The use of friction stir welding for the production of Mokume Gane-Type materials

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Abstract

Mokume gane is a highly desirable and unique decorative material but production is difficult at both workshop and industrial levels. This paper describes a novel method for producing mixed metal, multi-colored, layered materials using friction stir welding (FSW) and compares the results with mokume gane made by a number of different conventional methods. FSW was invented in 1991 by TWI, a UK-based research and technology organization, and has found widespread use in the joining of aluminum and, more recently, steels. By adapting FSW it has been possible to successfully bond many layers of dissimilar metals such as silver, copper and brass, etc., while simultaneously producing unique patterns in the metal, minimizing further processing, reducing waste and potentially lowering costs. The technique avoids many of the problems experienced in workshop production, e.g., melting or lack of bonding, while allowing production of small, customized ingots and mass production of large sheets, with up to several square meters possible.

1. Introduction

1.1 Introduction to Mokume Gane

Mokume gane is a unique process where sheets of different metal alloys are bonded together into a laminated billet. This billet is then carved or milled to expose interior layers before being hammered or rolled into a flat sheet, which is then formed into jewelry or hollowware. Traditionally, the technique is used to make sheet with a pattern similar to wood grain (mokume gane translates as wood-grain metal), although contemporary metalworkers have expanded the palette of patterns beyond this. Mokume gane is thought to have originated with Denbei Shoami (1651-1728), who lived in Akita prefecture in Japan. Shoami used the swordsmith's forge-welding techniques with Japanese copper and gold alloys to produce a layered piece of metal. Shoami first produced a carved laminate based on lacquer techniques before developing more complex wood-grain patterns.

Mokume gane has a long history of adoption and development in the West. The earliest technical description of the process was by Raphael Pumpelly, a mining expert and mineralogist invited to Japan by the Tokugawa shogun government in 1862.[1] American manufacturers, who operated without hallmarking restrictions, embraced the new material. Tiffany & Co. shocked the established European manufacturers in the Paris exhibition of 1878 when they received the Grand Prize for silverware incorporating mokume gane. The Japanese were also impressed by Tiffany's work. "The specimens of work made of the new metal formed by the lamination of all the noble metals and their alloys astonished even the Japanese, from whom the method was learned, many articles being purchased by them; their chief commissioner buying one of the principal specimens for his government."[2]

A growing number of craftspeople in North America, Europe and, most recently, Japan are now making jewelry using mokume gane. The use of colored karat gold alloys, platinum, and palladium has increased the market share for this jewelry. Although it is an ancient art, it has appeal in today's markets where "mass customization" and the idea of designer products are key.

The production of mokume gane materials is a difficult process. Traditional liquid-phase bonding is achieved by heating the stack of sheets in a hearth until close to melting point. Pressure is then applied to achieve a good bond in the billet. Consistently determining the bonding temperature by eye requires a maker with great experience. More advanced manufacturers use solid-state diffusion bonding to achieve control and consistency in the bonding process.[3] However, the stacked laminate produced by these methods needs to be extensively machined, rolled, and worked to develop the unique patterns. New research now looks at the production of net- and near-net-shape mokume gane jewelry using production techniques such as powder sintering[4] and hot-extrusion.[5]

This paper outlines the exciting potential that friction stir welding (FSW) possesses in terms of producing materials that have similar attributes to those produced by current mokume gane manufacturers. FSW allows us to simultaneously bond and produce unique patterns in the metal, reducing time and production costs. FSW also removes the size constraints that restrict all current production methods, allowing for the production of billets many meters square.

1.2 Introduction to Friction Stir Welding and Processing

Invention

Friction stir welding was invented in 1991 by TWI's Wayne Thomas and is a patented, friction forge welding technique. Consistent with the more conventional methods of friction welding, which have been practiced since the early 1950s, the weld is made in the solid phase, that is, with no melting of the components to be joined. Since its invention, the process has received worldwide attention, and today many companies around the world are using the technology in production, particularly for joining aluminum alloys.

Principle of Friction Stir Welding

In conventional friction stir welding a cylindrical, shouldered tool with a profiled probe is rotated and slowly plunged into the joint line between two pieces of sheet or plate material, which are butted together (a butt weld). The parts have to be clamped onto a backing bar in a manner that prevents the abutting joint faces from being forced apart. Frictional heat is generated between the wear-resistant welding tool and the material of the work pieces. This heat causes the latter to soften without reaching melting point. The plasticizing of the material allows the rotating tool to be traversed along the weld line (Figure 1). The plasticized material is transferred from the leading edge of the rotating tool probe to the trailing edge and is forged by the intimate contact of the tool shoulder with the surface and the flow induced by the rotating pin profile. A schematic of the process is shown in Figure 2. It leaves a solid-phase bond between the two pieces, just like traditional forge welding. Friction stir welding thus combines the best attributes of a forging process with a joining technique to create high-quality welds in most metals. Those metals and alloys that can be extruded or forged most easily are very amenable to being joined by friction stir welding.

Because it avoids any of the metals becoming liquid, it allows better control over the materials' microstructures and avoids the formation of undesirable porosity, intermetallic phases or grain growth. In fact, the high heating/cooling rates and high strain rates involved often can alter the microstructure in a way that can benefit the materials' properties.

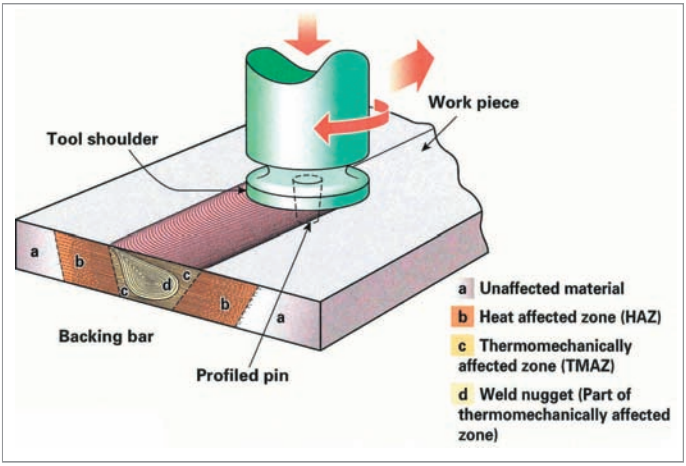

Figure 1 *Schematic of the friction stir welding process for a butt weld*

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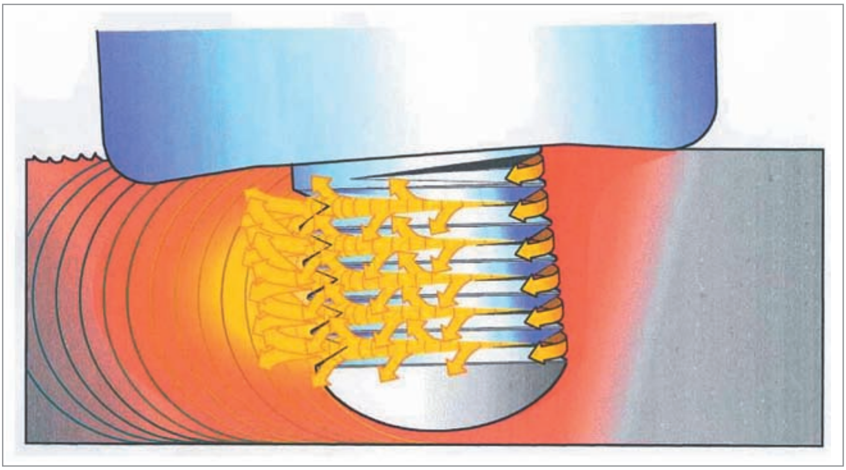


Figure 2 *Schematic of the friction stir process using a typical tool. Arrows show typical material movement. Tool is traversing from left to right (side view). Courtesy of TWI*

Benefits of Friction Stir Welding

The advantages of the process result from the fact that the FSW process takes place in the solid phase below the melting point of the materials to be joined. The benefits, therefore, include the ability to join materials that are difficult to fusion weld, for example, 2000 and 7000 aluminum alloys. Friction stir welding can be carried out using purpose-designed equipment or modified existing machine tool technology. The process is also suitable for automation and adaptable robot use. The main advantages are as follows:

- Low distortion and shrinkage, even in long welds
- Excellent mechanical properties in fatigue, tensile and bend tests
- No arc or fumes
- No porosity
- No spatter
- Can operate in all positions
- Energy efficient
- Long tool life (in 6000 series Al alloys >1000m)
- No filler wire required
- No gas shielding for welding aluminum
- No welder certification required
- Some tolerance to imperfect weld preparations—thin oxide layers can be accepted
- No grinding, brushing or pickling required in mass production
- Can weld aluminum and copper of >50mm thickness in one pass

Despite being a relatively new technology, the benefits of friction stir welding mean that it is already in service with many companies worldwide for a wide range of applications, mainly in aluminum. Safety-critical items such as the fuel tanks on spacecraft, including the Space Shuttle, are now friction stir welded as is the new Eclipse business jet. Grades of aluminum that cannot be welded by traditional fusion—gas or electric arc—processes are readily welded by friction stir and the process is also being adopted for welding of copper and mixtures of metals.[6,7,8,9,10,11,12]

More recent developments have seen the welding of steel[13] and the use of more complex weld geometries including corner welding and the use of fill wires to feed materials into a weld. Other materials such as copper, brass and precious metals are inherently suitable for FSW. This paper represents an extension of work on the subject to date, which has mainly focused on structural or electrical applications.

