
Articles you may be interested in

[Evidence of ionized metal clusters in ion plating discharges](#)

Appl. Phys. Lett. **81**, 1405 (2002); 10.1063/1.1501449

[Effect of sputter heating in ionized metal physical vapor deposition reactors](#)

J. Appl. Phys. **87**, 7198 (2000); 10.1063/1.372969

[Liner conformality in ionized magnetron sputter metal deposition processes](#)

J. Vac. Sci. Technol. B **14**, 2603 (1996); 10.1116/1.588993

[Magnetron sputter deposition with high levels of metal ionization](#)

Appl. Phys. Lett. **63**, 3285 (1993); 10.1063/1.110176

[Presence of doubly ionized metal ions in rare gas glow discharges](#)

J. Appl. Phys. **56**, 872 (1984); 10.1063/1.334027



Instruments for Advanced Science

Contact Hiden Analytical for further details:

W www.HidenAnalytical.com

E info@hiden.co.uk

CLICK TO VIEW our product catalogue



Gas Analysis

- › dynamic measurement of reaction gas streams
- › catalysis and thermal analysis
- › molecular beam studies
- › dissolved species probes
- › fermentation, environmental and ecological studies



Surface Science

- › UHV TPD
- › SIMS
- › end point detection in ion beam etch
- › elemental imaging - surface mapping



Plasma Diagnostics

- › plasma source characterization
- › etch and deposition process reaction
- › kinetic studies
- › analysis of neutral and radical species



Vacuum Analysis

- › partial pressure measurement and control of process gases
- › reactive sputter process control
- › vacuum diagnostics
- › vacuum coating process monitoring

ionization to higher discharge powers. The addition of large fluxes of metal atoms tends to cool the Ar RFI plasma, although this effect depends on the chamber pressure and probably the pressure response of the electron temperature. The technique has been scaled to 300 mm cathodes and 200 mm wafers and demonstrated with Cu, AlCu, and Ti/TiN. Deposition rates are equal to or in some cases larger than conventional magnetron sputtering. A primary application of this technique is lining and filling semiconductor trenches and vias on a manufacturing scale.

I. INTRODUCTION

Sputter deposition, in particular magnetron sputter deposition, is a broadly used process for depositing metal films for semiconductor applications, optical coatings, magnetic media, hard and decorative coatings, architectural glass, automobile parts, and so on. Physical sputtering results in the emission of mostly neutral atoms from the near surface region of the bombarded surface with an emission profile which is roughly dependent on the cosine of the emission angle from the surface normal. This effect, coupled with the extended area of most sputtering sources and possibly some gas scattering results in a deposition flux which arrives at a sample surface from many directions. While this is quite useful for forming continuous films over rough topography or steps, when applied to higher aspect ratios such as trenches or vias, the resultant deposition usually forms an enclosed channel or void (Fig. 1).

A second constraint to magnetron sputtering is the difficulty of drawing a significant bias current to a sample during sputter deposition. The magnetron plasma is generally located close to the cathode/target surface and relatively few ions are present in the vicinity of the sample. For many reactive deposition cases (particularly the nitrides for hard coatings), the addition of substrate bias is useful in adding the necessary energy to form the compound film. For conventional sputter deposition, it is necessary to heat the sample to 300–600 °C to form the nitride phase. Hard coating applications which could not allow significant heating (with substrates such as plastics, tool steels, etc.) led to the development of the unbalanced magnetron^{1,2} which, by suitable design of the magnetic field, can allow some of the electrons from the primary magnetron discharge to stream out towards the sample position. These electrons may either induce ions to follow by means of a weak potential gradient,

or may also form additional ions in the vicinity of the sample which can then be drawn to the sample as a bias current. This technology has been demonstrated on a commercial basis, but requires the use of large electromagnets to supply the magnetic field out to the position of the sample. Unbalanced magnetrons are not generally used for semiconductor applications of nitride films.

