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Study of the Effect of RF-power and Process Pressure on the Morphology of Copper and Titanium Sputtered by ICIS

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Abstract

Inductively Coupled Impulse Sputtering is a promising new technique for highly ionised sputter deposition of materials. It combines pulsed RF-power ICP technology to generate plasma with pulsed high voltage DC bias on the cathode to eliminate the need for a magnetron.

To understand the effect of power and pressure on the coating morphology, Copper and Titanium films have been deposited in a power-pressure matrix. The RF-power was increased from 2000 - 4000 W. The pressure was set to 6 Pa and 13 Pa respectively.

For Copper, the morphology changes from columnar to fully dense with increasing power and the deposition rate drops from 360 nmh^{-1} to 210 nmh^{-1} with higher process pressure.

Titanium morphology does not change with power or pressure. The deposition rate is lower than predicted by the differences in sputtering yields at 68 nmh^{-1} for a pressure of 6 Pa.

Keywords: ICIS, HIPIMS, IPVD, Pulsed Plasma, Magnetron Free Sputtering

1. Introduction

High Power Impulse Magnetron Sputtering (HIPIMS) processes, have recently been gaining increasing interest from the industry, due to their ability to enhance coating properties such as defect free films with increased film density [1]. Initially HIPIMS has been used in traditional magnetron processes [1 - 4], as an alternative to arc processes for hard coatings. However, due to the enhanced coating properties it is now deployed on rotating cylindrical cathodes [5,6] and in increasingly specialised applications such as deposition on polymer [7,8] substrates. The substrate temperature can be reduced compared to DC sputtering, due to the higher ion-to-neutral ratio.

To enable the highly ionised sputtering of thin films, we have developed the new inductively coupled impulse sputtering (ICIS) technique which combines generating an ICP plasma in a pulsed RF-coil with high voltage pulsed DC bias on the magnet free cathode [9 - 11]. This technology allows for highly ionised sputtering of ferromagnetic materials, as the plasma is generated in front of the cathode surface. The effect of process pressure and substrate temperature on the coating structure has been investigated by the development of structure zone diagrams, such as the Thornton model [12]. Here it is shown, that with increasing process pressure and substrate temperature the structure of the deposited film can be modified. This is caused by the change in the mean free path distance [13]. Fewer collisions allow for the impeding sputter flux to have a higher energy, thus increasing surface mobility.

Increasing the power applied to the magnetron also affects the morphology of the coating, as more gas can be ionised, increasing the collisions and thus the ionisation of the sputter flux increases, increasing the adatom mobility [14].

As will be shown in section 3, preliminary results have suggested that ICIS achieves greater ionisation degrees compared to magnetron based technology and the effect of this on the morphology and deposition rate under highly ionised conditions is unknown.

As only few publications on ICIS-deposition of thin films are available [9 - 11], the objective of this study was to deposit Copper and Titanium in a defined power-pressure matrix and use SEM analysis to gain an understanding of the effect that external parameters have on the coating morphology of various materials. Copper and Titanium were chosen due to the wide range of studies that have been conducted on their film properties and modelling of plasma properties [15 - 20], making them ideal to characterise the influence of a new plasma source on the coating.

2. Experimental

Copper and Titanium coatings were deposited in a Kurt J. Lesker CMS - 18 UHV chamber equipped with ICIS technology (fig. 1). The ICIS system has been described in detail elsewhere [11, 12].

75 mm diameter sputtering targets of 3N Copper and 3N Titanium were used.

A constant DC voltage of 1900 V was applied to the cathode to attract Ar ions and initiate sputtering. RF-power was varied in 1000 W steps from 2000 W to 4000 W, resulting in a maximum average power of 533 W. Constant pulse parameters of 500 Hz repetition frequency with a pulse width of 150 µs were applied simultaneously to both DC and RF-power supplies, equivalent to a duty cycle of 7.5 %.

Argon was used as process gas and the system was set at working pressures of 6 Pa and 13 Pa.

The base pressure of the vacuum system was in the region of 4×10^{-4} Pa.

To get an understanding of the influence of increasing RF-power at a constant process pressure and constant power with varied pressure, a power-pressure matrix was devised to deposit the coatings and examine their influence on the morphology.

For each material, three coatings were deposited at different RF-power settings respectively, and a further coating was deposited at the highest power and reduced pressure.

The substrates are silicon (001) wafers with viae and are positioned on the substrate holder. Viae have aspect ratios (AR) between 5:1 and 2:1. The bias of the substrate was kept at floating potential. No substrate heating was applied.