2. Method

2.1 The Friction Stir Equipment at TWI Yorkshire

Powerstir FSW

The initial friction stir welds in this study were made on TWI's PowerStir™ machine based at the South Yorkshire laboratory. This machine has a large working envelope (6m x 3m) and is fully instrumented, allowing monitoring and recording of all significant variables including the forces acting upon the FSW tool. The machine has a maximum down force capability of 150kN and a maximum torque limit of 2480Nm.

Figure 3 *The TWI Powerstir large friction stir machine*

Precision Spindle FSW

Later welds in the study were made using the Precision Spindle TTI (Transformation Technologies International) friction stir welding machine. The machine is fully instrumented to monitor all of the relevant process parameters and is capable of producing a down force of 98kN. The working envelope is 2m x 0.6m. The unique feature of the machine is its high concentricity spindle, which allows research into the ceramic tools being developed for FSW. Machines without a high concentricity spindle are prone to tool chatter, which quickly leads to premature failure of ceramic tools.

***Figure 4** The TWI Precision Spindle TTI friction stir machine*

2.2 Materials

In order to investigate the viability of bonding dissimilar metals in laminate form using friction stir welding, a number of materials were chosen that had properties close to those anticipated in the final product (e.g., precious metals) but for which there was already some experience of FSW and that were readily available and affordable. The materials used in FSW were 99.9% copper, a 60:40 brass and 925 silver, all in the rolled and annealed condition. Precious metals were loaned to the project by Cooksons Precious Metals Ltd. in the UK.

Mokume gane materials produced using a number of conventional methods were used as comparative materials for the study of the bonding mechanism and microstructures typically produced by these methods. There were three types of material: (1) solid-state bonded, (2) liquid-phase bonded and (3) soldered.

The solid-state bonded material was produced from copper, fine silver, and 99% copper. The metal sheets were compressed with torque plates, placed in a stainless steel bag with charcoal granules, and bonded in a kiln.

The liquid-phase bonded mokume was made from copper, fine silver, and 99% Cu. The metal sheets were bound together with heavy binding wire and then heated in a gas forge until the surface of the sheets was seen to be "sweating." The stack was then removed from the forge and hammered while hot to compress.

The solder bonded material was made from fine silver and copper. The metal sheets were soldered together in pairs, cleaned, and then soldered together into a larger laminate block.

The 99% copper alloy used in the above mokume gane materials was a 99Cu1Au alloy known as shakudo.

2.3 Method of Friction Stir Welding Laminates

Conventional friction stir welding was initially developed and optimized for simple butt welds (two plates butted together and a vertical interface between the materials is joined). In the formation of laminate mokume gane materials, the interfaces between the materials are horizontal and also there is more than one of them. This presents a significant challenge in terms of optimizing the friction stir welding process.

The initial test parts were produced from stacked sheets of copper and brass in order to minimize material costs in the early experimental stages. Laminates consisting of Cu and brass sheets were stacked in up to eight alternating Cu/brass layers (typically 1mm or 2mm thick each). Typically a laminate structure measuring 300mm x 150mm was used.

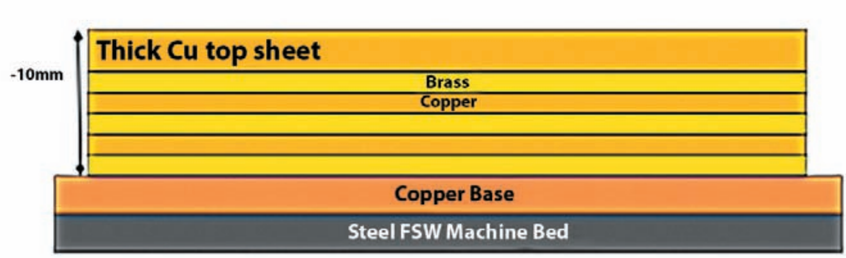


Figure 5 *Schematic of a typical lay-up for FSW of Cu/brass laminates*

Once the early stage feasibility trials with Cu and brass were complete, then samples of Cu and 925 silver were used in a similar format to produce Ag/Cu mokume gane sheet. The metal sheets were used in the as-received condition (rolled and annealed). No cleaning or special treatment of the surfaces was carried out beyond a simple cleaning to ensure there was no loose debris or excessive oil, grease or water present.

Following the results of initial trials, a number of improvements in the way the laminates were stacked and clamped were developed. For example, to avoid waste of Ag sheet and to provide better clamping, a system was devised where the sheets to be welded were contained within a recess in a copper block, a copper sheet was used as the "lid" to this container and the friction stir tool was applied from the "rear" face. In the main example of Cu/Ag, for which results are shown below, a thick sheet of 925 silver was set within a recess in a copper block and a thinner copper sheet used as a lid on the container.

2.4 The FS Tool and the FS Conditions and the Way in Which the Parts Were Processed

The material and design of the friction stir tooling are very important factors in successful FSW. In this case tools made from Nimonic 105 alloy were used. These tools provided both sufficient strength and wear resistance and also a good degree of mixing and bonding within the materials (as demonstrated later in the Results section). The design of the tool tip in particular, incorporating three flutes and a thread, provided very good mixing of materials in both the vertical and horizontal directions.

Figure 6 *A typical friction stir tool as used in the manufacture of FSW mokume gane (largest diameter = 30mm)*

Initial trials were used to determine the typical friction stir conditions for producing bonds between the Cu/brass or Cu/Ag systems. Success was judged as a friction stirred zone that had produced an obvious bond and also minimized or eliminated any voids within the friction stir zone (often a small void can be formed at the lower rear extremity of the friction stir tool probe if the conditions are not ideal). The primary friction stir parameters that were optimized were rotational tool speed (rpm), tool linear traverse speed (mm/minute), downwards force (N) and tool tilt (degrees). Also considered were the speed at which the initial plunge is made and the dwell time at the initial plunge site (to initiate the heat formation).

The formation of a bond between the materials and the mixing of the materials take place only in a region defined by the width and depth of the tool probe and in a small region immediately outside this zone. In order to form a sheet that is fully bonded across its width, the tool needed to make a number of passes to ensure a full bond between the sheets. The exact number of passes and the distance between each pass are among the parameters that require optimization.

2.5 Materials Characterization

The materials that were produced were characterized using a number of methods in order to understand how the two materials had bonded and mixed during the FSW and how FSW materials compared with conventionally produced mokume gane materials.

Ingots were cut from the primary sheet using a band saw and were typically 50mm x 100mm x 7mm-10mm. After FSW the top surface is normally rough and heavily deformed, and this surface was removed to a depth of approximately 1mm using surface grinding. Top surfaces and side surfaces were then ground and polished using standard metallurgical methods in order to observe the mixed metal structure that had been formed. Both transverse and longitudinal sections were prepared in this way. Visual inspection and photography were carried out on samples prepared as described above with particular attention being paid to the presence of any voids or porosity and also the amount of material mixing that had occurred both in the horizontal and vertical directions.

Some polished sections were etched in order to reveal the changes that had occurred in the materials' microstructures after FSW. Grain size and grain morphology were observed using optical microscopy and scanning electron microscopy (SEM). Micro-hardness testing using a Vickers indenter and a load of 300g was used to measure the materials' hardness prior to and after FSW.

