Study of the ionisation in a nickel plasma by Inductively Coupled Impulse Sputtering (ICIS)

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Abstract

Inductively coupled impulse sputtering (ICIS) removes the need for a magnetron, while delivering equal or higher ion to neutral ratios compared to other ionised PVD technologies such as high power impulse magnetron sputtering (HIPIMS). This is especially advantageous for the sputtering of magnetic materials, as these would shunt the magnetic field of the magnetron, thus reducing the efficiency of the sputtering and ionisation process. ICIS produces highly ionised metal-dominated plasmas inside a high power pulsed RF coil with a magnet free high voltage pulsed DC powered cathode.

ICIS processing with Ti and Cu has been attempted before; however operation with magnetic target materials has not been attempted so far. The paper aims to clarify the effects of power and pressure on the deposition flux and structure of deposited Ni films.

The setup comprises of a 13.56 MHz pulsed RF coil operating at a frequency of 500 Hz and a pulse width of 150 µs, which results in a duty cycle of 7.5 % . A pulsed DC voltage of 1900 V was applied to the cathode to attract Argon ions and initiate sputtering.

Optical emission spectra (OES) for argon and nickel species sputtered at a constant pressure of 14 Pa, show a linear intensity increase for peak RF powers of 1000 W - 4800 W. Ni neutral line intensity increased linearly exhibiting two different slopes for powers below 2000 W and those above 2000 W RF - power.

The influence of pressure on the process was studied at a constant peak RF power of 3000 W for pressures of 3.2 – 26 Pa.

The intensity of nickel neutrals rises linearly for pressures of 3.2- 26 Pa and saturates for pressures from 12 – 21.4 Pa. Argon neutrals rise linearly with increasing pressure. Ni ions have not been visible in the OES spectra and analysis into the ion to neutral ratios will be conducted by other techniques.

From the Ti process we know, that the intensity of neutrals and ions increases linearly with power and pressure. Intensity modelling is also conducted for the Ni process. The deposition rate for Ni is 50 nmh^{-1} for a RF-power of 3000 W and a pressure of 14 Pa.

The microstructure of the Ni coatings shows columnar dendritic growth. Bottom coverage of unbiased vias with width 0.300 µm and aspect ratio of 3.3:1 was 15 % and for an aspect ratio of 1.5:1 was 47.5 %. Parameters for this coating are mean values from a power and pressure matrix. To investigate ionisation influence, coatings have also been deposited at higher power and pressure.

The current work has shown that the concept of combining a RF powered coil with a magnet-free pulsed DC powered cathode works very well for the sputtering of hard magnetic material in very stable plasma.

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

1 Introduction

Deposition of thin films of magnetic material is of great importance for various applications such as data recording [1] and magnetic microelectromechanical systems (MagMEMS) [2].

Magnetron-based sputtering techniques suffer from low target utilisation rates of 40% [3] and short service intervals for magnetic materials as targets need to be thin.

A high degree of ionisation of sputtered species is preferable, as this allows deposition on structured surfaces because ions follow the electric field lines that are created by the potential difference between the plasma bulk and substrate surface. This makes it possible to deposit even coatings on sidewalls and the bottom of high aspect ratio features of the substrate.[3]

Inductively Coupled Impulse Sputtering (ICIS) is a new development which aims to solve the previously mentioned issues by eliminating the need for a magnetron. ICIS is based on an experimental development by Yukimura and Ehiasarian [4], which utilises high pulsed RF-power in the coil and high pulsed DC voltage on the target to generate a plasma and attract argon (Ar) to initiate sputtering

In this work ICIS technology was adapted to work inside a deposition system and uses a 13.56 MHz RF-power supply.

In the current work the plasma composition and ionisation is studied by Optical Emission Spectroscopy (OES), the coating microstructure and thickness by Scanning Electron Microscopy (SEM) and the coating chemical composition and contamination by sputtered RF coil material by Energy - Dispersive X-ray spectroscopy (EDX).

A model [5] based on the optical emission of DC and RF magnetron discharges is used to explain the connection between the intensity (I(λij)) and power (P) for highly ionised magnetron plasma processes. Dony et.al. explain the dependence of the optical emission of the plasma to the power on the cathode. As with ICIS the plasma is generated inside the induction coil, in this study we correlate the optical emission of the plasma to the power applied to the coil. As the model is based on magnetron processes, it is very useful to compare the ionisation efficiency of ICIS with conventional magnetron processes.

2 Experimental Details

The experimental ICIS system (shown schematically in Fig.1) consists of a UHV chamber (Kurt J. Lesker CMS - 18), Hüttinger PFG 5000 RF power supply (13.56 MHz), a Hüttinger HIPIMS power supply HMP6/16, 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 study a pressure - RF-power matrix was used to examine the influences of working pressure and RF- power on the ionisation of the discharge and deposition properties. The pulsed DC parameters were kept constant at 1900 V. The working pressure was varied from 2.96 - 21.4 Pa and the RF power was varied between 1000 W - 4800 W. The repetition frequency was 500 Hz with a pulse width of 150 µs. The substrate was silicon dioxide (SiO_2), an insulator, with vias, held at floating bias voltage. Temperature on the substrate at the beginning of the process was between 20 - 28 ºC and during deposition rose by approx. 5 ºC within one hour.