The coated samples are fractured along the die to analyse the cross section in an FEI Nova NanoSEM 200 SEM.

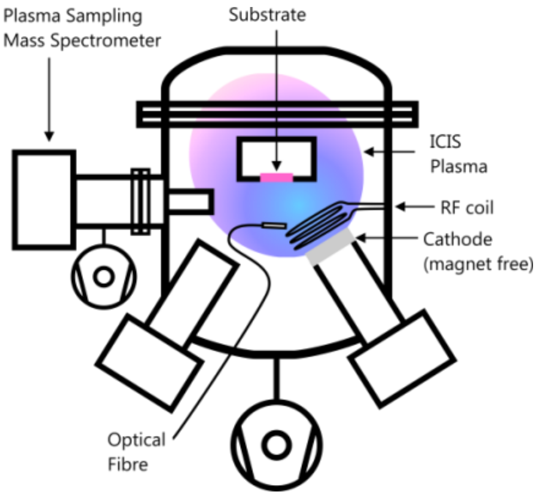

Figure 1: Experimental setup for ICIS plasma measurements inside the UHV system.

Optical emission spectra were acquired by OES monochromator (Jobin Yvon Triax 320, HORIBA Synapse CCD detector) with *in vacuo* quartz optical fibre and collimator installed as shown in fig. 1.

Plasma sampling mass spectrometry measurements were taken in time resolved mode in 150 µs steps over the whole pulse duration of 6.6 ms by a Hiden Analytical PSM 003 mass spectrometer. For these measurements we used the Ar^{1+} ion (40 amu) and Ni^{1+} ion (58 amu).

3. Results

A preliminary plasma study by optical emission spectroscopy has shown that for Ti sputtered by ICIS, the ionisation of the plasma is higher compared to magnetron based processes. The ICIS process was operated at a pressure of 13 Pa. Average power was set to 570 W on the RF-coil, at a duty cycle of 25 %.

In fig. 2 the three lower graphs show the comparison of emission spectra for magnetron sputtering, RF-coil enhanced magnetron sputtering and HIPIMS conducted by Ehiasarian *et al.* [21]. The top graph shows the newly added optical emission spectra for ICIS of Ti.

It can be seen that the intensity of the neutral Ti lines is significantly higher for magnetron sputtering compared to the other techniques, while the ionised Ti lines are not identifiable. While there is some increased ionisation in RF-coil enhanced magnetron sputtering, the ion to neutral ratio is greatly increased for HIPIMS and ICIS.

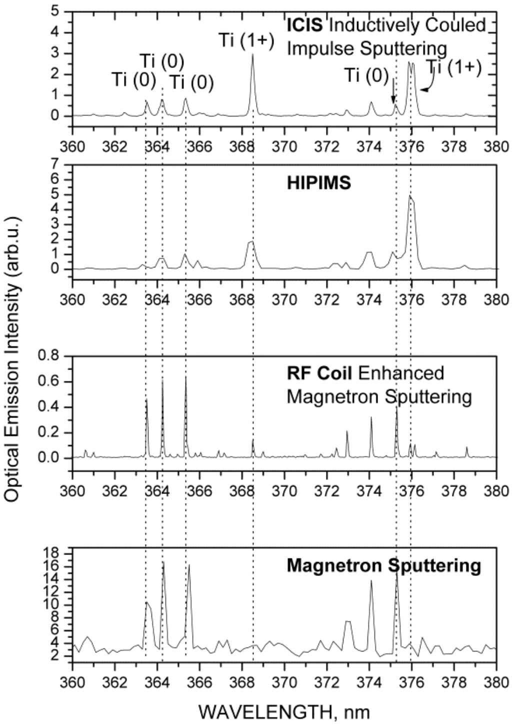

Figure 2: Comparison of optical emission spectra of Titanium deposited by magnet sputtering (bottom), RF-coil enhanced magnetron sputtering (second from bottom) and HIPIMS (second from top) as shown by Ehiasarian *et al.* [21]. Top graph ICIS emission spectra showing increased ionisation, even exceeding HIPIMS ionisation.

On the basis of these results we have devised the experiments with Cu and Ti as target materials to investigate the effect of power applied to the RF-coil and process pressure on the coating morphology, in the absence of a permanent magnetic field.

3.1 Copper Coatings by ICIS

Copper was deposited on the Si substrates for 120 min to achieve an estimated film thickness of 200 nm.

