SAND2011-3616C

Pulsed DC Magnetron Sputtered Nickel Thin Films: A Study of Stress, Density and Electronic Properties

Program Number: TF-ThP2

***Eric. D. Jones Jr.* and David P. Adams**

Sandia National Laboratories, Albuquerque, New Mexico

AVS New Mexico Chapter

2011

Abstract:

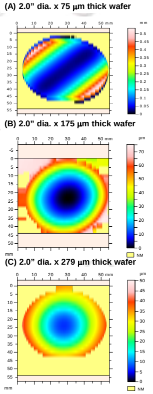
With the advent of magnetron pulsed direct current (PDC) sputter deposition technology, there have been gains made in the deposition of various inorganic thin films.[1] Most notably used for reactive sputtering of oxides and nitrides, PDC sputtering has led to the deposition of dense coatings with improved properties such as adhesion and wear.

In this work, we explore the effects of using PDC methods for sputtering monolithic ~ 200 nm thick nickel films. The power supply used for this study is the Advanced Energy Pinnacle Plus 10 kW series. Experiments are conducted in a 5 x 10^{-8} Torr base pressure vacuum system employing a side-sputtering geometry. We demonstrate the effects of argon sputter pressure (from 1 to 25 mTorr) on residual stress, film density and film resistivity for various PDC processes and contrast these with the properties formed during continuous DC sputtering. For the PDC experiments we show how these three film properties (stress, density and resistivity) depend on argon pressure for each of four different pulse frequencies (50, 150, 250 and 350 kHz - reversal time is held constant at 1.0 µs). In general, a range of in-plane tensile stress can be tailored during pulsed and non-pulsed DC sputtering through the control of argon sputter pressure. Pulse frequency is shown to have a minor effect on residual stress. We further evaluate the stress of all films in terms of intrinsic and extrinsic contributions to isolate the role of PDC sputtering process parameters on intrinsic stress. Extrinsic stress effects (due to mismatch of thermal expansion coefficients) are factored out from the residual stress using measured deposition temperatures. Film resistivity increases with argon sputter pressure for both continuous and PDC modes. Nickel film density is affected by argon sputter pressure with PDC generating less dense coatings.

The Goals of this study include:

- Determine optimal wafer thickness for nickel thin film stress studies.
- Evaluate the in-plane stress in nickel films.
- Determine the film stress dependence on argon pressure and pulsed DC frequency.
- Determine the effects of heat (generated during the sputter deposition process). on the residual stress of the film.
- Evaluate film resistivity vs. argon pressure/ PDC frequency using non-contact eddy current mapping.
- Measure film density and surface roughness using X-ray reflectivity.

Determining Correct Silicon Substrate Thickness for Film Stress Studies



Key Findings:

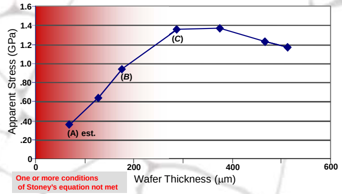
(A) The 75 µm thick wafer shows cylindrical bowing on an oblique axis that runs NW to SE. It has a large bow of 450 µm, along the oblique axis, and a small bow (<50 µm) along the cylindrical axis.

(B) The 175 µm thick wafer shows non-spherical bow of ~60 µm.

(C) The 279 µm thick wafer exhibits spherical bowing with nearly the same bow in all directions averaging ~ 35 µm.

Identifying a valid range of wafer thicknesses is critical to applying Stoney's[2] equation, which calculates stress from measured curvature. Non-linear effects[4] show up under moderate to high levels of stress when using thin wafers. Conversely, excessively thick wafers can give misleading values of stress if the change in curvature is near the detection limit of the instrument.

Apparent Stress vs. Wafer Thickness with ~200 nm Nickel @ 5 mT, 150 kHz



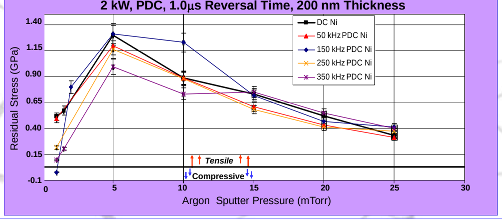
Evaluating Thin Film Stress as a Function of Pulsing Frequency and Argon Sputter Pressure

Controlling argon sputter pressure to affect stress is a well known technique. In this presentation we explore the residual film stress as a function of pressure and pulsed DC frequency.