The nature of the bonding between the two metallic materials was characterized using optical microscopy and scanning electron microscopy equipped with microprobe elemental analysis (energy dispersive x-ray analysis, EDX). The combination of high magnification SEM and elemental line scans across the metal-to-metal interfaces allowed the characterization of the type of bond formed. The bonding and amount of mixing and deformation varies across the FSW zone and as such the type of bonding was characterized in a number of regions, from the outer regions of the FSW zone to the center of the FSW zone. This was carried out on FSW materials and also on the three conventionally produced Cu/Ag mokume gane materials: (1) solid-state bonded, (2) liquid-phase bonded and (3) soldered.

The three-point bend tests were carried out to obtain qualitative visual data on the strength of the bonds between the laminates and, if the bonds failed, the location of the failure. The samples used were deliberately chosen to contain small voids from non-perfect FSW runs in order to determine if failures initiating at the voids would propagate through the rest of the structure.

Test bars were cut from the ingots and were then subjected to three-point bed testing (Figure 7). The ingots were approximately 65mm x 10mm x 10mm and were tested on an apparatus where the span was 50mm and the rollers were 10mm in diameter. The bars were deformed to the maximum extent possible on the apparatus. The test bars were then examined for evidence of any areas where the metal-to-metal bond had failed. Images of the samples were taken after the bend test.

Figure 7 *The three-point bend test equipment used to test the integrity of the bond in the mokume gane materials made by friction stir welding*

3. Results

3.1 FSW Conditions for Processing Copper, Silver and Brass Laminates

For both Cu/brass and Cu/Ag the range of friction stir parameters within which successful bonding of the metals and void-free friction stir welds were produced was:

Tool: Nimonic 105

Rotational Speed: ~200-400rpm

Traverse speed: ~100-300mm/min

The exact machine setting depended on the type of tool being used, the size and type of laminate stack being processed and the combination of materials.

3.2 Copper/Brass Mokume Gane

The nature of FSW is such that within the friction stir zone (FSZ), i.e., the area through which the tool tip passes, the materials are intimately mixed and highly plastically deformed but no melting takes place. The result is a region within the material in which the laminates of the two materials have been mixed in both a horizontal and vertical direction in a repeating pattern formed by the rotating tool. This mixing and patterning was observed in the various samples that were produced and is shown in the photographs of the polished sections (Figures 8-10). The patterning is formed as an integral part of the bonding process and is one of the major potential advantages of this method of manufacturing.

The initial feasibility work used simple stacked laminates, but it should also be possible to experiment with variations on the lay-up of the materials to be joined/mixed in order to produce the desired pattern, e.g., bars laid side by side and welded together, materials inset vertically into plates of material, etc. The early work on multiple layers of stacked copper and brass sheets was carried out to test the feasibility of bonding multiple sheets and to explore the patterns that could be obtained by using multiple sheets. Images taken from longitudinal and transverse cross section of the copper-brass laminates after friction stir welding are shown in Figure 8 and Figure 10.

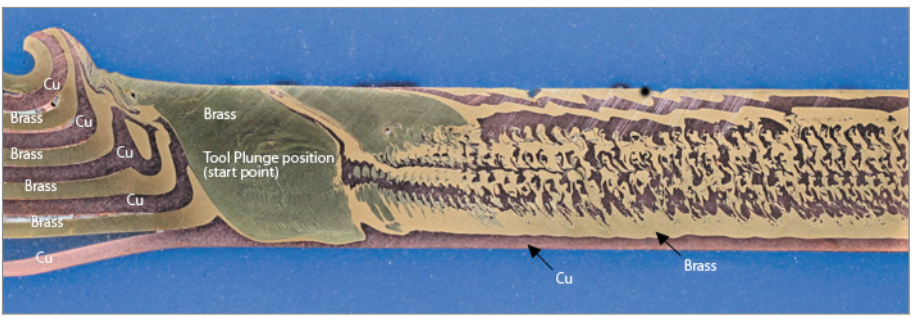


Figure 8 *A longitudinal cross section through a ten-sheet laminate of copper and brass approximately along the center line of the friction stir zone. The tool plunge point (start point) is shown and the original unbounded sheets can be seen to the far left of the image (total ingot thickness ~10mm).*

Figure 9 *A close-up of the patterning formed when FSW was performed on ten alternating sheets of copper and brass (longitudinal section through center of FSZ as shown in Figure 8). Height of ingot ~10mm*

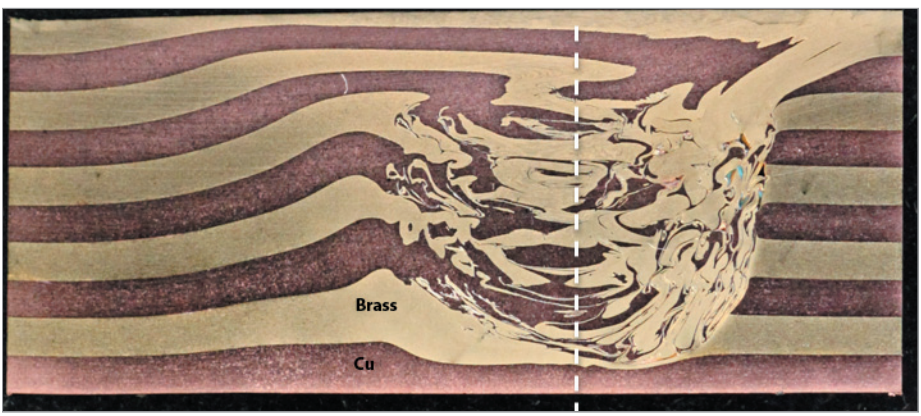


Figure 10 *A transverse section taken through the friction stir zone in a ten-laminate stack of copper and brass. Tool approximate center line is marked as dotted line. Tool was moving into the image along this line. Total ingot thickness ~10mm*

It is clear from Figures 8-10 that the tool produced significant material flow in both the horizontal and vertical directions. Material has been displaced a number of millimeters from its original position, and the copper and brass layers have become intimately mixed. The pattern formed is unstable for the first 10mm or so as the conditions reach equilibrium, but then a stable repeating pattern is formed as the tool and materials reach their steady-state conditions.

In order to explore what kind of patterns would emerge from such a material and to test its behavior during subsequent processing, a sample similar to that shown in Figure 8 was further processed. Samples cut from a FSW stack of Cu/brass sheets were rolled to form a sheet and then worked as would be the case for traditional mokume gane, i.e., shallow craters were gouged into the surface to reveal the layers and then the sheet rolled again until flat. Images of the resulting material are shown in Figures 11-13. The ingot survived the rolling process with no obvious defects or de-bonding occurring. After the initial rolling (Figure 11) the patterns were observed to spread out but to retain an element of repeatability.

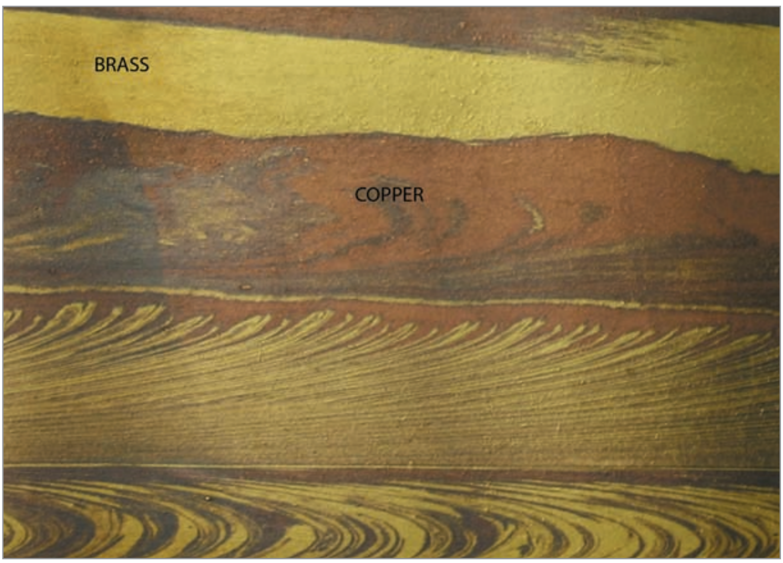


Figure 11 *Image of the patterns formed in ten stacks of Cu/brass FSW mokume gane after rolling (width of image ~90mm)*