For a power of 2000 W and pressure of 13 Pa (fig. 3a and 3b), the deposition rate is 120 nmh^{-1}. The morphology is dense columnar with perpendicular growth on the top surface. At the edge of a via, the growth is at an oblique angle with increased tilting towards perpendicular at the sidewall. The protuberant material has a hemispherical shape over the thickness of the coating. The sidewalls and bottom of the via are coated evenly with increasing attenuation as the depth increases for high AR. Viae with an AR of 3:1 or smaller remain open and the sidewalls and bottom are coated evenly.

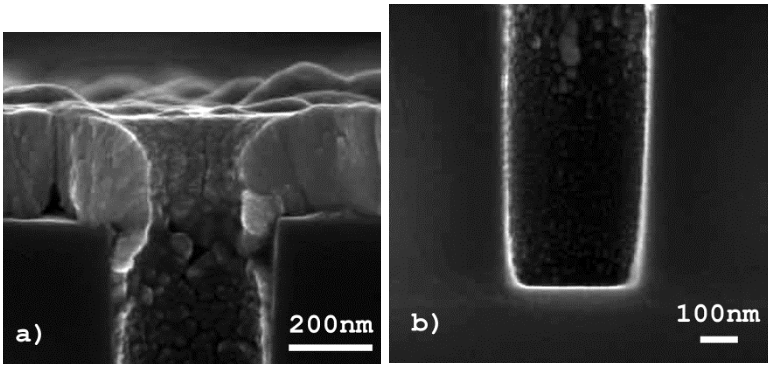

Figure 3: a) Cross-section of the top of a via with an AR of 3:1, coated with Copper by 2000 W ICIS at a pressure of 13 Pa. **b)** Cross-section of the bottom of the via in fig. 3a coated with Copper on the sidewalls and bottom.

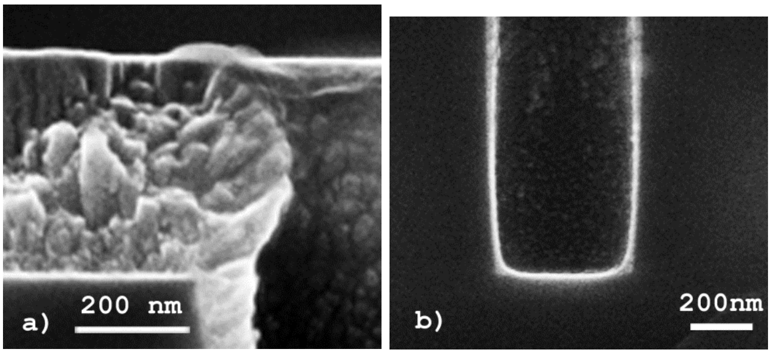

Figure 4: a) Cross-section of a corner of a via coated by 3000 W Cu ICIS at a pressure of 13 Pa. **b)** Cross-section the bottom of a via coated by 3000 W Cu ICIS at a pressure of 13 Pa. Bright areas are Cu coating, dark areas are SiO_2 substrate.

For an RF-power of 3000 W the deposition rate increases to 200 nmh^{-1} (fig. 4a and 4b) at a pressure of 13 Pa. The morphology is dense columnar with a higher densification of the columns. The protruding material at the edge of the via with an AR of 2:1 has a hemisphere shape over the whole top coating thickness with increased sidewall coating thickness (fig. 4a). Viae with a ratio of 3:1 fully close after approx. 80 min (not shown). The rate of via closure increases with coating thickness. While the via is open, the coverage of side walls and bottom is even, attenuating towards the bottom of the via (fig. 4b).

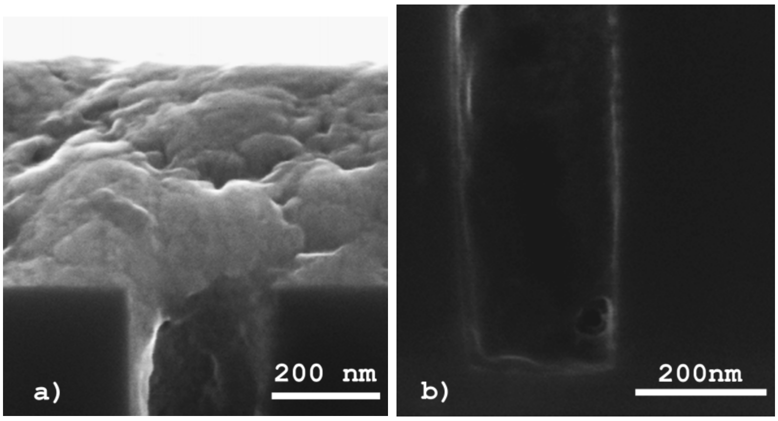

Figure 5: a) Cross-section of a corner of a via coated by 4000 W ICIS at a pressure of 13 Pa. b) Cross-section of the bottom of the via to indicate coating of sidewall and bottom of a **3**:**1** ratio via.