Recently, a technique has been developed which can address these two problems.^{3,4} If a significant fraction of the sputtered atoms from the cathode could be ionized in the region between the cathode and the sample, then the sputtered metal ions could be accelerated to the sample by means of a simple dc potential. The directionality of the metal ions would be well controlled: ions would be accelerated perpendicularly to the sample by the electric field in the plasma sheath at the sample surface. This effect has been demonstrated in a related experiment by Holber *et al.* using metal evaporation into an electron cyclotron resonance (ECR) plasma.⁵ In addition, the kinetic energy of the ions could be controlled at will simply by adjusting the relative potential difference between the plasma and the sample. Typically each of these effects only requires a simple negative dc bias on the sample.

II. EXPERIMENTS

Several systems were outfitted with a variety of commercial, planar magnetrons. These range from a 5 cm diam cathode operating at a few hundred watts to 30 cm rotating-magnet planar magnetrons which operate at powers up to 30 kW. The results described below were all taken with either a 20 cm planar (fixed magnet) cathode or with the larger, rotating magnet devices. These latter cathodes are used routinely on semiconductor manufacturing-scale systems such as the Varian M/2000 or Applied Materials 5500 integrated processing systems. No modifications were made to any of the cathodes.

³Current address: Department of Electrical and Computer Engineering, Northeastern University, Boston, MA 02115.

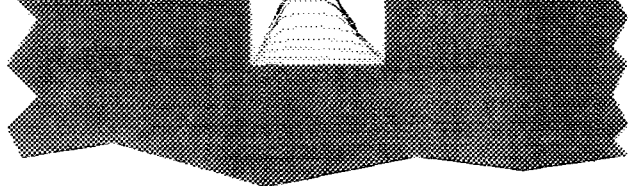


FIG. 1. Sketch of a cross section of a sputter deposited, layered film onto a medium aspect ratio feature.

A dense, inductively coupled rf (RFI) plasma was generated in the region between the sputtering cathode and the sample plane (typically a Si wafer mounted on a holder). The plasma was generated by applying 13.56 MHz power to a multiple turn coil of water-cooled Cu tubing located inside the vacuum chamber. A number of coil designs and locations were explored (Fig. 2). Much of this work utilized two to three turn coils which had a diameter approximately the same as the magnetron cathode and were located ~ 4 cm from the cathode surface and 2–3 cm from the sample surface. Spiral coils were also used on some of the rotating magnet systems. These latter systems required great attention to coil symmetry, as the moving magnetic field coupled weakly into the RFI plasma, causing variations on the scale of the magnet rotation rate.

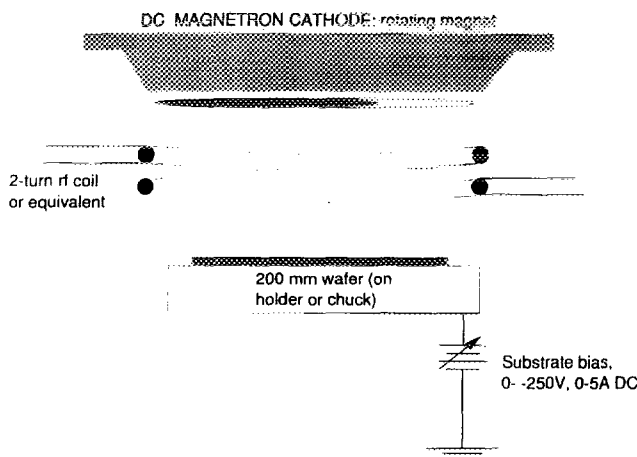


FIG. 2. Schematic of the experimental design.

these experiments were performed in test chambers of ~ 50 l vol and a base pressure of 10^{-8} Torr. Manufacturing systems for semiconductor applications typically would have a base pressure of 10^{-9} Torr, but generally do not operate at pressures above 5 mTorr by design.