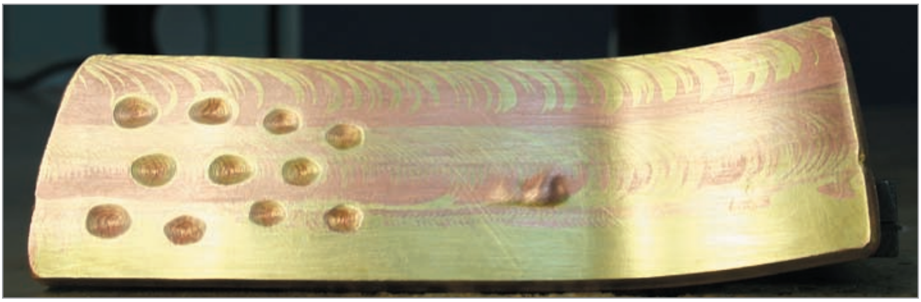


Figure 12 *A rolled sheet of Cu/brass mokume gane made from a FSW laminate of ten sheets of Cu and four sheets of brass. Shallow craters made in the patterned metal are shown prior to the subsequent rolling stage (length ~100mm, width ~90mm).*

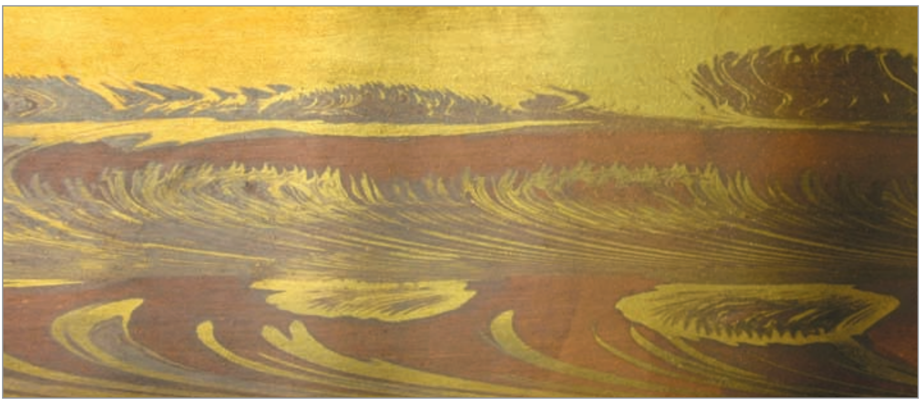


Figure 13 *Patterns formed in Cu/brass FSW mokume gane after the forming of shallow craters and subsequent rolling (width of image ~200mm)*

3.3 Copper/Silver Mokume Gane

The results from an early test on Cu/Ag with two thick sheets of copper and one thin sheet of Ag are shown in Figures 14 and 15. The laminate was formed from two thick sheets of copper with a thin silver layer sandwiched between these two layers. After the removal of the un-bonded edges and surface grinding of the deformed surface, the resulting plate was ~230mm x 100mm x 10mm in size.

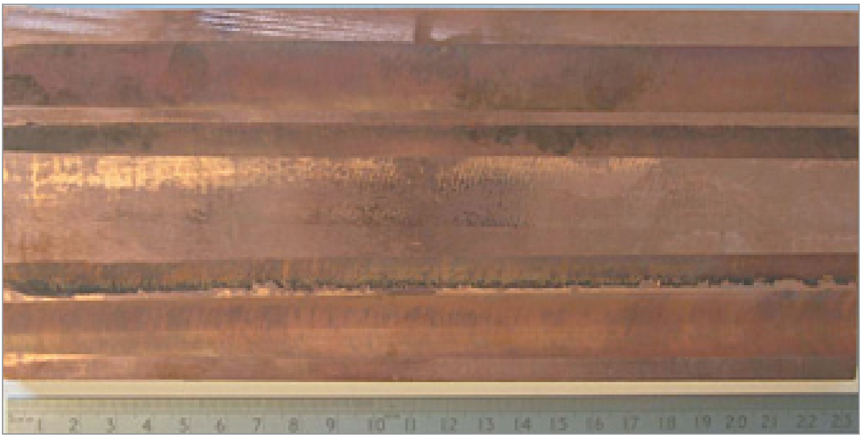


Figure 14 *A view (looking from above) of a Cu/Ag FSW sample measuring ~230mm x 100mm x 10mm. This surface has received six overlapping FSW passes.*

Figure 15 *A transverse section of the plate of Cu/Ag FSW laminate (2 x thick Cu and one thin Ag layer in the center) with six overlapping FSW passes (width shown ~100mm)*

It was clear that the six overlapping passes had bonded the three sheets together well and no voids were observed. The silver sheet had been distorted and moved from its original position and had begun to form a wavy pattern in the copper.

Using a different lay-up, a thick sheet of 925 silver was positioned in a recess inside a copper block and a copper sheet was used to close the container. The friction stir welding was applied from the "rear" of this container. The process of FSW closed the container, both mixing the metals and welding the "lid" to the container.

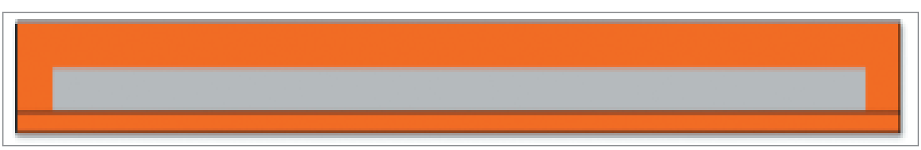


Figure 16 *Schematic of the lay-up for the copper/silver mokume gane sample shown in Figure 17. The silver sheet is encased in a copper recess and a sheet of copper is used to close the container (here shown on the bottom).*

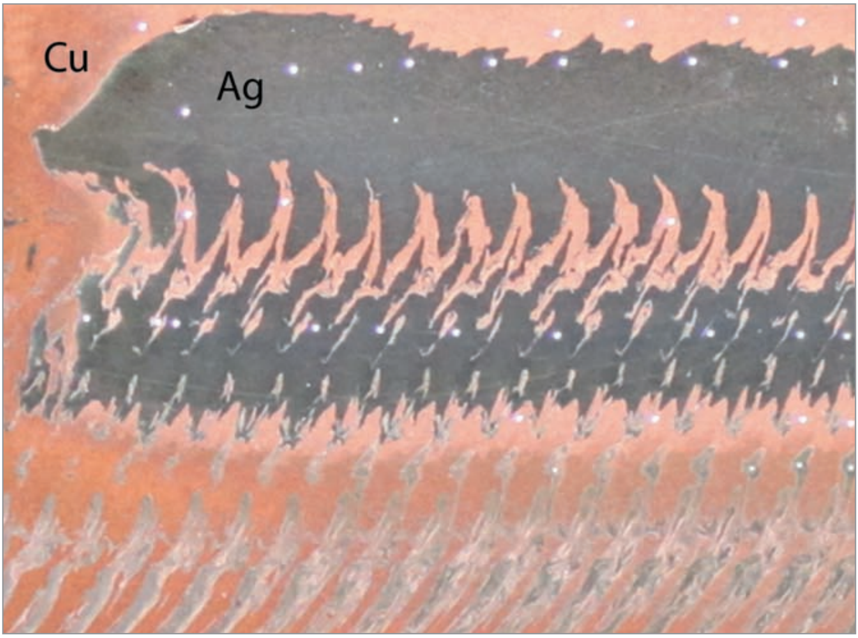


***Figure 17** Longitudinal cross section of a copper/silver mokume gane material made by friction stir processing*

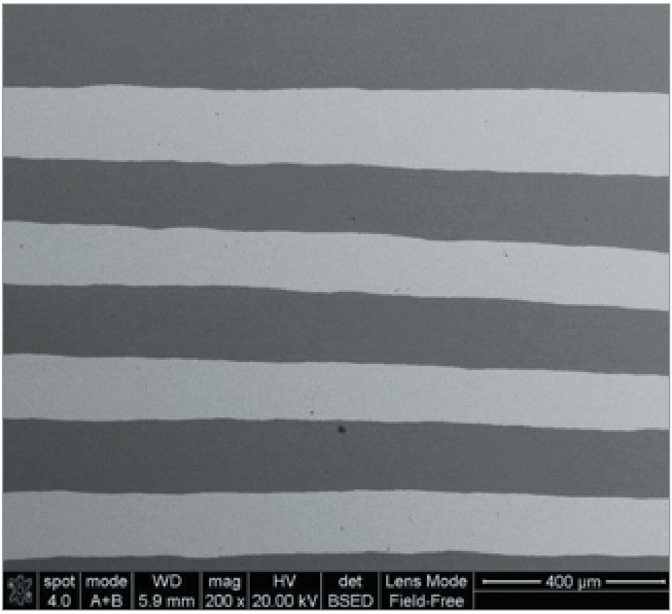
The longitudinal section shown in Figure 17 demonstrates that the copper and silver are well bonded and extensively mixed in both the horizontal and vertical directions and that the patterns formed repeat in a consistent manner.