In figure 5 a pronounced change to the morphology is recognisable for an RF-power setting of 4000 W. There are no distinguishable grain boundaries identifiable in the dense morphology. The deposition rate rose marginally to 210 nmh^{-1}.

Coating growth was at an oblique angle from the top edge of the via until the opening was fully covered. For an AR of 3:1, the via opening was covered after approx. 40 min. The sidewalls and bottom were coated evenly while the via was open and the coating thickness is in the range of 5-10 % of the top cover.

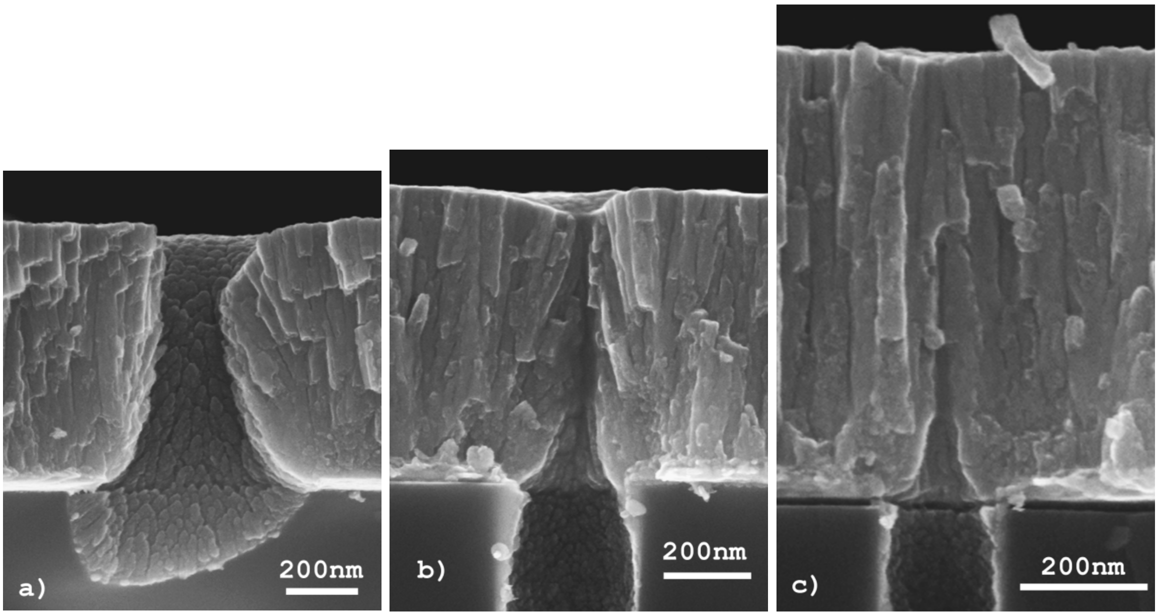

Figure 6: Cu coating deposited at a power of 4000 W on the ICIS coil, at a pressure of 6 Pa. The deposition rate is 360 nmh^{-1}. **a)** Top coating with the via remaining open for of a 2:1 ratio

via. **b)** For a ratio of 3:1, the via is closed after approx. half the deposition time, with an indent above the via visible in the coating surface. **c)** Via with a ratio of 5:1 are closed after approx. 30 min. The coating has no visible defect on the surface.

Due to the fracturing of the sample, unfortunately, the whole via is not visible for this process. We can however show the change in the duration the via is open in relation to the AR.

When the pressure is reduced to 6 Pa at an RF-power of 4000 W, the morphology changes to dense columnar (fig. 6 a-c), and the deposition rate increases to 360 nmh^{-1}. The via remains open for the whole duration of the process for a ratio of 2:1 (fig. 6a). For a via with an AR of 3:1, the via remains open for the whole duration, however, the opening narrows significantly after 60 min of deposition (fig. 6b).The opening of the via is closed fully after 30 min for an AR of 5:1 (fig. 6c). From the edge of the via the coating growth is at an oblique angle. After the columns from the edge meet and close the via, the columns then grow vertically as from the substrate top surface.

