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A novel sputtering technique: Inductively Coupled Impulse Sputtering (ICIS)

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Abstract. Sputtering magnetic materials with magnetron based systems has the disadvantage of field quenching and variation of alloy composition with target erosion. The advantage of eliminating magnetic fields in the chamber is that this enables sputtered particles to move along the electric field more uniformly. Inductively coupled impulse sputtering (ICIS) is a form of high power impulse magnetron sputtering (HIPIMS) without a magnetic field where a high density plasma is produced by a high power radio frequency (RF) coil in order to sputter the target and ionise the metal vapour. In this emerging technology, the effects of power and pressure on the ionisation and deposition process are not known. The setup comprises of a 13.56 MHz pulsed RF coil pulsed with a duty cycle of 25 % . A pulsed DC voltage of 1900 V was applied to the cathode to attract Argon ions and initiate sputtering. Optical emission spectra (OES) for Cu and Ti neutrals and ions at constant pressure show a linear intensity increase for peak RF powers of 500 W - 3400 W and a steep drop of intensity for a power of 4500 W. Argon neutrals show a linear increase for powers of 500 W - 2300 W and a saturation of intensity between 2300 W - 4500 W. The influence of pressure on the process was studied at a constant peak RF power of 2300 W. With increasing pressure the ionisation degree increased. The microstructure of the coatings shows globular growth at 2.95×10^{-2} mbar and large-grain columnar growth at 1.2×10^{-1} mbar. Bottom coverage of unbiased vias with a width of 0.360 µm and aspect ratio of 2.5:1 increased from 15 % to 20 % for this pressure range. The current work has shown that the concept of combining a RF powered coil with a magnet-free high voltage pulsed DC powered cathode is feasible and produces very stable plasma. The experiments have shown a significant influence of power and pressure on the plasma and coating microstructure.

Keywords: ICIS, Ionised PVD, Magnet-free sputtering, deposition on high aspect ratio vias

1. Introduction

During sputtering charged particles i.e. ions, follow the electric field lines that are created in the chamber by the electric potential difference between the plasma bulk and the substrate surface. [1] This means that sputtering with a highly ionised metal plasma makes it possible to deposit coatings with good bottom coverage within high aspect ratio structures [2 - 4].

Conventionally, to achieve the high electron density necessary to ionise the working gas, a magnetic field needs to be applied on the target surface. To ionise the metal vapour additionally high power is necessary. Especially for sputtering magnetic materials the field is shunted and limits the usable target thickness. Furthermore, this additional magnetic field has the disadvantage of disturbing the uniform flow along the electric field lines. While this disturbance is advantageous for the initial

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ionisation of the gas and metal vapour as the collision probability is greatly enhanced, during the movement to the substrate the electrons are forced on cycloidical paths along the magnetic field lines and are confined near the target thus trapping gas and metal ions. [1]

Yukimura and Ehiasarian [5] have designed a magnet free experimental apparatus that consists of 2 electrodes immersed in an 200 kHz RF plasma that is produced by an inductor.

To extend the work described, we examine the ionisation degree in a plasma generated with a RF frequency of 13.56 MHz and a coil positioned within a vacuum chamber. In the current work an radio frequency (RF) powered Copper (Cu) coil is assembled in front of the target (Cu or Ti) to sputter and ionise the metal vapour. The current flowing through the coil induces a magnetic field that confines electrons to the inside of the coil thus enhancing the collision probability[6]. To attract the argon (Ar) ions to the target for sputtering, a high voltage pulsed DC is applied to the target. In the current work the effect of RF power on ionisation is studied and modelled by optical emission spectroscopy (OES) and the bottom coverage of Cu in vias is studied.

2. Experimental Details

The experimental ICIS system (schematic in Fig.1) consists of a UHV chamber (Kurt J. Lesker), a Hüttinger PFG 5000 RF power supply (13.56 MHz), a Advanced Converters HIPIMS power supply, a 2-turn 80 mm diameter solid rod copper coil and a magnet free 75 mm diameter cathode.