2.1 Plasma and Coating Characterisation Techniques

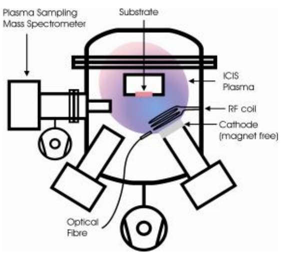

Figure 1 Experimental setup of the ICIS deposition system with the assembly of the inductive coil, magnet-free cathode and OES location.

Plasma composition analysis was carried out by OES monochromator (Jobin Ivon Triax 320) with quartz optical fibre and collimator *in vacuo*. The comparison of the excitation efficiency of ICIS with conventional RF-ICP magnetron sputtering is realised by fitting the OES results for increasing RF-Power to a model for ionisation by electron and Penning collisions developed for RF-coil enhanced magnetron sputtering. [5]

Scanning electron microscopy (SEM) (FEI NovaSEM 200) was used to examine the coating properties and to determine the structure and bottom coverage, i.e. the ratio of the coating thickness on the bottom and top surfaces of vias.

2.2 Modelling of the Excitation Properties

The results showed an increase in metal excitation and ionisation as a function of power. The relation between emission intensity and RF power in the coil is expressed by a power law with exponent β, which is derived from the slope in a log - log graph: $I_{Ar} = P^{\beta}$ (1).

As the excitation of Ar is predominantly by electron collision the intensity is proportional to the electron density, $I_{Ar} \propto n_e$ (2).

For the intensity of Ar neutrals and metastables we obtain the following equation:

$\log(I_{Ar}) = \beta \log P$(3), where I is the intensity of Ar for a certain wavelength, β is the slope and P is

the power applied to the RF-coil.
For the excitation of metal neutrals the intensity is $I_{Me} = K_{Me} \cdot n_{Me} \cdot n_e \cdot C^{Me}$(4). K_{Me} and C^{Me} are constants. From the definition of the sputtering yield, $n_{Me} = \varepsilon\gamma_e n_{Ar^+}$ (5) where ε is a constant and γ_e is the sputtering coefficient and because plasma is considered to be quasineutral, $n_{Ar^+} \approx n_e$(6), it can be concluded that $I_{Me} \propto n_e{}^2$ (7). Following from equations (1) and (2) for metal neutrals we get $I_{Me} = \left(P^{\beta}\right)^2$ (8). For Ni neutrals we get: $\log(I_{Ni^0}) = 2\beta \log P$ (9). As a further electron collision is necessary to ionise the excited metal atoms for Ni ions the equation is: $\log(I_{Ni^{1+}}) = 3\beta\ logP$ (10). This means that the slopes of Ni neutrals are expected to be twice as steep and three times steeper for Ni ions as those of Ar neutrals.

3 Results and Modelling

3.1 Optical Emission Measurement

OES results for a ICIS 3000 W Ni plasma in fig.3 show strong emission from Ni neutrals. Ni ions which were expected at 333.188 nm were below the detection limit of the OES. Most Ni ions can be expected at lower wavelengths very much further in the ultraviolet spectrum. Further experiments are intended to detect these.

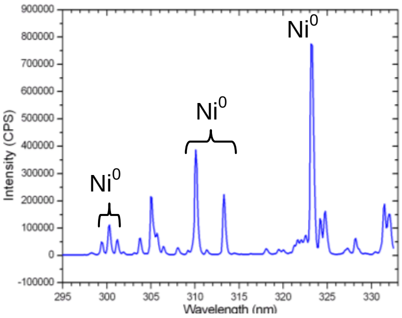

Figure 2 OES Measurement of a ICIS Ni Plasma at 3000 W RF-Power

3.2 Modelling the Influence of Power on Ionisation

As has been discussed, according to the prediction model the intensity of the sputtered species rises linearly with increasing RF-Power in a logarithmic graph. Fig. 4 shows measured OES intensities against RF coil power in a logarithmic graph. As no intensities for Ni ions were measured the graphs only contain the data for Ar and Ni neutrals. For the ICIS of Ni plasma there are two distinct slopes for powers below and above 2000 W respectively. For powers below 2000 W the slope is three times higher for Ni neutrals compared to Ar neutrals. According to the model a factor of 2 is to be expected. The reasons for this behaviour need to be examined further. For powers above 2000 W the slopes of Ar and Ni neutrals react as predicted by the model.

Table 1: Comparison of the slopes of Ar and Ni neutrals in ICIS plasma.