Analysis of the coating by SEM has revealed a number of promising features for the morphology, as well as for the coating of high AR features on a substrate. The morphology exhibits increased densification of the coatings for increasing applied RF-power from dense columnar to the very dense feature reduced coating as in fig.5. This change can be attributed to the higher ionisation of the metal flux and the resulting increased adatom mobility. The morphology is comparable to zone 2 for the 2000 W and 3000 W coatings; the 4000 W sample is in between zones 2 and 3 compared to the Thornton model. Perpendicular film growth over the whole length of the sidewalls in the via shows that the majority of the deposition flux is ionised, as only ions can follow the electric field lines and act perpendicular to the surface. The bottom and sharp edges to the sidewalls of the via are also covered uniformly suggesting resputtering inside the feature. Material build up at the edge of the via is not present for ratios larger than 2:1. Increased thickening of material protruding into the via appears with increasing film thickness, attenuating towards the bottom of the via. As been described by Rossnagel [22], the edge rounding at the top corner of the coating is caused by highly energetic ions resputtering any material build up. For low deposition rates, this effect helps to keep the via open for the process time. The deposition rate for 2000 W applied RF-power is 120 nmh^{-1} increasing to 200 nmh^{-1}

for 3000 W, due to increased ionisation of the process gas increasing the sputter rate. Film growth rate at 4000 W only increases marginally to 210 nmh^{-1}, yet the density of the film increases substantially. This may be caused by gas rarefaction reducing the sputter rate and, as the plasma source is above the target surface, allowing for the majority of the added power to increase the ionisation of the flux of sputtered material. When reducing the pressure to 6 Pa, the deposition rate increases to 360 nmh^{-1} and the coating structure is dense columnar. The change in morphology may be an effect of the increased mean free path leading to a reduction in collisions and thus increased ionisation fraction. The higher energetic ion fraction leads to more adatom mobility allowing the densification of the film. The higher deposition rate can also be attributed to the increased mean free path, as the sputter flux is less disturbed by collisions leading to more material arriving at the substrate.

3.2 Titanium Coatings by ICIS

From preliminary experiments it was calculated that to achieve a coating of 200 nm thickness, the process time needs to be 300 min. Apart from the deposition time, the process parameters were identical with the previous Cu coatings to be able to additionally compare how the same settings affect a different material.

The first coating deposited at 2000 W exhibits preferential deposition in large globular grains (fig. 7), creating a rough surface with dense coating in between. The deposition rate was 21 nmh^{-1} measured in the preferred growth regions. Film growth on the sidewalls is perpendicular and the coating thickness reduces until the half-way point for an AR of 3:1. For the lower half of the via, the coating has a uniform thickness over the sidewalls and bottom. For an RF-power of 3000 W the structure is dense globular. As can be seen in fig. 8, the sidewall and bottom coverage is continuous and there is no deposited material build-up protruding into the via for an AR of 1.6:1. There are some large material build-ups over the surface of the sample. The deposition rate was 14 nmh^{-1}.

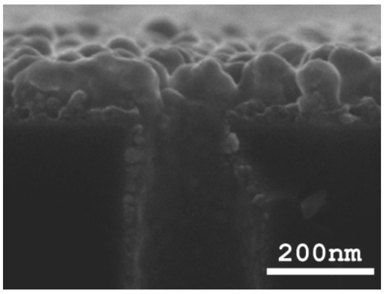

Figure 7: Cross-section of a 5:1 AR ratio via coated with Titanium by 2000 W ICIS at a pressure of 13 Pa.

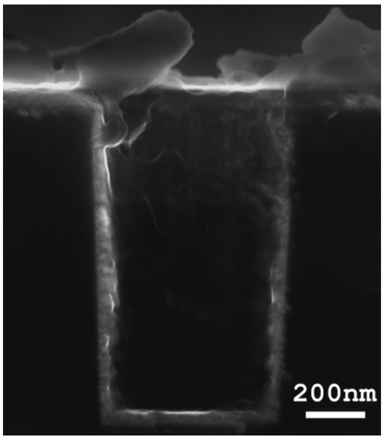

Figure 8: Cross-section of a corner of a 2:1 AR ratio via coated with Titanium by 3000 W ICIS at a pressure of 13 Pa.

For a pressure of 13 Pa and an RF-power of 4000 W, the Ti coating is deposited at a rate of 24 nmh^{-1} (fig. 9a and 9b). The morphology is dense with no distinct grain boundaries and a very rough surface caused by large preferentially deposited globules, which do not protrude over the top of the coating significantly. The coverage of the sidewalls is even and there is no further protruding of material at the top of the via.

