mm diameter hole for the placing of specimens. TEM specimens are very small and require extensive preparation. Specifically, TEM samples must be suitably thin to allow the electron beam to pass or transmit through the sample (hence Transmission Electron Microscopy). The thickness of a TEM sample is governed by the accelerating voltage; most instruments operate between 100-400 kV<sup>154</sup>. For the purposes of this project, the TEM at SHU was operated at 200 kV. A typical TEM sample (or foil) will be less than 100  $\mu\text{m}$  thick, prior to electrolytic thinning. The specimen holder is designed to tilt the specimen in one axis without moving laterally and therefore to achieve this *eucentric height*, the holder can be adjusted. The eucentric height is a very important factor when imaging using diffraction patterns.

#### **E2.5 The objective and intermediate lenses.**

The objective lens which is situated under the specimen, forms the first (intermediate) image or diffraction pattern. Either of these is then enlarged by a set of projector lenses before being displayed for the operator to view. The first projector or *intermediate lens* is adjusted between the image and diffraction modes by focussing on either the image plane of the objective lens (for the image) or the back focal plane of the objective (for the diffraction pattern). Both of these modes are illustrated in Figure 93.

The final magnification of the image is the responsibility of the second projector lens. When working in diffraction mode, the magnification of the diffraction pattern is related to the effective *camera length* and is an essential factor in the measurement of diffraction spots and in the calibration of a TEM for diffraction pattern analysis.

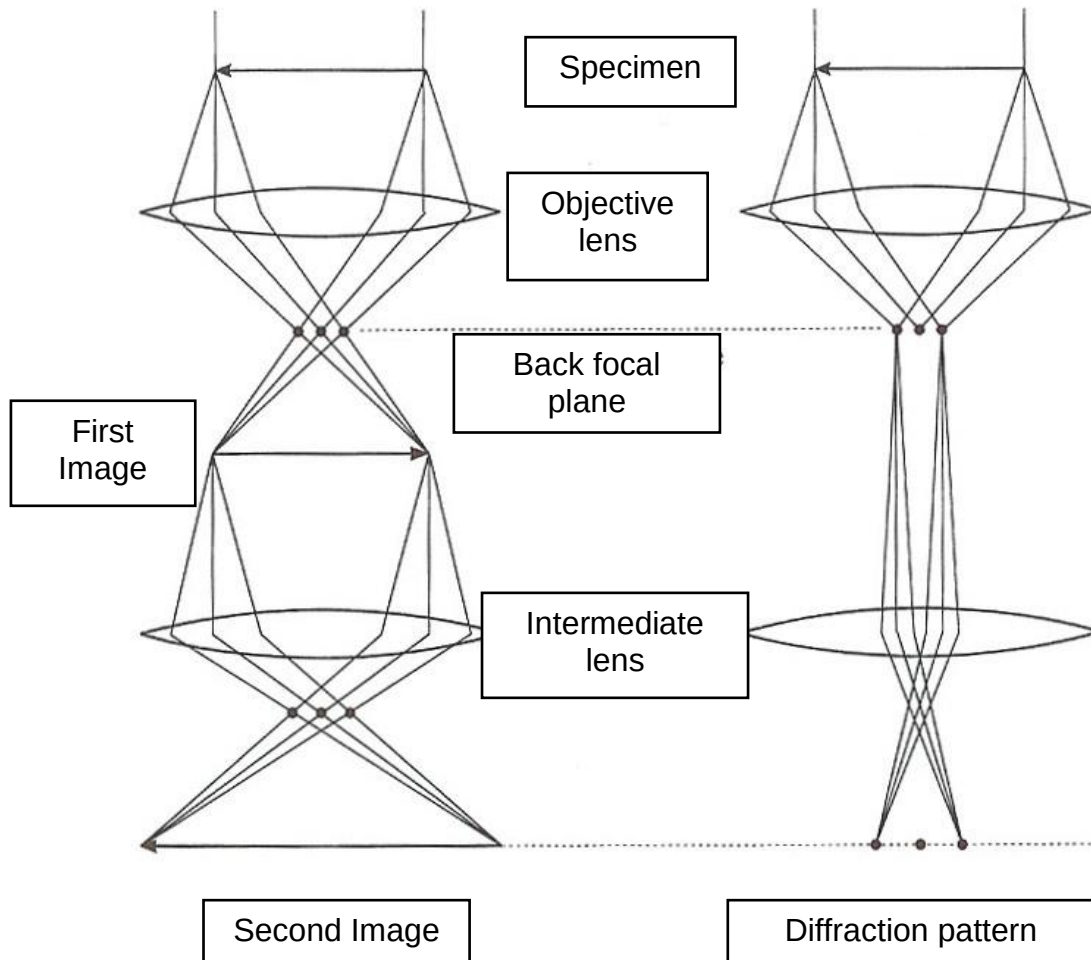


**Figure 93:** Showing the objective and first intermediate lens in both (a) image and (b) diffraction modes<sup>155</sup>.