The plasma discharge is created within the RF powered coil. When the plasma has ignited, pulsed DC power is applied to the cathode. RF and DC power pulses are synchronised by a pulse generator.
In the current work a pressure - RF-power matrix was used to examine the influences of working pressure and RF- power on the discharge while the pulsed DC parameters were kept constant. During the experiments the working pressure was varied from $2.14x10^{-1}$ - $2.96x10^{-2}$ mbar and the RF power was varied between 500 W - 4500 W the DC voltage remained constant at 1900 V. The repetition frequency was 500 Hz with a pulse width of 500 µs, which relates to a duty cycle of 25%. The substrate was silicon oxide (SiO_2), an insulator, with vias, the bias voltage was floating. Temperature on the substrate at the beginning of the process was between 20 - 28 ºC and during deposition the temperature rose by aprox. 5 ºC within one hour.

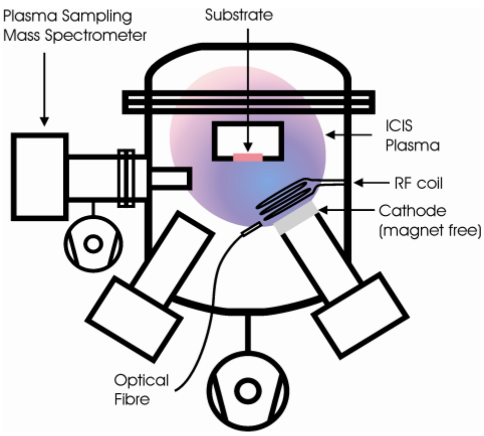

Figure 1. Experimental set up of the ICIS discharge with the assembly of the inductive coil, magnet free cathode and optical emission spectroscopy.

2.1. Plasma and coating characterisation techniques

Analysis was carried out by Optical Emission Spectroscopy (OES) (Jobin Ivon Triax 320) with quartz optical fibre and collimator *in vacuo*. To compare the ionisation performance of ICIS with the well understood conventional rf coil enhanced magnetron sputtering technique, the results of the OES measurements were fitted into a model based on magnetron sputtering based on electron ionisation collisions.[7]

Scanning Electron Microscopy (SEM) (FEI NovaSEM 200) was used to determine the bottom coverage, i.e. the ratio of the coating thickness on the bottom and top surfaces of vias.

2.2. Modelling

The used model correlates the optical emission of the plasma to the power on the cathode [7], in case of this work, it is the power applied to the induction coil. The model explains the connection between the intensity *(I(λij))* and power (P) for highly ionised magnetron plasma processes. It shows the increase in metal ionisation as a function of power. The intensity is expressed by a power law with exponent β. β is derived from the slope in a log — log graph.
The following subchapter will describe the basis of the model
The intensity of emission is expressed by the density of the components,

$$I(\lambda ij) = c(\lambda ij)\frac{Aij}{\sum_j Aij} \cdot n_0 \cdot n_e \cdot C^i \quad (1),$$

where $I(\lambda ij)$ is the intensity, $c(\lambda ij)$ is the spectral response of the spectrometer, Aij is the radiative frequency, n_0 is the gas neutral density, n_e is the electron density and C^i is the production rate by electron collision. The spectral response of the spectrometer *(c(λij))*, the radiative frequency *(Aij)* and the production rate by electron collisions can be assumed as constant *(Kij)* with respect to electron density.
For the excitation of Argon ($e + Ar \rightarrow Ar^*$) Eq.1 can be written as follows:
$I_{Ar} = K_{Ar} \cdot n_{Ar} \cdot n_e \cdot C^{Ar}$(2) As most parts of the equation are constant $I_{Ar} \propto n_e$. Further it is assumed that C^i is constant with increasing power so that $n_e \propto P^\beta$, where P is the power and β is the slope in a log-graph. This concludes that $I_{Ar} = P^\beta$ (3).
We determine β by taking the logarithm of both sides of the equation are logarithmised to get and obtain: $\log(I_{Ar}) = \beta \log P$ (4).