To measure the relative ionization level of the metal fluxes (to the sample) one system was configured with a differentially pumped, gridded energy analyzer in place of the sample. The collector of this analyzer contained the active area a quartz crystal microbalance. In this way, the weight of the depositing flux to the collector surface as well as the current could be measured. This allows differentiation between the working gas ions (Ar) and the depositing metal ions. By suitable biasing of the grids in the energy analyzer, ions could be repelled, allowing the neutral metal deposition flux to be measured. In this way, the relative ionization level of the metal flux could be inferred. This measurement technique was only useful at low RFI densities. As the applied rf power exceeded about 500 W, the plasma density was high enough to penetrate the grid structure in the energy analyzer. It is estimated that this occurs at about 3×10^{11} ions/cm³ for the 300 mesh grids used. Higher rf powers were monitored by means of optical emission spectroscopy (OES) from the plasma, observing both metal ion and neutral emission lines.

III. RESULTS AND DISCUSSION

A. Relative Ionization

The work of Holber *et al.*⁵ used ECR ionization of metal atoms evaporated from a source which was not in the line-of-sight of the sample. The observed level of ionization was essentially 100%. In the present experiment, the ionization fraction was expected to be less than 100% and a function of the operating parameters, such as the system pressure, the rf power to the RFI plasma, and the injection rate of metal atoms into the Ar RFI plasma. In Fig. 3, the relative ionization for Al (from an Al cathode) as a function of chamber pressure at constant magnetron and RFI power is shown. At low pressure (a few mTorr), there is apparently little ionization and the deposition flux is almost entirely composed of neutral Al. This case would be similar to conventional magnetron sputtering and would result in features much like Fig. 1. As a function of increasing chamber pressure, the relative ionization rate climbs and saturates in the 30–40 mTorr range at $\sim 80\%$. This is consistent with the reduction of the mean free path for the sputtered atoms from a distance ex-

FIG. 3. Relative ionization level for sputtered Al as a function of chamber pressure for Ar and Ne working gases using a 20 cm diam planar AlCu cathode.

ceeding the cathode-to-sample (throw) distance at low pressures to a path length of a few millimeters at the higher pressure. Slowing down the sputtered atoms results in a greater chance for ionization due to an increased cross section⁶ and a longer residence time in the plasma.

A similar dependence is observed in the relative metal ionization as a function of applied rf power to the RFI plasma. At constant magnetron power (constant metal flux) and constant pressure (at the high end), the relative ionization climbs and saturates as a function of rf power (Fig. 4). The saturation is again ~80% for these conditions. Measurements at the upper end of this range are somewhat questionable, because at slightly higher powers the plasma penetrates the grids. This would indicate curvature in the sheaths near the grids at powers ~500 W, which may result in significant defocusing of the ions and reduced collection.

The effect of high rates of metal sputtering, and the subsequent high fluxes of metal atoms entering the RFI plasma, is to cool the RFI plasma. This was observed with both the energy analyzer as well as OES. The results of increasing the

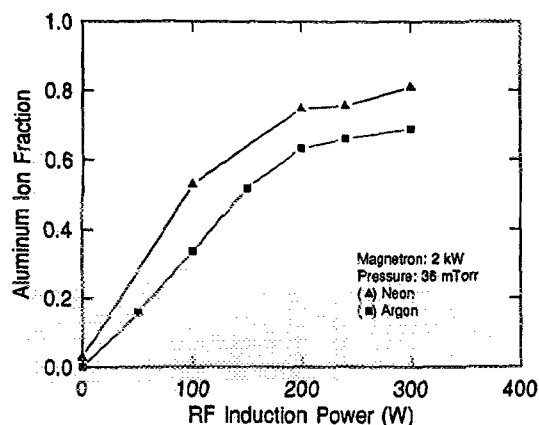


FIG. 4. Relative ionization for sputtered Al at constant pressure as a function of rf power into the inductively coupled plasma. The cathode used was a 20 cm diam AlCu planar cathode at 36 mTorr pressure.