1.Ar 750slope:	0.53	2.Ar 750slope:	0.41
1.Ni 341 slope:	1.40	2.Ni 341 slope:	0.79
1.Ni 345 slope:	1.41	2.Ni 345 slope:	0.74
1.Ni 346 slope:	1.32	2.Ni 346 slope:	0.66

Figure 4 describes the influence of pressure on the ionisation at a constant power. It can be seen that for higher pressures the intensity of Ni neutrals reduces. Similar studies with ICIS of Ti showed that the reduction of neutrals is accompanied by an increase in Ti ion intensity, due to an increased efficiency of ionisation at higher pressures.
It can be expected that this effect would be valid in Ni and the reduction might be attributed to an increasing ionisation. Even though the intensity from Ni ions was not measurable, this indicates that the ionisation processes are more efficient at higher pressures. This could be attributed to a reduction in mean free path of collisions between Ni atoms and Ar atoms. This has two consequences. One is the reduction of energy of Ni atoms leading to longer transit times

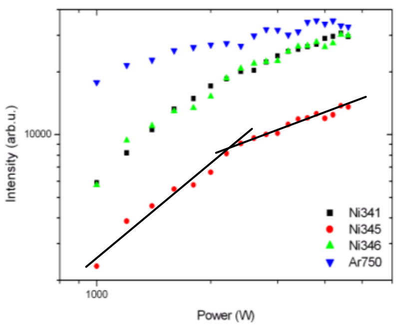

Figure 3 Measured results for Ar and Ni neutrals for a constant pressure of 12 Pa with varying RF-Power. The black lines highlight the different slopes below and above 2000 W

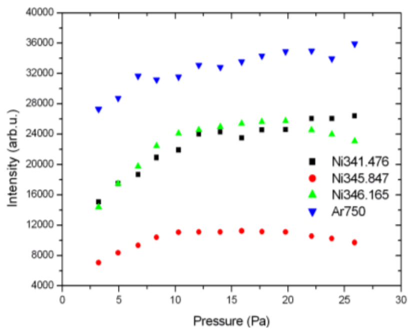

Figure 3 Measured results for Ar and Ni neutrals for a constant RF-Power of 3000 W and varying pressure.

through the ionisation zone of the RF coil. Another consequence is the greater frequency of collisions with Ar metastables. Both of these consequences result in a higher probability of ionisation through a Penning process.

3.3 Coating properties

The cross section image (Fig. 5) of a high aspect ratio(AR) via 2:1 coated by ICIS of Ni at RF-power of 4000 W and low pressure (6.4 Pa) being conditions that would result in a medium ionisation degree of the sputtered material. The films exhibit dense columnar dendritic growth. The bottom-coverage (BC) for these process settings and AR is 21.2 % with a floating bias. The deposition rate was approx. 125 nm/h.

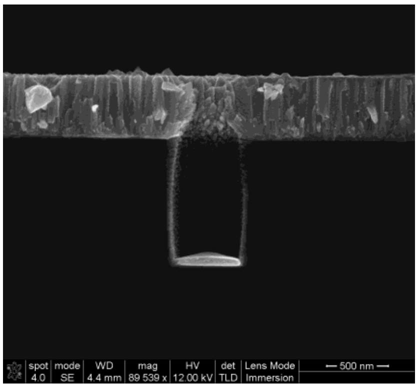

Figure 5 SEM cross section of Ni coated high aspect ratio vias deposited with 4000 W RF-Power at 6.4 Pa.

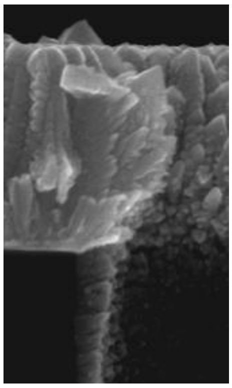

Figure 5a Close up of the corner of the via from figure 5. Dendritic growth of the Ni coating can be seen along with the perpendicular growth on the sidewalls.

In fig. 5a it can clearly be seen, that the growth of columns on the sidewalls is perpendicular to the substrate surface and homogeneously level over the whole depth of the feature.
The accumulation of material at the bottom of the structure indicates the deposition of ions. The overall distribution of the deposit indicates that the majority of the deposited species were metal ions. The dense structure of the bottom coating and no visible separation between the sidewall and bottom as well as the even sidewall coverage suggest only modest resputtering.

4 Conclusion

Successful deposition of magnetic material has been demonstrated by ICIS achieving high degrees of ionisation. While ions have not been detected by OES measurements there are numerous indicators that the sputtered target material does indeed get ionised. These are the deposition into high aspect ratio structures with good sidewall and bottom coverage, the horizontal orientation of columns on the sidewalls of vias and the reduced intensity of metal neutrals at higher pressures.
Further calculations of the excitation in ICIS processes has shown to be comparable to magnetron based systems but further work needs to be completed to measure the actual ionisation and to explain the higher excitation rate at lower powers.

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