3.4 SEM/EDX of the Metal-Metal Interface in Traditional Mokume Gane

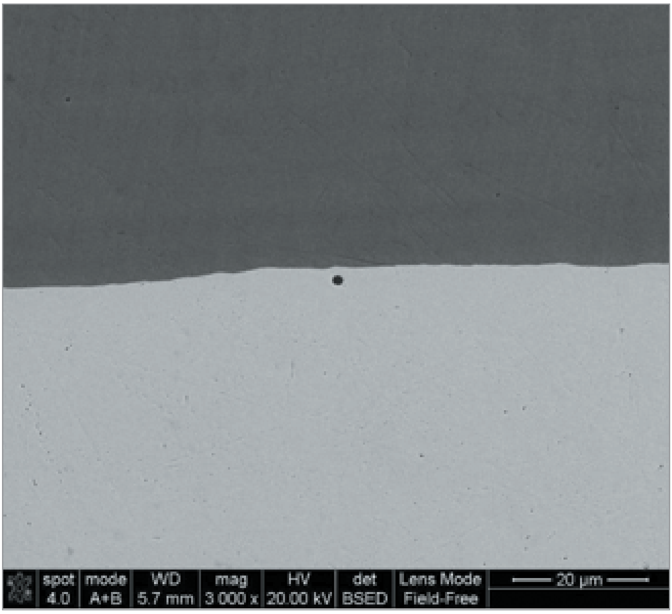
The three conventionally formed mokume gane materials, (1) solid-state bonded, (2) liquid-phase bonded and (3) soldered, were analyzed using SEM and EDX as described above and the results are shown in Figures 18-22.

Solid-State Bonded Mokume Gane

In the solid-state bonded mokume gane the metals were observed to be in intimate contact and there was no porosity or any other significant defects at the interface (Figure 18). An EDX line-scan showed that there was no evidence for any significant inter-diffusion between the metals (Figure 19).



(a)



(b)

Figure 18 *A SEM image (atomic number contrast) showing (a) the alternating layers of Ag (bright) and Cu (dark) in the solid-state bonded mokume gane sample and (b) a higher magnification image of the interface*

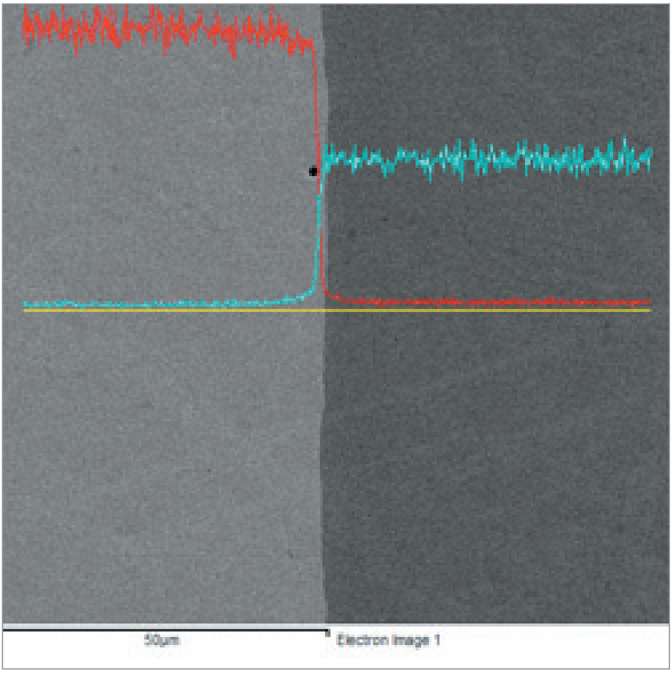


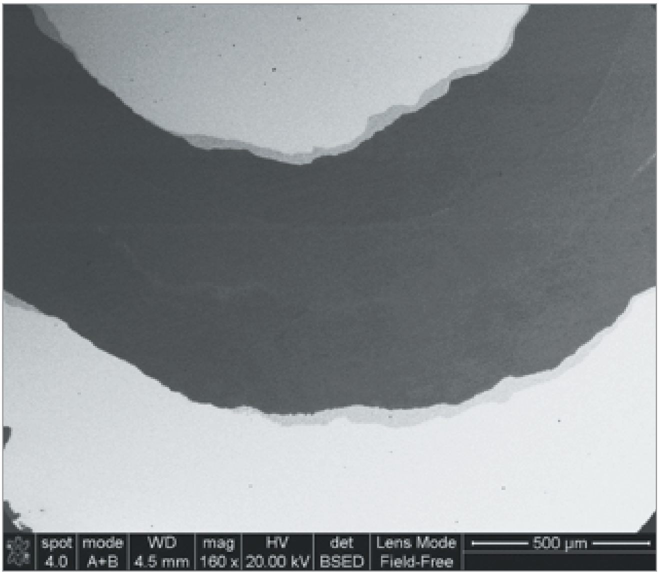
Figure 19 *A SEM image with an overlaid EDX line scan showing the concentrations of Ag and Cu in the two materials. At the interface there is a sharp change between the Ag and the Cu with no evidence of significant diffusion between the two materials.*

Liquid-Phase Bonded Mokume Gane

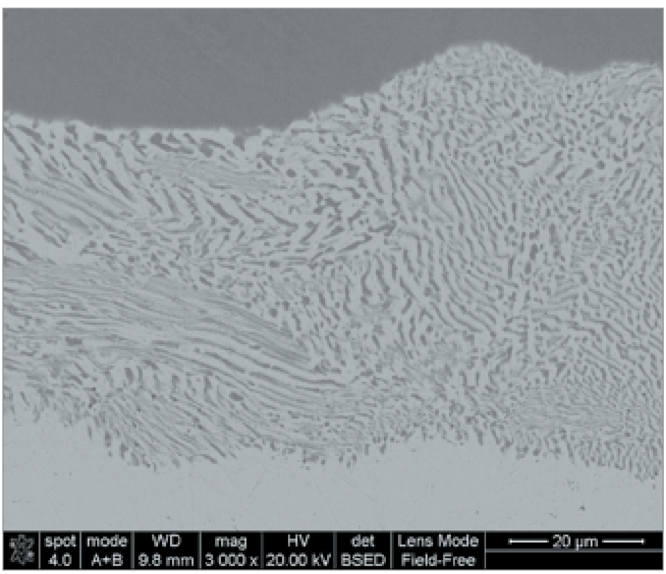
The liquid-phase mokume gane sample had already been processed to form circular patterns and as such the features observed were those present in this condition, i.e., round bulls-eye patterns typical of mokume gane. In the liquid-phase mokume gane there was evidence of the formation of a liquid phase, which involved the solution Cu into the Ag phase and the subsequent formation of the Ag/Cu eutectic structure on re-solidification in a region about 50µm thick and shown in Figures 20 and 21. The boundary between the Ag and Cu materials and the eutectic region was sharp with little evidence of Ag or Cu diffusion beyond the eutectic region (Figure 21).

Soldered Mokume Gane

The soldered mokume gane material was in laminate form. SEM imaging of the cross section revealed the presence of a number of voids at the interfaces between the Ag and the Cu (Figure 22). As was the case for the liquid-phase bonded material, there was evidence for the formation of a liquid phase that has involved the solution Cu into the Ag phase and the subsequent formation of the Ag/Cu eutectic structure at the boundary between the Ag and Cu materials on re-solidification. Analysis of the elemental composition of the eutectic region revealed the presence of zinc (Zn) up to about 5 wt.% in addition to the Ag and Cu, which would be expected for a silver solder material.