JVST B - Microelectronics and Nanometer Structures

FIG. 5. Relative ionization for sputtered Al as a function of increasing rf power and increased metal sputtering rate for a 20 cm dis. AlCu cathode.

magnetron power, and hence the metal sputtering rate, is shown in Fig. 5, which shows the relative ionization as a function of rf power for several magnetron powers. Even though the depositing metal flux increases markedly, the relative number of ions decreases, consistent with a cooling of the electron temperature in the RFI plasma. Optical emission from the Ar also showed a similar effect (Fig. 6), although some amount of this reduction may also be due to gas heating and rarefaction.⁷ Because of the metal-rich character of these plasmas, there was no attempt to make classical Langmuir probe measurements of the electron temperature. Previous measurements in magnetron plasmas have proven difficult due to the deposition of metal on the insulating parts of the probe.⁸

By using OES and observing both metal neutral and metal ion emission, the relative ionization could be qualitatively extended to higher rf powers. This is shown in Fig. 7, in which the relative ionization inferred from OES is normalized to a low power (200 W) measurement with the electrostatic energy analyzer. This indicates that the cooling effect of the metal ion fluxes can be countered somewhat by additional rf power to the RFI plasma.

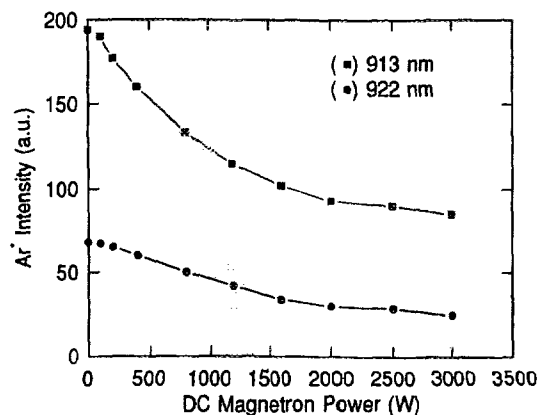


FIG. 6. Optical emission from Ar as a function of increased metal sputtering rate (dc magnetron power). Optical emission from the region above sample location, 10 cm from the cathode.

FIG. 7. Relative ionization level for sputtered Al as a function of rf power to the RFI plasma. The solid data points to the left-hand side are from Fig. 5 using the mass-sensitive energy analyzer. The open data points were taken using OES, comparing the intensity of an ionized Al line to the sum of the intensity of that ionized line and a nearby Al neutral emission line. The OES data were then normalized to the energy analyzer data at 200 W.

The high levels of metal ionization (50%–90%) should not be surprising given the nature of the experiment. The RFI plasma was generated using Ar, which has an ionization potential of 15.7 eV. The electron temperature at these pressures (30 mTorr) is relatively low (1–2 eV), but it is sufficient to create a high density plasma ($>10^{11}/\text{cm}^3$). The typical metal species used (Cu, Ti, Al) have ionization potentials in the 5–8 eV range, although their cross sections at these energies are not well characterized. The introduction of a metal atom into the 1–2 eV plasma should result in a relatively rapid ionization of the metal atoms. Approximate calculations (because of the lack of well defined cross sections for ionization of the metal species) are consistent with the observation of high levels of ionization as the pressure is increased. The increase in pressure results in a longer residence time for the metal atom in the plasma. At low pressures, the sputtered atoms transit the plasma rapidly at several eV of kinetic energy.