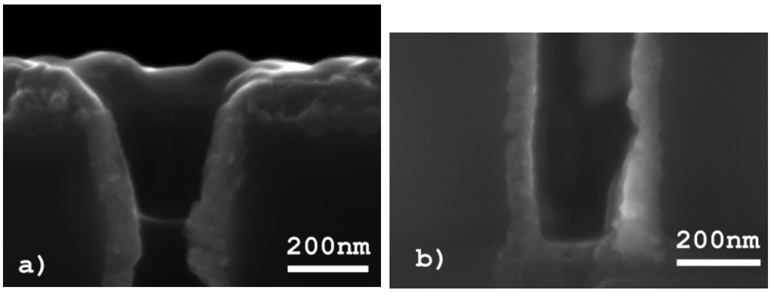

Figure 9: a) Cross-section of the top of a via coated by 4000 W ICIS at a pressure of 13 Pa. The fracture plane is off centre causing uneven edges between the coating and the

substrate. **b)** Cross-section of the bottom of the via coated by 4000 W ICIS at a pressure of 13 Pa. The fracture plane is off centre causing uneven edges between the coating and the substrate.

As can be seen in fig. 10, for the reduced pressure of 6 Pa, the morphology is dense globular. Similarly to the 2000 W deposited sample, the surface is very rough due to areas of preferential growth (fig. 10a). The sidewall and bottom coverage (fig. 10b) is uniform, with some directional growth at an oblique angle. As can be expected, the deposition rate increases approx. threefold to 68 nmh^{-1} compared to the 4000 W power setting. This is due to fewer collisions as the mean free path is extended.

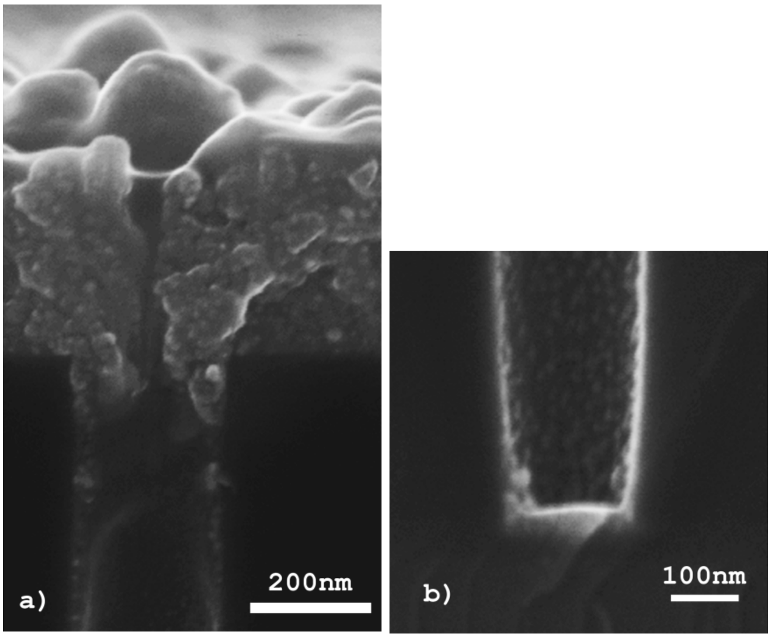

Figure 10: Titanium coating on a 5:1 AR via deposited by 4000 W ICIS at a pressure of 6 Pa **a)** Cross-section of the top of a via coated. Dense globular coating morphology. A narrow channel in the coating remains open over the via. **b)** Bottom of a via. Coating growth is visible on sidewalls and bottom.

There are no changes in the morphology or densification by changing power or pressure. Deposition rates are lower than expected compared to results from preliminary experiments. For increasing applied RF-power, the deposition rates are in the range of 20 nmh^{-1} at a constant pressure of 13 Pa, increasing to 68 nmh^{-1} at the lower pressure of 6 Pa. It was observed that the RF-coil is coated by target material during the process. As the preliminary experiments were conducted using a coil that had been used for several hours of Ti ICIS processes, it is possible that initially more material is deposited until the coil is fully coated by target material. Once a certain level of coating is achieved, the net deposition may be counteracted by resputtering from the coil and as such increasing the deposition rate on the substrate. The rate increase at low pressure can be related to the reduced mean free path allowing particles to travel further before colliding. The coatings all feature dense globular structure which corresponds to zone 2 in the Thornton model. Viae show uniform coating deposition on side walls attenuating with increasing depth as well as uniform coating at the bottom of the via, with no build up at the edges caused by resputtering of material. The uneven coating surface for Ti could be a result of highly textured (002) microstructure forming at angled deposition as has been demonstrated by Alvarez *et.al.* [23].

Further experiments need to be conducted to investigate the cause of the, in comparison with the copper and the respective sputter yields, lower deposition rate for titanium.