(a)



(b)

Figure 20 *A SEM image (atomic number contrast) showing (a) the alternating layers of Ag (bright) and Cu (dark) in the liquid-phase bonded Ag/Cu mokume gane sample. Note the regions of differing contrast located at the interface between the Ag (light) and the Cu (dark) areas, shown close-up in (b).*

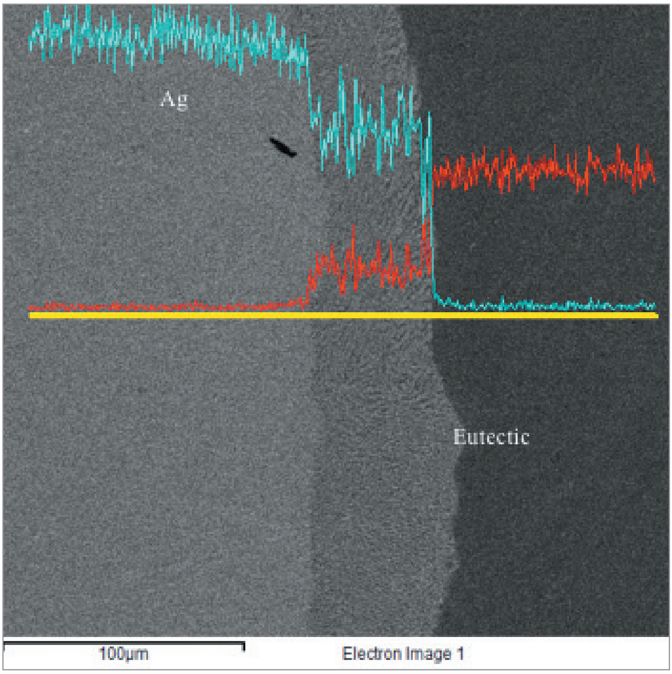
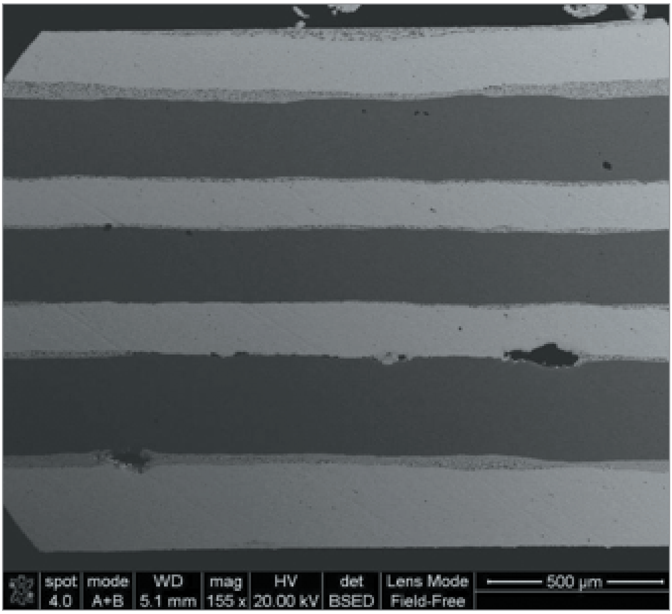
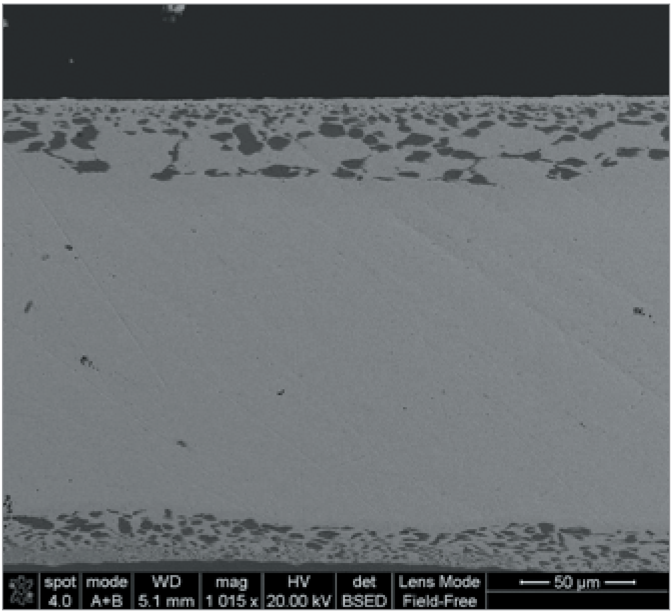


Figure 21 *A SEM image with an overlaid EDX line scan showing the concentrations of Ag and Cu across the interface in a liquid-phase bonded Ag/Cu mokume gane sample showing the eutectic region (both Cu and Ag present), which contains both Ag and Cu and extends over approximately 50µm*



(a)



(b)

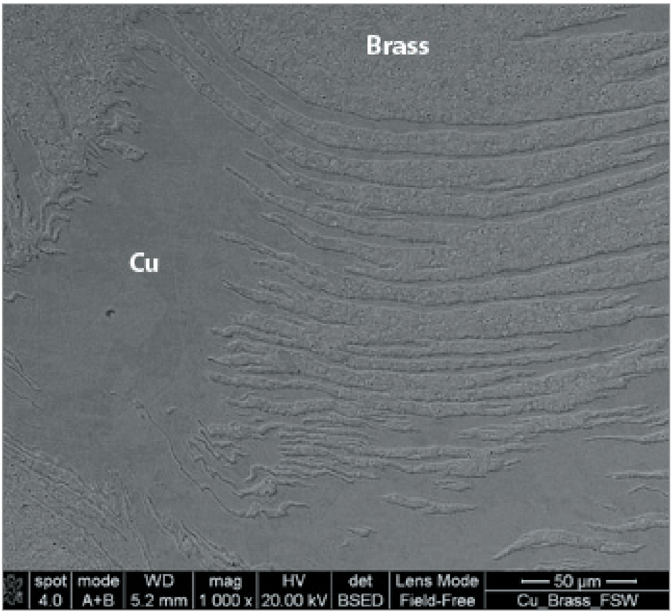
***Figure 22** SEM images (atomic number contrast) showing (a) the alternating layers of Ag (bright) and Cu (dark) in the soldered Ag/Cu mokume gane sample. Note the voids present at the interfaces and the regions of differing contrast located at the interface between the Ag (light) and the Cu (dark) areas, shown close-up in (b).*

Jones

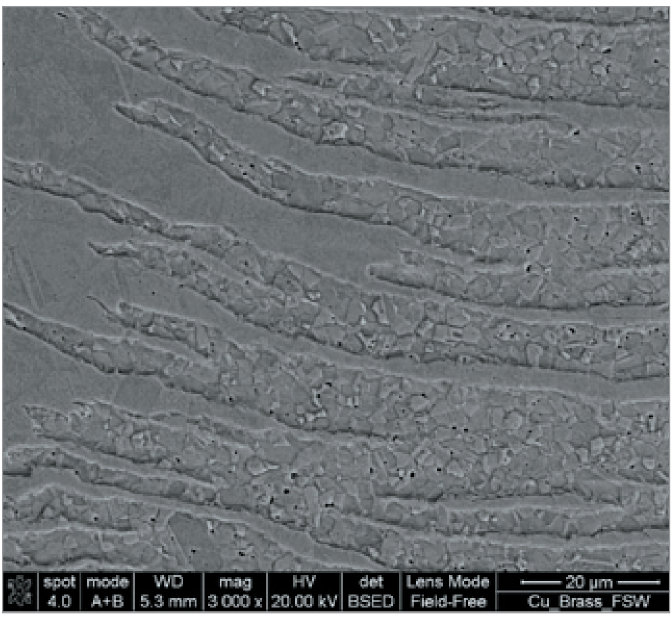
3.5 SEM/EDX of Copper/Brass FSW Mokume Gane

In copper/brass mokume gane materials a degree of mixing of the materials was observed as shown in Figure 8. The interfaces between the copper and brass material are shown in Figures 23 and 24, where the material was etched to differentiate between the copper and brass more easily. The images show that there is a good bond between the copper and brass and no visible voids or porosity, etc.

High magnification of the interface between the copper and brass in the FSZ reveals an intimate bond between the two materials but no significant diffusion or reaction between the two materials. In a region away from the FSZ the interface between the copper and brass was observed to be less well bonded as shown in a comparison of Figure 24 (within friction stir zone) and Figure 25 (away from friction stir zone).