### **E2.6 Electron scattering.**

In electron microscopy, it is the electrons that enter and leave the specimen that generate the image. There is a high probability that the electrons will interact with the constituent atoms in the specimen and be *scattered*. The probability of an electron being scattered is dependent on the cross-section (more atoms increases the chance of scattering). However, in thin foils such as are used in TEM, electrons will tend to either pass straight through the specimen or be scattered once. Thicker specimens such as are used in an SEM can result in single, plural or multiple occurrences of scattering.

When electrons interact with atoms in a specimen, the resultant scattering is considered to be either *elastic* or *inelastic*. Elastic scattering is defined as: post interaction with the electrostatic charge of an atom, if there is no discernible change in energy in the electron (although the direction of travel may have altered) the process is classed as elastic (otherwise known as Rutherford scattering). Inelastic scattering occurs when there is a noticeable reduction of energy from the incident electron. The majority of the kinetic energy lost from the incident electron will become heat within the specimen however a small portion of the energy can result in secondary effects which (among others) may occur as light, X-rays or Auger or secondary electrons<sup>156</sup>. These effects, which include backscattered electrons, are used in the analytical aspects of electron microscopy.

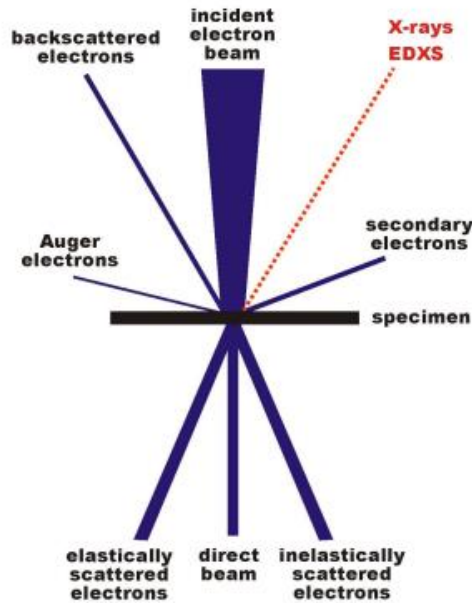


**Figure 94:** Showing some of the resultant signals from the interaction of an electron beam impacting a specimen<sup>157</sup>.

The probability  $p(\theta)$  of an electron being elastically scattered through an angle  $\theta$  is shown as:

$$p(\theta) \propto \frac{1}{E_0^2 \sin^4 \theta}$$

Where  $E_0$  = the energy of the primary electron.

Therefore, the probability of small angle scattering is more likely to occur than high angle scattering. However, the probability of scattering occurring decreases as energy of the electron increases.

### **E2.61. Electron scattering in thin specimens.**

If a beam of electrons is directed at a suitably thin sample for example a simple cubic crystal, the incident electrons will be elastically scattered by some of the atoms in the crystal. In Figure 95, some of the electrons from the incident beam (I) has been

elastically scattered by interacting with some of the constituent atoms (A and B). If the intensity of the beam is sufficient for transmission of the electrons through the specimen, one of three effects can occur to the incident electrons:

1. No deviation and the electrons transmit through the specimen unaltered (T),
2. Electrons diffract as a result of interaction with the crystal planes (D). The distance between the planes (atomic spacing) is shown by  $d$ ,
3. Electrons are scattered in other directions (N).

For diffracted (D) electrons, if the path length between the waves diffracted from A and B are in precipitates (i.e. constructive interference occurs) then path length between the waves must be an integral number. For this to be true in Figure 95, then  $CB + BE = n\lambda$  where  $n$  = an integer and  $\lambda$  is the wavelength of the diffracted electron beam.