3.3. Plasma diagnostic analysis

The ion flux produced by an ICIS plasma was analysed quantitatively to determine the growth condition. ICIS of Nickel was used as a model system, where the time- and energy- resolved mass spectroscopy analysis (fig. 11a and 11b) reveals that during the pulse on-time (detected in the first 1 ms), growth starts with an intense highly energetic flux of gas ions. The flux of metal ions is relatively small as sputtering and ionisation processes build up sufficient vapour.

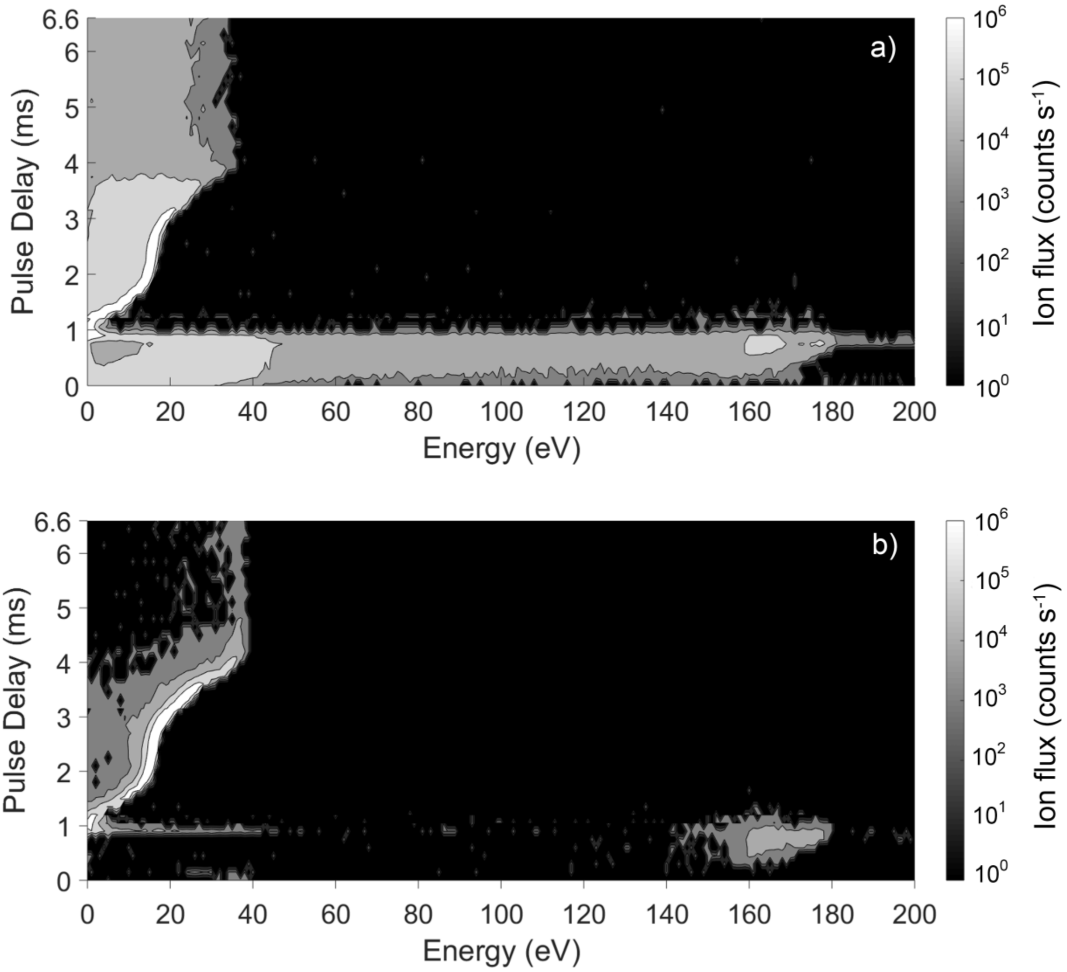

Figure 11: a) Time-resolved measurement of 4000W ICIS ionised Argon (40amu) at a pressure of 13Pa. **b)** Time-resolved measurement of 4000W ICIS ionised Nickel (58amu) at a pressure of 13Pa.

After the end of the pulse, detected with a 1 ms delay, there is a gradual increase in metal ion flux until the equilibration of the two fluxes. The energy of the ions drops to < 3 eV and for the remainder of the time gradually increases to around 40 eV. The highest intensity ion flux is observed at an energy of ~20 eV. It has been shown [25] that the energy follows the evolution of RF voltage applied in the RF-coil.