Excitation of Ti^0 ($e + Me \rightarrow Me^* + e$) is: $I_{Me} = K_{Me} \cdot n_{Me} \cdot n_e \cdot C^{Me}$ (5).
Because plasma is considered quasineutral, $n_{Ar^+} \approx n_e$ and from the definition of sputtering yield it is known that $n_{Me} = \varepsilon \gamma_e n_{Ar^+}$ (6). Where ε is a constant and γ_e is the sputtering coefficient, it can be concluded that $I_{Me} \propto {n_e}^2$. From this it follows that $I_{Me} = (P^\beta)^2$ (7) and $\log(I_{Ion}) = 2\beta \log P$ (8).

For the ionisation of Ti ($e + Me \rightarrow Me^+ + 2e$), the intensity is $I_{Ion} = K_{Ion} \cdot n_{Ion} \cdot n_e \cdot C^{Ion}$(9).
Where $n_{Ion} \approx n_{Me} \cdot n_e \approx {n_e}^2$. Thus $I_{Ion} \propto {n_e}^3$ and $I_{Ion} = (P^\beta)^3$ (10) which results in $\log(I_{Ion}) = 3\beta \log P$ (11).

3. Results and modelling

3.1. Current and voltage

The current and voltage behaviour on the cathode during a pulse can be seen in Fig. 2. The influence of the DC voltage on the current can be clearly seen, with the rise in current during the pulse. Hereby the target is biased to 1900V. Note that the voltage does not drop to zero as the current is too low to discharge the generator over the plasma.

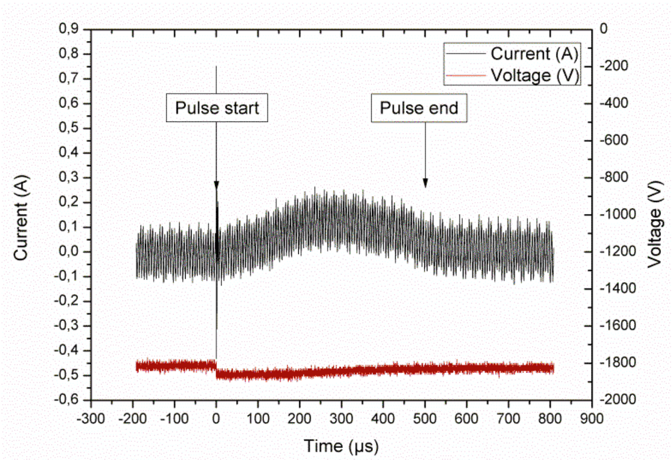

Figure 2 Current and voltage over time on the Cu-cathode during a pulse at 1.2x10-1 mbar. The target is biased to 1900V. The current induced by the impinging ions is upto 0.3A.

3.2. Optical Emission

The OES measurements in fig. 3a and 3b show the intensities of the individual species in the ICIS plasma. In both Ti and Cu cases, strong emission from single-charged metal ions is present. Ar and Cu neutrals are detected as well. Due to limited availability of spectral lines for metal ions and neutrals in the optical spectrum, lines that are visible at all powers were chosen. Emission lines were transitions to ground state with similar upper excitation levels that were generally lower than the Ar neutral excitation level of 15 eV. Spectra lines data was taken from NIST.[8]

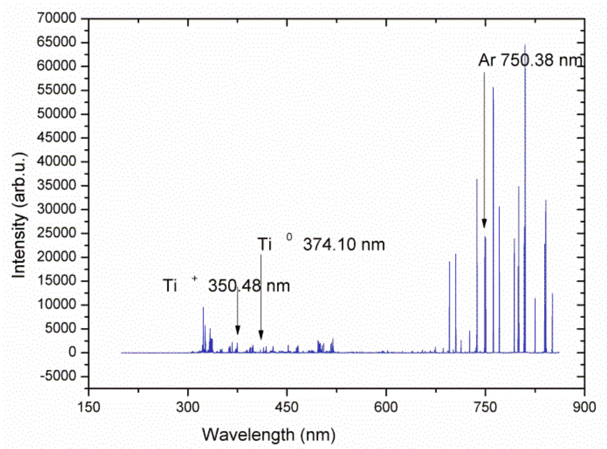

Figure 3a OES measurement of Ti ICIS plasmas at 1.2x10-1 mbar, 2300 W RF power. Lines used for analysis have been marked.