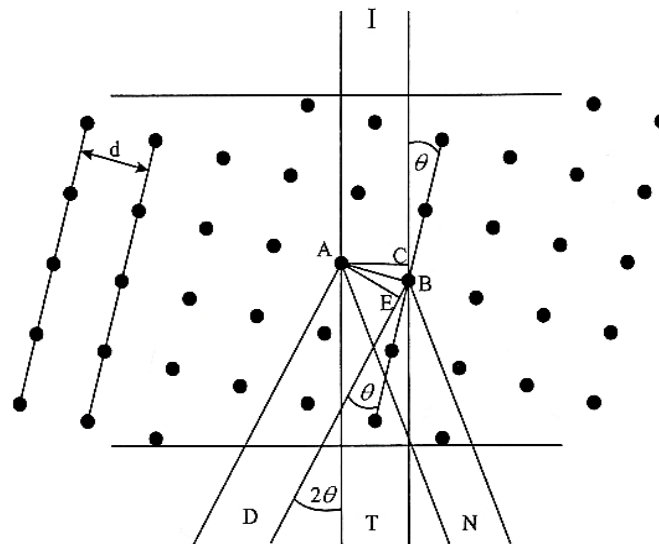


**Figure 95:** Showing the scattering of a beam of electrons in thin crystalline sample (Goodhew, Humphreys and Beanland, 2001)<sup>158</sup>

Further to this in Figure 95,  $CB = BE = d \sin \theta$  and ultimately this can be written as:

$$2d \sin \theta = n\lambda$$

This is commonly known as Bragg's law of diffraction. Where the diffracted electrons meet the criteria of Bragg's law (i.e. the waves are perfectly in phase), they produce bright spots or reflections in the SADP, which can be shown on photographic film or a phosphor screen.

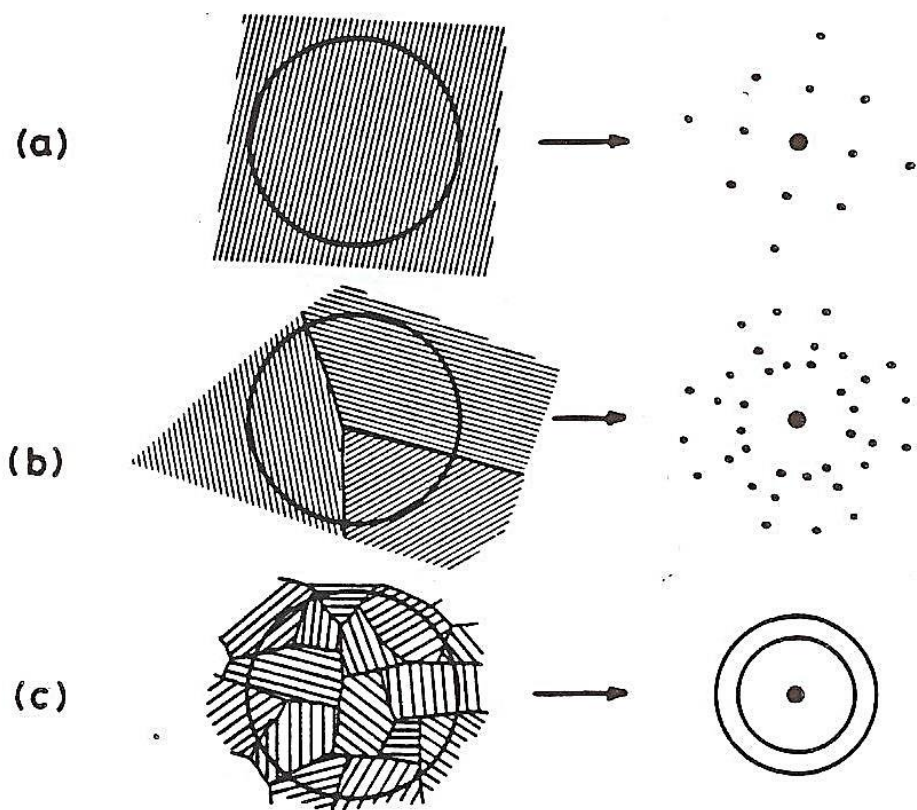


**Figure 96: Showing diffraction spot patterns for a) a single crystal, b) a small number of grains, c) a large number of grains.**

As shown in Figure 96a, diffraction of a single crystal will provide a diffraction pattern which can be used for further investigation (see Section 5). If a number of grains, with different crystal orientations are diffracted at the same time (Figure 96b), a number of



spot patterns will be visible and will begin to form in a circular pattern. For a polycrystalline material (Figure 96c) the spots form rings. This is often used for calibration of the TEM, using standards, such as thallus chloride, see Section 5.1.2 for calibration of the TEM.