(a)



(b)

Figure 23 *SEM images of a friction stirred zone with intermixed material in copper/brass mokume gane (etched)*

Jones

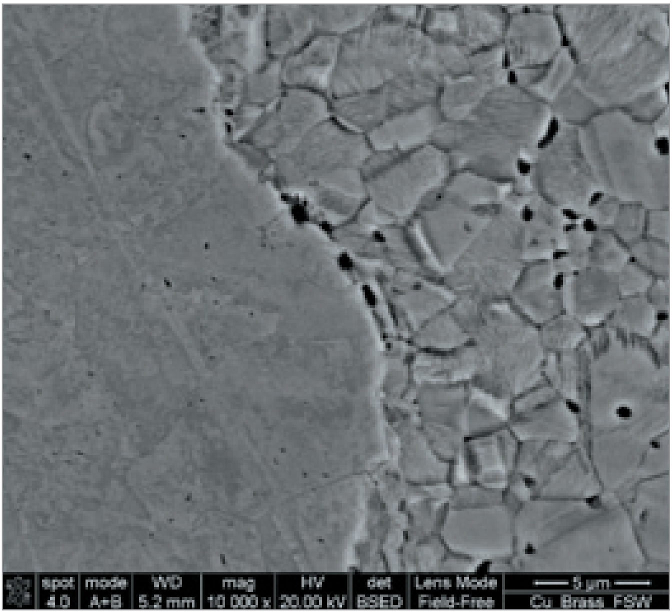


Figure 24 *High magnification SEM image showing the interface between copper (left) and brass (right) within the friction stir zone of copper/brass mokume gane (etched)*

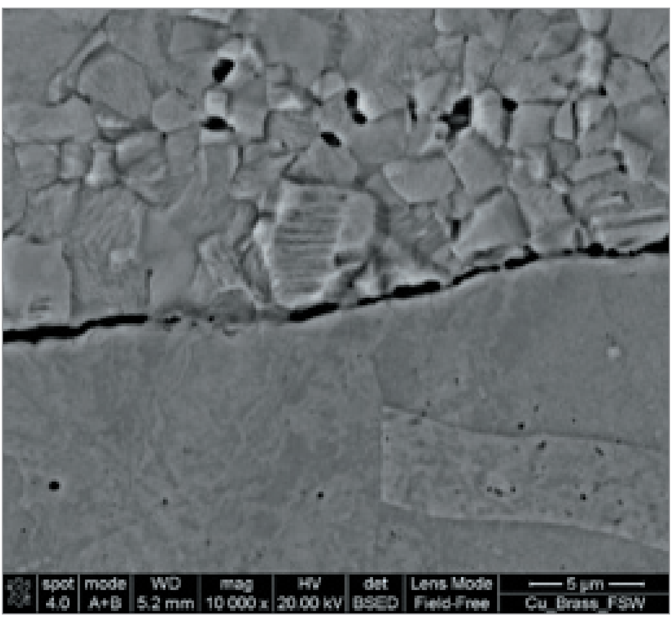


Figure 25 *A SEM image of the interface between copper (bottom) and brass (top) in a region away from the friction stir zone in copper/brass mokume gane (etched)*

3.6 SEM/EDX of FSW Ag/Cu Mokume Gane Materials

A laminate of 925 silver and 99.9% Cu processed by FSW was subject to the same analysis as described above for the conventionally produced mokume gane materials. The SEM images show that in the FSW zone there was intimate mixing of the Cu and Ag phases, forming intricate patterns in the metal that appeared to repeat with some degree of consistency as shown in Figure 26.

The mixed structure viewed at higher magnifications (Figures 27 and 28) reveals a structure that has been formed by a solid-state process rather than through the melting/dissolution/re-solidification process of the liquid-phase and soldered materials. While the Cu content of the original 925 silver is ~7.25 wt.%, the Cu content of the highly mixed zones was measured by SEM/EDX to be ~30-35 wt.% Cu.

The silver material in the friction stir zone has effectively been mechanically alloyed with the copper by the large deformations induced by the rotating friction stir tool, but this has probably been enhanced by the heat introduced by the friction stir process. In regions where the mixing was less intense, on the boundaries of the FSZ, the materials were in intimate contact but there was no evidence of diffusion between the two materials (Figure 29).

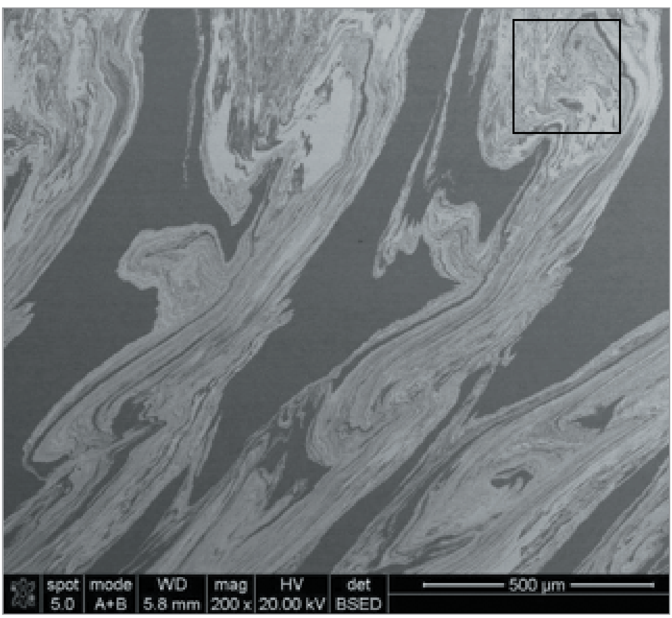


Figure 26 *SEM image (atomic number contrast) showing the alternating patterns of Ag (bright) and Cu (dark) in the FSW Ag/Cu mokume gane sample. The typical area indicated by the box contained 30-35 wt.% Cu.*

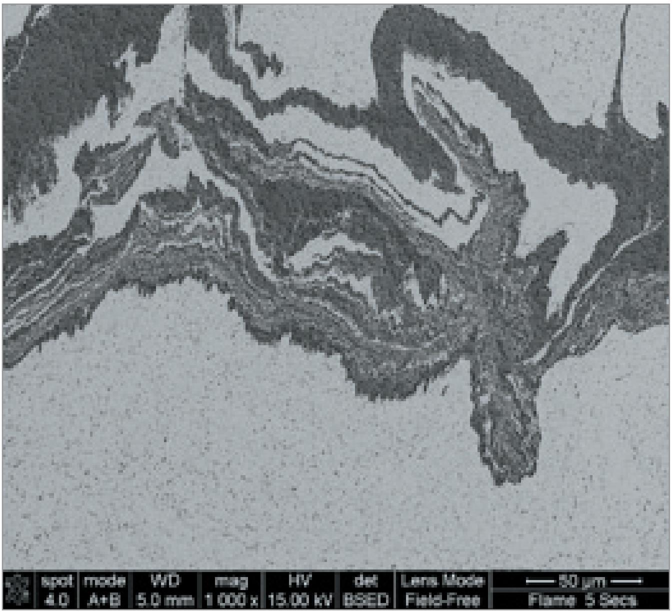


Figure 27 *SEM images of the intimate mixing of Ag and Cu in the Cu/Ag FSW mokume gane sample (etched)*

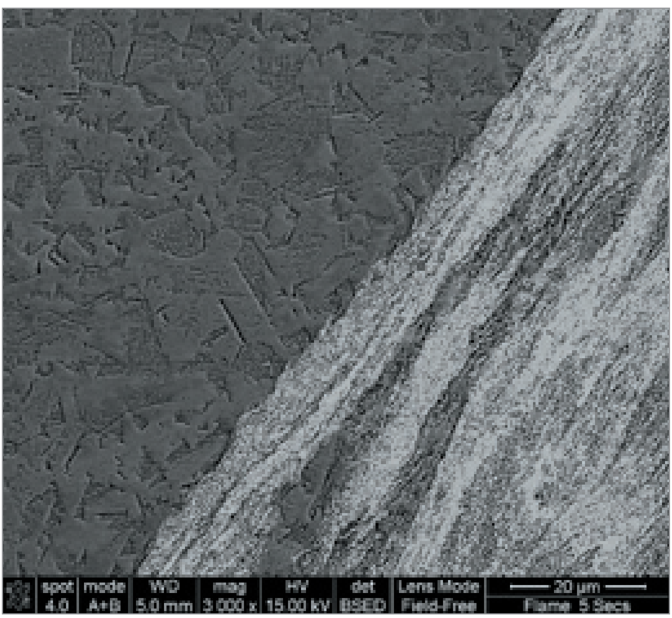


Figure 28 *A SEM image, from within the friction stir zone, of the interface between the intimately mixed Ag/Cu and the Cu in the Cu/Ag FSW mokume gane sample*