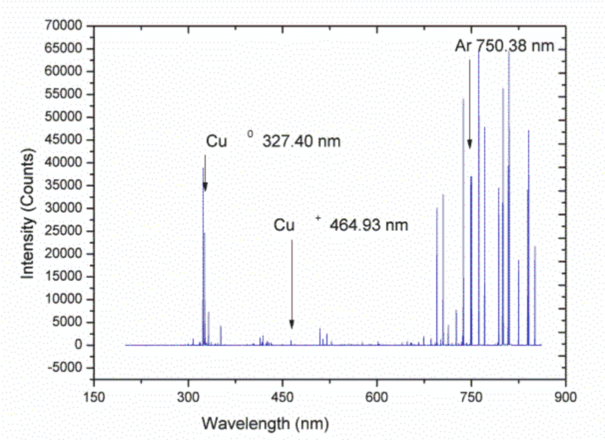

Figure 3b OES measurement of Cu and Ti ICIS plasmas at 1.2x10-1 mbar, 2300 W RF power. Lines used for analysis have been marked.

3.3. Modelling the influence of power on ionisation

Calculations from the model predict a linear rise of intensity with increasing power in a log-log graph. In the case of ICIS of Ti plasma, measurements show that the slope of Ar is 0.61±0.08. As the model does not differentiate between sputter elements this means that according to the model the slope of the Ti^0 should be 1.22±0.08 and the slope of Ti^+ should be 1.83±0.08. As the slopes of the metal species are based on the Ar slope, they all share same error margin.

Figures 4a-c clearly show that all measured values fit very well into the error margin of the slope of the model. The Ti neutrals and ions rise with increasing power, with a sharp drop for very high RF power. Ar intensity rises continuously with power.

The rate of rise of metal ions is faster than that of metal neutrals. Thus the ionisation degree increases with increasing power.

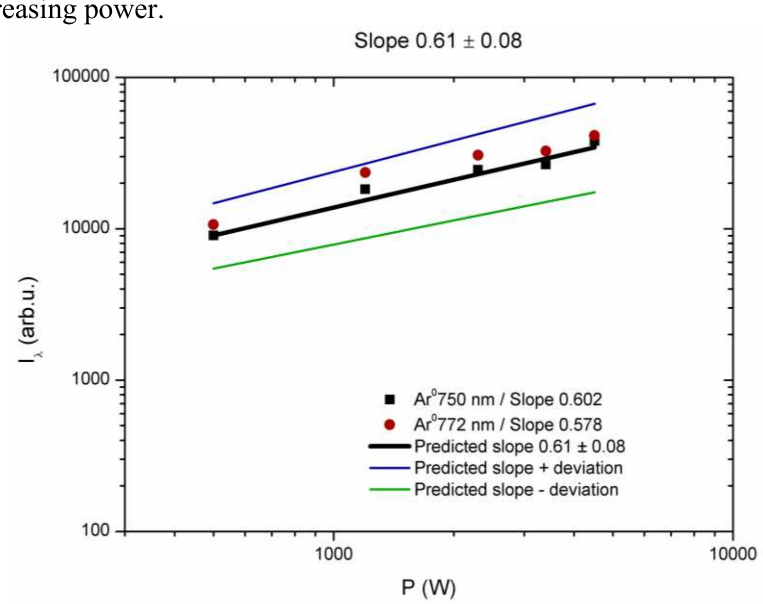

Figure 4a Measured and predicted results for the increase of ionisation with increasing power.