### **E2.7 Specimen preparation.**

As has already been mentioned, TEM specimens are very thin foils that need to be electron transparent. See Section 5.1.1 for the sample preparation technique used in this work.

**Annex F: Particle diameter measurements.**

| No. | Sample 5  |       | Sample 6i         |       | No. | Sample 5  |       | Sample 6i         |       |
|-----|-----------|-------|-------------------|-------|-----|-----------|-------|-------------------|-------|
|     | (Ambient) |       | 36 hours @ 53.1°C |       |     | (Ambient) |       | 36 hours @ 53.1°C |       |
|     | T         | NT    | T                 | NT    |     | T         | NT    | T                 | NT    |
| 1   | 21.61     | 31.77 | 32.65             | 29.27 | 51  | 26.90     | 20.57 | 26.16             | 16.77 |
| 2   | 22.39     | 27.37 | 33.48             | 24.35 | 52  | 19.23     | 24.01 | 25.53             | 20.96 |
| 3   | 26.47     | 34.29 | 23.39             | 24.97 | 53  | 18.53     | 21.32 | 21.46             | 25.59 |
| 4   | 13.96     | 26.85 | 27.29             | 23.87 | 54  | 24.71     | 31.35 | 23.40             | 22.67 |
| 5   | 16.30     | 22.57 | 23.14             | 22.83 | 55  | 23.66     | 18.61 | 32.81             | 22.07 |
| 6   | 17.28     | 16.87 | 17.02             | 21.00 | 56  | 26.50     | 20.65 | 32.41             | 28.76 |
| 7   | 19.16     | 21.32 | 18.57             | 21.96 | 57  | 27.71     | 20.56 | 27.17             | 17.76 |
| 8   | 23.20     | 30.88 | 31.66             | 24.94 | 58  | 21.69     | 30.03 | 29.82             | 21.96 |
| 9   | 15.94     | 25.18 | 26.94             | 26.48 | 59  | 21.50     | 23.77 | 28.39             | 23.19 |
| 10  | 17.08     | 20.09 | 27.35             | 28.86 | 60  | 25.46     | 23.46 | 21.45             | 19.11 |
| 11  | 24.32     | 26.96 | 21.80             | 27.76 | 61  | 21.30     | 18.61 | 24.90             | 24.82 |
| 12  | 26.52     | 37.23 | 25.47             | 31.45 | 62  | 25.55     | 26.21 | 24.13             | 16.06 |
| 13  | 25.71     | 26.44 | 31.18             | 22.33 | 63  | 19.39     | 23.19 | 20.32             | 20.47 |
| 14  | 15.08     | 21.13 | 21.86             | 24.91 | 64  | 26.49     | 27.25 | 24.32             | 27.16 |
| 15  | 19.02     | 25.96 | 26.80             | 20.07 | 65  | 23.84     | 23.37 | 30.65             | 24.32 |
| 16  | 22.93     | 24.88 | 32.67             | 24.27 | 66  | 25.71     | 37.67 | 22.48             | 25.58 |
| 17  | 28.12     | 22.57 | 37.03             | 31.69 | 67  | 15.50     | 22.68 | 20.10             | 25.73 |
| 18  | 25.90     | 19.23 | 20.81             | 27.12 | 68  | 22.49     | 22.73 | 28.87             | 19.98 |
| 19  | 24.30     | 23.94 | 20.02             | 22.23 | 69  | 21.42     | 22.92 | 17.47             | 19.93 |
| 20  | 26.18     | 25.75 | 20.97             | 23.09 | 70  | 18.17     | 24.26 | 20.29             | 23.37 |
| 21  | 29.09     | 17.69 | 34.11             | 21.44 | 71  | 24.42     | 28.88 | 24.83             | 21.54 |
| 22  | 19.90     | 26.01 | 26.73             | 25.04 | 72  | 23.38     | 23.51 | 25.21             | 21.96 |
| 23  | 31.11     | 24.11 | 27.00             | 27.69 | 73  | 28.66     | 17.35 | 33.55             | 21.46 |
| 24  | 20.63     | 19.27 | 30.51             | 19.91 | 74  | 23.99     | 25.80 | 22.83             | 22.63 |
| 25  | 35.46     | 19.72 | 32.88             | 21.53 | 75  | 15.14     | 29.93 | 22.15             | 26.61 |
| 26  | 24.92     | 15.48 | 25.68             | 25.35 | 76  | 20.88     | 42.29 | 25.06             | 21.96 |
| 27  | 27.01     | 17.78 | 20.81             | 21.32 | 77  | 24.64     | 42.44 | 19.90             | 25.74 |
| 28  | 28.92     | 16.08 | 28.73             | 29.14 | 78  | 23.23     | 16.32 | 26.16             | 31.58 |
| 29  | 16.76     | 17.18 | 18.56             | 34.43 | 79  | 15.58     | 17.41 | 19.46             | 21.45 |
| 30  | 23.92     | 37.31 | 25.42             | 26.00 | 80  | 24.45     | 23.56 | 17.64             | 21.07 |
| 31  | 21.55     | 26.32 | 17.34             | 22.45 | 81  | 22.15     | 41.21 | 19.52             | 24.66 |
| 32  | 24.15     | 18.84 | 28.41             | 25.32 | 82  | 25.71     | 23.83 | 18.51             | 26.15 |
| 33  | 28.59     | 33.32 | 24.74             | 26.63 | 83  | 19.28     | 34.31 | 17.78             | 36.79 |
| 34  | 21.05     | 26.37 | 24.72             | 21.32 | 84  | 25.02     | 27.75 | 14.30             | 22.64 |
| 35  | 26.04     | 29.20 | 22.72             | 25.10 | 85  | 24.99     | 28.98 | 29.98             | 24.08 |
| 36  | 23.20     | 28.95 | 22.55             | 25.90 | 86  | 29.71     | 15.05 | 34.22             | 24.48 |