The observed cooling of the plasma, as implied by both the reduced relative ionization as well as the reduced OES signal from the inert gas, is also a fairly obvious result. The metal fluxes into these plasmas can be rather significant, even at modest magnetron sputtering rates. For example, for the sputtering of Cu, the sputter yield is ~ 2.0 . At a magnetron discharge power of 2 kW, the typical discharge current might be 5 A. This results in a net emission rate from the cathode of 6×10^{19} atoms/s, or the equivalent of over 100 sccm. This is somewhat misleading, though, because the pumping speed is effectively infinite for these metal atoms. However, since the majority of the metal atoms can be observed to be ionized, it can be assumed that they are also rapidly absorbing energy and emitting photons. This effect can be qualitatively addressed by measuring the amount of additional rf power needed to recover the same ion saturation current to the sample as the metal flux is increased. The

diffusion barriers along the sides and bottom of high aspect ratio trenches. Thin films of TiN have many interesting characteristics: they are hard, chemically inert, moderately conducting, and have an attractive gold color. Under conventional sputter deposition conditions, the deposition of TiN requires either significant sample heating (300–700 °C) or measurable sample bias currents (milliamps per square cm) which require the use of unbalanced magnetrons. Collimated or filtered magnetron sputtering has been used successfully to deposit thin TiN films on the sides and bottom of trenches with good conformality.¹⁰ This process is enhanced by the much less than 1.0 sticking (or reaction) coefficient for the depositing film atoms, which allows some redistribution of the atoms within the trench, leading to better than expected coverage. However, collimated sputtering can be moderately slow and there are concerns with particle formation on the collimator, which receives a large deposition flux.

TiN films were deposited using the present experiment by reactively sputtering a Ti cathode in a combination of Ar and

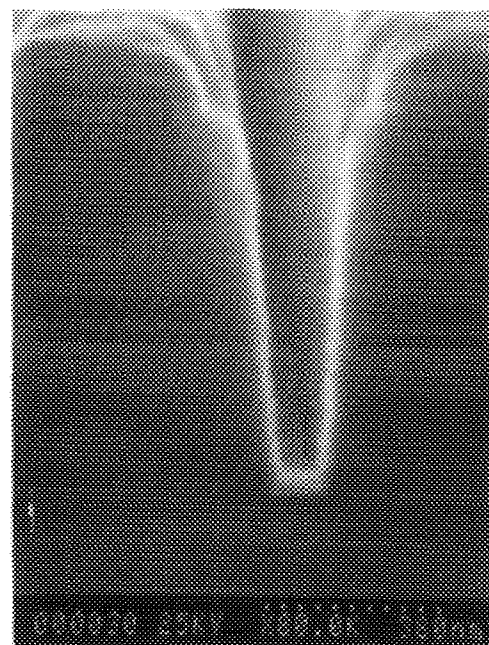


FIG. 8. Cross section of high aspect ratio trench which has been lined with TiN at room temperature using ionized magnetron sputter deposition.

Explore Litigation Insights

Docket Alarm provides insights to develop a more informed litigation strategy and the peace of mind of knowing you're on top of things.

Real-Time Litigation Alerts



Keep your litigation team up-to-date with **real-time alerts** and advanced team management tools built for the enterprise, all while greatly reducing PACER spend.

Our comprehensive service means we can handle Federal, State, and Administrative courts across the country.

Advanced Docket Research



With over 230 million records, Docket Alarm's cloud-native docket research platform finds what other services can't. Coverage includes Federal, State, plus PTAB, TTAB, ITC and NLRB decisions, all in one place.

Identify arguments that have been successful in the past with full text, pinpoint searching. Link to case law cited within any court document via Fastcase.

Analytics At Your Fingertips



Learn what happened the last time a particular judge, opposing counsel or company faced cases similar to yours.

Advanced out-of-the-box PTAB and TTAB analytics are always at your fingertips.

API

Docket Alarm offers a powerful API (application programming interface) to developers that want to integrate case filings into their apps.

LAW FIRMS

Build custom dashboards for your attorneys and clients with live data direct from the court.

Automate many repetitive legal tasks like conflict checks, document management, and marketing.

FINANCIAL INSTITUTIONS

Litigation and bankruptcy checks for companies and debtors.

E-DISCOVERY AND LEGAL VENDORS

Sync your system to PACER to automate legal marketing.