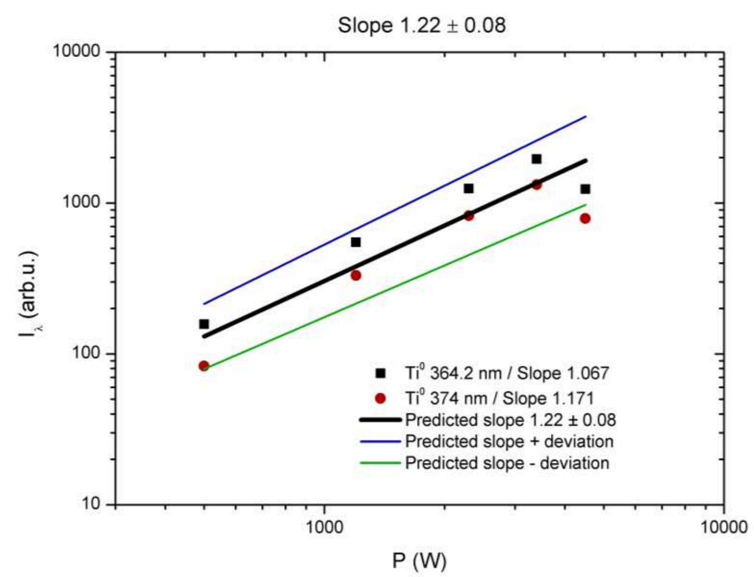

Figure 4b Measured and predicted results for the increase of ionisation with increasing power

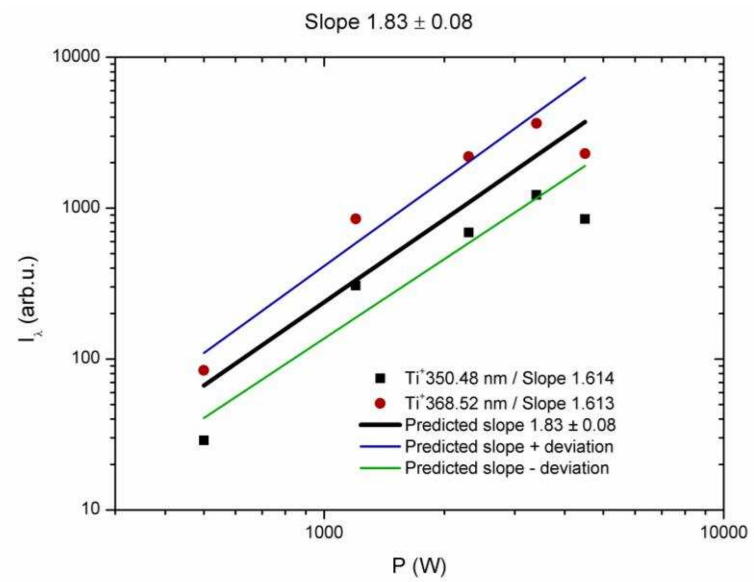

Figure 4c Measured and predicted results for the increase of ionisation with increasing power

Fig. 5 shows the increase in the ionisation degree as an ion to neutral ratio. For powers above 1000 W RF the rise is linear with increasing power. Below 1000 W the power is too low to generate Ti ions.