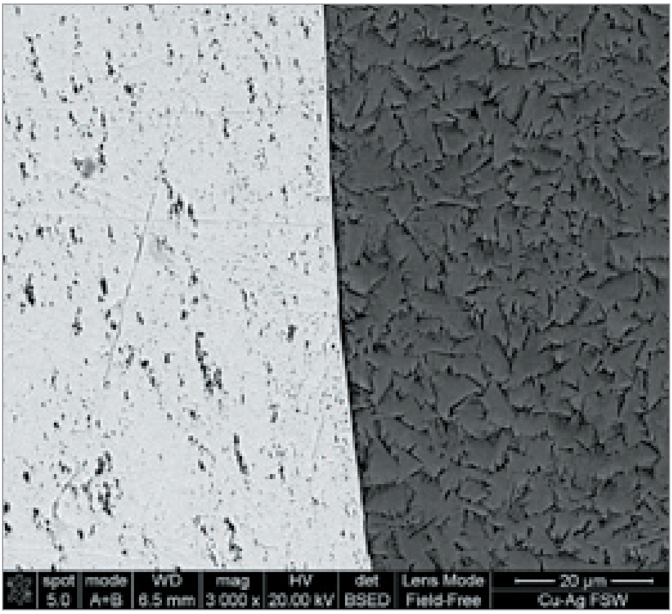


Figure 29 *A SEM image (atomic number contrast) of an etched Cu/Ag sample from a region where the materials' mixing is lower (copper is on the right)*

3.7 Bend Testing of FSW laminates

When three-point bending was applied to a laminate of Cu/brass in the region of a friction stir zone, the test bar was significantly deformed. Inspection of the test bar showed that within the friction stir zone, i.e., where the tool had passed through, the material remained well bonded as shown in Figure 30. However, away from the friction stirred zone, i.e., away from the actual weld, the copper and brass layers had de-bonded and some separation occurred between them. The separation of the layers propagated along the join lines but was stopped when it reached the welded friction stir zones. The void within the friction stir zone was deliberately included in order to determine its influence on the properties. There was no sign of the void leading to failure within the friction stir zone.

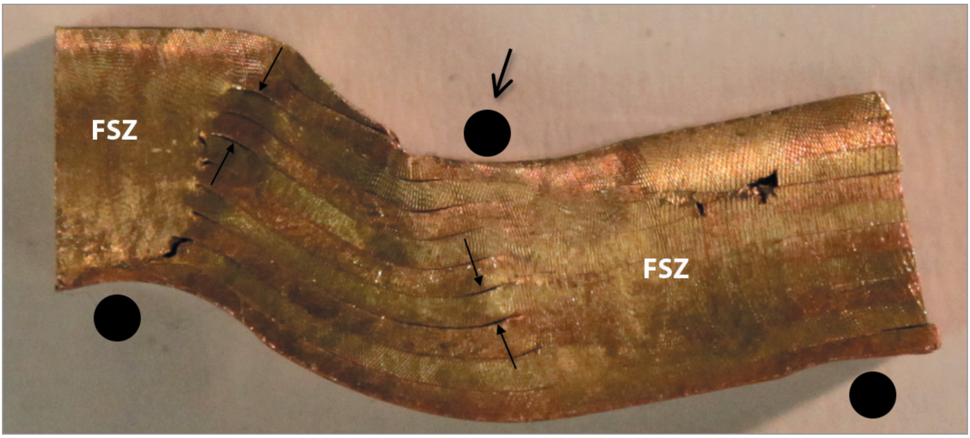


Figure 30 A Cu/brass laminate produced by FSW after a three-point bend test. Support points and bend position and direction of applied force are shown as black circles and arrow. Friction stir zones (FSZ) are labelled and arrows show regions of de-bonding. (height of ingot ~10mm)

4. Discussion

It has been shown that it is possible to use friction stir welding to bond mixed metal laminates in copper and brass and copper and silver. It has been found that successful joining can be carried out using a moderately sized friction stir machine, i.e., the TWI Precision Spindle TTI Friction Stir machine, and relatively inexpensive tools.

Laminates consisting of up to 10mm x 1mm sheets of copper and brass have been successfully processed by using a series of passes of a friction stir tool. Composites of copper and 925 silver have also been processed successfully using FSW.

Within the FSZ both good bonding and material mixing is achieved. The mixing of the two materials being processed is on both a macroscopic and a microscopic scale. The macroscopic mixing provides repeating patterns in the material that are desirable in terms of the manufacture of mokume gane materials.

When conventionally made mokume gane materials were studied using SEM and EDX, two types of bonding were observed. In solid-state bonded materials the boundary between layers was a sharp interface. EDX, whose resolution is limited to 1 micron or so, could not detect a significant diffusion of Cu or Ag into the opposing material. Any bonding was therefore formed at the metal-to-metal interface with metal-to-metal bonding between the layers on a scale less than 1 micron.

In both liquid-phase bonded mokume gane and in the soldered mokume gane there was evidence for the formation of a liquid phase and the subsequent solution of copper into the silver at the boundary (the melting point of Ag being about 120°K lower than that of Cu). The classic Cu/Ag eutectic structure was observed as a layer of about 50µm thick at the interface between the materials.

In the FSW materials there were two types of interface observed. In regions within the FSZ, where mixing and deformation were at their greatest, the copper/silver materials exhibited a microstructure of silver with sub-micron regions of copper, with the copper content increased from 7.25 wt.% to ~30-35 wt.%. This was formed by a combination of high-temperature and high-shear stresses in the friction stir zone, resulting in a thermally assisted, mechanically alloyed structure.

Just outside the FSZ the copper and silver were in intimate contact but with no evidence of significant diffusion of Cu into Ag. In these regions the bonding resembled that observed in the solid-state bonded mokume gane materials, i.e., metal-to-metal bonding at the interface.

In copper/brass FSW materials, while the intermixing of the copper and brass in the FSZ was extensive, there was less evidence for thermally assisted mechanical alloying of the two materials on a sub-micron scale, with the materials mixing but not interacting strongly with each other.

The difference in behavior of the copper/silver and copper/brass pairs is influenced by the difference in melting points, the difference in solubility of one phase in the other, and the difference in thermal conductivity. The solubility of copper in silver at elevated temperature is high and this occurs in the liquid-phase, conventionally made mokume gane. However, in copper/silver FSW mokume gane the structure in the mixed regions consists of an elevated level of copper (up to 35 wt.%) but this has presumably been formed without the silver phase becoming liquid.

The integrity of the bond between the two metals has been shown to be very good within the FSZ. Heavy deformation of the laminate structure was unable to separate the materials within these regions. However, outside of the FSZ, where the material is in intimate contact but no diffusion or mechanical alloying has taken place, deformation resulted in de-bonding of the two materials.

5. Conclusions

- Given the above results it is clear that it would be feasible to use FSW to produce mokume gane materials from a number of different metals including gold, silver, platinum, palladium, copper and brass.
- FSW, used in the correct manner, can produce well-bonded materials in laminate form in which the materials have been both bonded and mixed, forming an attractive and repeating pattern that can be reproduced.
- The fact that mixing occurs at the same time as the bonding provides a reduction in the amount of subsequent work required to form the patterns that give mokume gane its appeal.
- The method does not require high-temperature furnaces or the need to avoid oxidation of the metals, and very little cleaning or sample preparation is required. It is also a relatively low-energy process since the whole sample does not require heating.

- The patterns formed are unique to FSW and have the potential to be widely varied by changing the lay-up of the materials to be bonded and the friction stir conditions. After reaching steady-state conditions the patterns formed are stable and repeat in a regular manner but with a small natural and random variation, making each piece unique.
- The bonds formed in the friction stir zone are a combination of the intimate physical contact between the metals resulting in metal-to-metal bonds plus the thermally enhanced mechanical alloying, producing microstructures not possible by conventional processing.
- The nature of FSW means that large ingots of bonded material can be produced relatively easily using multiple passes of the friction stir tool through laminate layers. Other possible material lay-ups are possible, giving many possible variations in pattern, for example, bars of materials side by side or materials with regions inset with a second material.
- On the smaller machines, such as the TTI, the maximum ingot size is ~ 300mm x 150mm, with thickness determined by the number and thickness of the layers to be bonded but in the region of 10mm to 30mm. Such large sizes are impossible using any other mokume gane production technique. In theory the larger PowerStir machine could make much larger sheets measured in meters.
- With the constant development of FSW and the increasing availability of machines and tooling capable of carrying out FSW, there is a great potential for both large companies and smaller individual makers to begin to experiment and develop new mokume gane materials.

Acknowledgements

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