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|                   |       |       |         |       |     |       |       |       |       |
|-------------------|-------|-------|---------|-------|-----|-------|-------|-------|-------|
| 37                | 29.43 | 21.90 | 28.01   | 23.08 | 87  | 31.28 | 20.19 | 28.74 | 23.43 |
| 38                | 23.74 | 43.09 | 19.74   | 21.44 | 88  | 31.83 | 26.77 | 30.70 | 21.42 |
| 39                | 30.26 | 21.26 | 19.33   | 24.19 | 89  | 23.44 | 26.77 | 18.23 | 26.16 |
| 40                | 18.32 | 19.86 | 18.76   | 18.54 | 90  | 22.81 | 26.45 | 21.26 | 18.49 |
| 41                | 20.39 | 21.62 | 19.39   | 18.88 | 91  | 26.28 | 20.45 | 18.79 | 27.58 |
| 42                | 27.69 | 29.23 | 26.24   | 28.47 | 92  | 29.57 | 14.67 | 21.88 | 22.74 |
| 43                | 19.75 | 19.83 | 26.69   | 24.96 | 93  | 20.21 | 19.92 | 18.12 | 26.15 |
| 44                | 26.53 | 19.92 | 19.90   | 27.91 | 94  | 24.63 | 16.85 | 15.72 | 26.39 |
| 45                | 25.21 | 23.91 | 25.77   | 25.73 | 95  | 20.21 | 21.18 | 16.70 | 28.15 |
| 46                | 21.68 | 33.29 | 30.03   | 24.21 | 96  | 33.29 | 34.59 | 17.79 | 23.76 |
| 47                | 23.92 | 18.31 | 25.14   | 17.03 | 97  | 25.55 | 21.28 | 23.99 | 19.33 |
| 48                | 22.63 | 34.98 | 27.81   | 23.04 | 98  | 30.48 | 25.83 | 24.54 | 26.43 |
| 49                | 16.18 | 24.30 | 29.93   | 20.52 | 99  | 23.07 | 32.07 | 20.76 | 15.54 |
| 50                | 15.08 | 28.79 | 21.34   | 25.94 | 100 | 33.25 | 15.67 | 19.58 | 19.88 |
| T =<br>Threshold  |       |       |         |       |     |       |       |       |       |
| NT = No threshold |       |       | Average |       |     | 23.57 | 24.90 | 24.39 | 23.92 |

All measurements in nm.

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