4. Discussion

4.1 Deposition rates

Copper coatings exhibit a highly dense columnar morphology for power settings of up to 3000 W at a pressure of 13 Pa. For a power of 4000 W, the morphology turns to fully dense with no distinguishable grain boundaries. Deposition rates of up to 210 nmh^{-1} are achieved for these settings. For a power of 4000 W and a pressure of

6 Pa the morphology is dense columnar and the deposition rate increases to 360 nmh^{-1}.

For Titanium the deposition rate is very low (20 nmh^{-1}) for all power settings at a pressure setting of 13 Pa. The lower deposition rate of Ti in comparison to Cu cannot be linked directly to the different sputter yields. According to the NPL Ar sputter yield table [14], the sputter yield of Cu is 2.6 times higher than Ti. However, the actual deposition rate is 9 times lower than Cu. This may be attributed to a net deposition of target material on the RF-coil in the Ti process, while additional sputtering of coil material in the Cu process may take place.

Comparing the nickel deposition rate from our previous work [6] has shown that the deposition rate for Ni is twice the rate for Ti in this current work. This corresponds very well with the sputter yield table [14], where the yield for Ni is twice that of Ti. This shows that the net deposition of target material on the coil is not dependent on the target material, if different from the coil material. Also the deposition rate is equal for non-magnetic and ferromagnetic materials.

At a pressure of 6 Pa, the deposition rate rises to 68 nmh^{-1}. For the lower pressure the difference in deposition rate of Ti is half of the rate expected by the sputter yield.

The morphology of the Ti coatings is dense globular, with a very uneven surface on the top side of the substrate, for all examined cases.

The presence of globular grains for both Cu and Ti films is an indication of renucleation.

4.2 Film Growth

The coverage and film growth is governed by the temporal evolution of the deposition flux.

The low overhang seen in Ti and Cu deposition cases may be explained by the high ion energies observed in the beginning of the pulse. The energies are produced by acceleration fields set up across the substrate sheath, i.e. between the plasma potential and the floating potential of the substrate surface. This indicates that ions are accelerated close to the substrate surface and, guided by the local electric field, arrive at the surface at a normal inclination. It also means that the flux is more concentrated at the sharp edges or corners where the field vector becomes inclined and effectively focuses the flux towards the edge. This reduces the ingress of overhang material.

The uniform coverage of the via's side walls may be attributed to resputtering from the bottom of the vias and re-deposition onto the sidewalls. This process is enabled by the high ion energies which are produced in ICIS in the first phase of the pulse, analogous to conventional ICP sources operating in continuous mode [24].

During the second phase of the pulse, the ion flux is rich in metal ions and this is where the nucleation and growth is set to occur. The high ionisation degree means that material is guided down to the bottom of the via by the electrostatic potential within the sheath thus enhancing deposition rate at the bottom of the via. Due to the separation in time between the metal and gas ion flux the renucleation probablity is quite high with phase one creating potential renucleation sites which are occupied during phase 2.

The second phase provides better conditions for growth through lowering the energy of ions to ~ 20 eV which changes the balance from resputtering to deposition. The lower ion energy promotes surface diffusion of arriving metal species and tends to produce dense morphology by annihilating some of the vacancies and grain (intercolumnar) boundaries. This results in larger grains.

The result of the competition between resputtering during the first phase and dense film growth during the second is a dense overall structure with large grains. The grains however are unlikely to form columns but are rather equiaxed in nature.

The relatively high deposition pressures achieved through low pumping speeds also mean that there is a heightened degree of contaminant gases. The inclusion of foreign phases such as oxygen and hydroxide groups in the structure restricts diffusion near the grain boundaries and promotes faster growth in the centre of the column. This predisposes the system to a dome-shaped growth front and formation of intercolumnar voids. The globular grain morphology observed in the current experiments may be attributed in part to this effect. Grain boundaries remain dense as the high mobility of deposition flux and re-nucleation processes push the deposited adatoms to overcome grain boundaries and results in the embedding of the foreign phase into the growing film.

5. Conclusion

In this work we have presented the effect ICIS has on the morphology of non-magnetic materials. A significant find is that when sputtering Cu the deposition rate normalised by the sputter yield is disproportionately large compared to Ti and Ni. For both Cu and Ti we have observed even coating of vias with an aspect ratio of up to 4:1, with perpendicular column growth on sidewalls and continuous coverage of the bottom of the via. This paper has shown that ICIS produces good coverage into high aspect ratio features due to high degree of ionisation and increased energy of the deposition flux. The highly energetic growth conditions produce a globular grain morphology with a high density of intergranular boundaries. The deposition likely proceeds through an initial stage of resputtering by highly energetic ions followed by a stage of deposition by mobile adatoms to produce dense films with excellent via coverage.

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