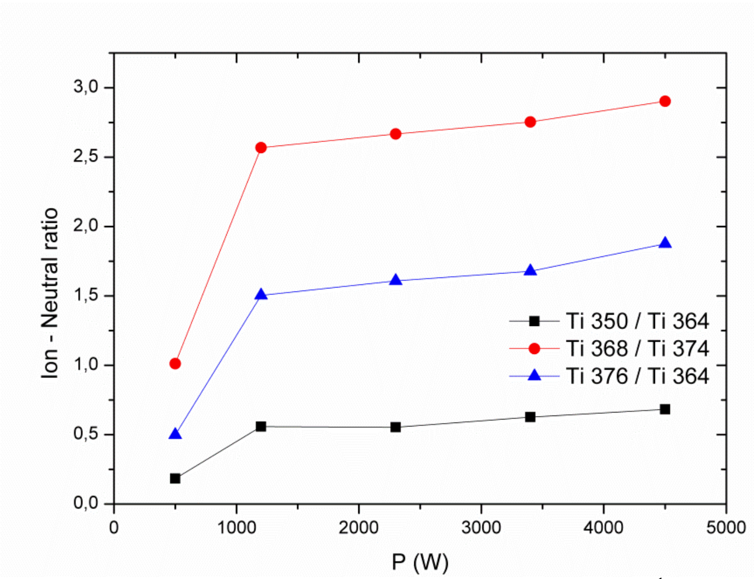

Figure 5 Ion to Neutral ratio for Ti ICIS plasma at $1.2x10^{-1}$ mbar

Fig.6 shows similar measurements for an ICIS of Cu plasma. When comparing the optical emission from ICIS of Ti and Cu plasmas it can be seen that upto a power of 2300 W both show similar behaviour for the metal species with a linear increase of ionisation as a function of RF power. For higher powers there is a saturation for the Cu metal species.
When sputtering Ti the intensity of Ar rises continuously with increasing power, while when sputtering Cu the intensity of Ar saturates for higher powers.

Table 1 lists the slopes as predicted by the model for Ti and the slopes of the measured Cu plasma species. Cu deviates from the model as the metal slopes are not clearly related to those of the Ar. Additionally, the slope for Ar is significantly greater when Cu is used. The reasons behind this need to be examined further.

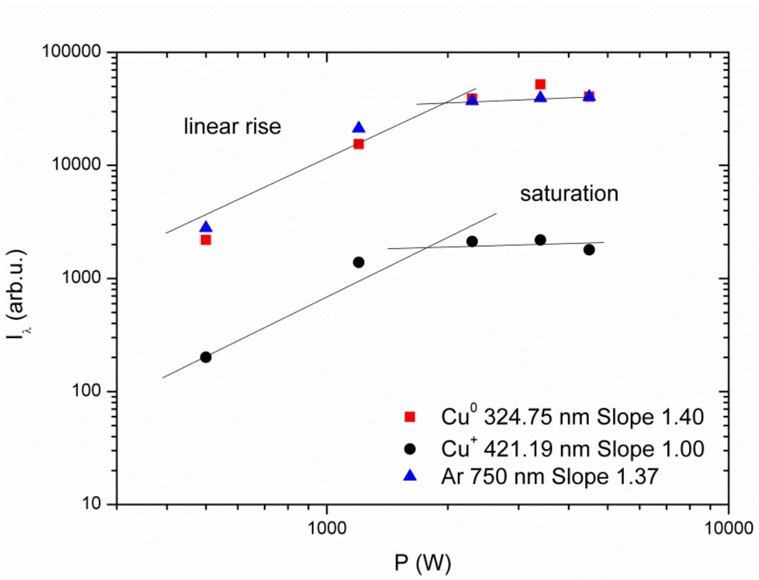

Figure 6 Ar and Cu neutral and Cu ion intensities for an ICIS Cu plasma. The rise in intensity is linear with power. Pressue $1.2x10^{-1}$ mbar.

Table 1 Comparison of the slopes of Ar and Cu plasmas. The rise in intensity is linear with power for Ti. Also the Model will need to be adapted to take into account the lower ioniastion potential of metal ions compared to argon.

Ar^0 slope:	0.61	Ar^0 slope:	1.37
Ti^0 slope:	1.22	Cu^0 slope:	1.41
Ti^+ slope:	1.83	Cu^+ slope:	1.76

To get better results for high density plasmas, the model will need to be adapted to take into account the lower ionisation potential of metal ions compared to argon. This can be seen in the results for Cu, where the measured results do not fit into the model prediction. Cu has a higher sputter rate compared to Ti and as such, probably the metal ion density is considerably higher.
Also due to the possibillity of successive excitations the model results overestimate the presence of metal ions.

3.4. SEM

The cross sectional (Fig. 6) SEM image of a high aspect ratio via coated with ICIS of Cu shows good overall coverage. The deposition process was carried out at the higher end of power and, respectively, metal ionisation degree. For this high pressure ($1.2*10^{-1}$ mbar) process the bottom coverage is 21.6 %. The deposition rate was ca. 80 nm/h at 2300 W.

Table 2 compares the bottom coverage between high and low pressure depositions. A general trend was that bottom coverage improved at higher pressures.
The microstructure was influenced strongly by the pressure as well. Dense columnar structure was observed at the higher pressure ($1.2*10^{-1}$ mbar) and dense globular growth at the lower pressure of $2.9*10^{-2}$ mbar. The deposition rate is 99-119 nmh^{-1} for RF-power of 2300 W, average target power of 67 W and a pressure of 1.2×10^{-1} mbar.

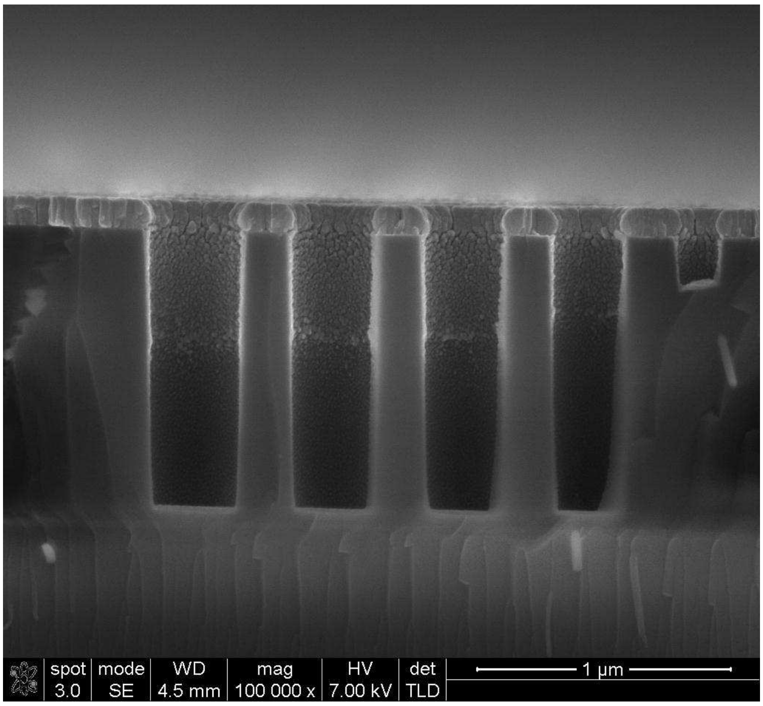

Figure 7 SEM cross section of ICIS Cu coated high aspect ratio vias at 2300 W, 1.2*10-1 mbar.

Table 2 Bottom coverage of vias obtained at high and low pressure depositions.

	Pressure:	$1.2 *10^{-1}$ mbar	
Height	Width	Top thickness	Bottom thickness
1,40 µm	315 nm	120 nm	18nm
Ratio:	4:1	Bottom coverage:	21,6 %
	Pressure:	$2.96 *10^{-2}$ mbar	
495 nm	143 nm	91.8 nm	15.3 nm
Ratio:	3.5:1	Bottom coverage:	14,0 %
240 nm	143 nm	91.8nm	15.3 nm
Ratio:	1.7:1	Bottom coverage:	14,0 %
163 nm	143 nm	91.8 nm	20.4 nm
Ratio:	1.2:1	Bottom coverage:	18.70%

4. Conclusion

Successful experiments were carried out by sputtering Cu and Ti that present ICIS as a feasible sputtering technique. Working without magnetic field eliminates the racetrack that is created with magnetron sputtering, thus enhancing target utilisation and allowing ions to follow the electric field lines without disturbance.

The measured results of the rate of increase for gas neutrals, metal neutrals and metal ions fit very well to the predicted values of the model for magnetron sputtering. Calculations from the model confirm the increase of the ionisation degree as a function of power for Ti. Further examination has to be done into the sharp drop in intensity for metal neutrals and ions for high RF

Power and adjustments to the model have to be made to take this and the lower ionisation potential, as well as successive exitation into account.These results show that the ionisation mechanisms for ICIS are the same, by type as well as quantity, as for RF-enhanced magnetron sputtering, but without the influence of the magnetic field of the magnetron.

Copper deposition on high aspect ratio vias has shown very promising results. The bottom coverage in vias with aspect ratio of 4:1 is 21.6 % with dense columnar growth and without bias.

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