Key Findings: From the graph below

- Non-linear effects of pulsed DC (50-350 kHz) across the range of sputter pressures used (1-25 mTorr).
- The greatest differences in residual stress for pulsed DC operation occur for pressures <15 mTorr.
- Residual stress of films grown by continuous DC and PDC is affected by argon sputter pressure.
- The majority of Ni films exhibit an in-plane tensile stress.

Stress of Nickel Thin Film as a Function of Pressure and Frequency 2 kW, PDC, 1.0µs Reversal Time, 200 nm Thickness



Evaluating the Effects of Heat Produced During the Sputtering Process on Residual Stress

Wafer heating occurs during the sputter deposition process is revealed by Omega temperature dots. This is due to the kinetic energy of sputtered particles, charged particles and the released condensation heat[3]. Extrinsic stress in the films can be calculated using the following formula:

$\Delta\sigma_{ext}/\Delta T = M_f * (\alpha_s - \alpha_f)$

So $\Delta\sigma_{ext} = [M_f * (\alpha_s - \alpha_f)] * \Delta T$ = extrinsic stress

Intrinsic stress $(\sigma) = \sigma_{residual} - \Delta_{ext}$

Where:

σ is the value of stress, typically MPa

T is the temperature in °C

M_f is the biaxial modulus of nickel[5] (2.9E+11 Pa)

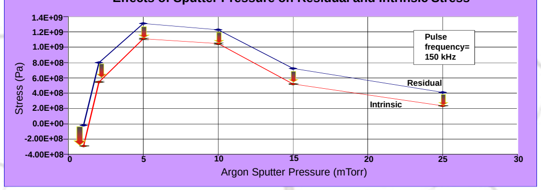
α_s is the substrate thermal expansion coefficient (2.841E-6/°C)

α_f is the film thermal expansion coefficient (13.437E-6/°C)

Key Findings: From the graph below

- The residual stress of Ni films is 10-25% (ave.) more tensile than the intrinsic stress across the pressures used and shown here @ 150 kHz.
- More heat is generated at lower pressures resulting in larger differences in the intrinsic stress.

Effects of Sputter Pressure on Residual and Intrinsic Stress



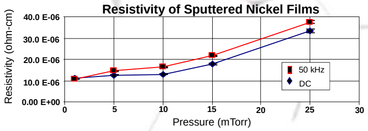
Evaluating Film Resistivity vs. Argon Sputter Pressure/Frequency

We explore the effects of sputter pressure and pulsed power frequency on the resistivity of the sputtered films.

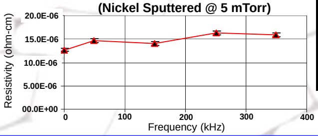
Key Findings: From the graphs below

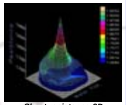
- Continuous DC sputtered films are less resistive than pulsed DC sputtered films.
- Resistivity increases with sputter pressure (for both modes).
- At a sputter pressure of 5 mTorr, resistivity changes only slightly across a range of pulsed DC frequencies.

Resistivity of Sputtered Nickel Films



Resistivity vs. Pulse Frequency (Nickel Sputtered @ 5 mTorr)



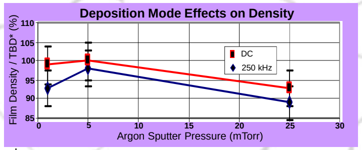


Sheet resistance 3D contour map of DC sputtered nickel @25 mTorr

Measurement of Film Density and Surface Roughness Using X-Ray Reflectivity

Using X-ray reflectivity, we determine the density and roughness of the nickel films and compare the values developed during continuous DC and 250 kHz pulsed DC sputtering, across the range of pressures of 1-25 mTorr. X-ray reflectivity is a non-destructive measurement technique used for estimation of density, thickness and roughness of thin film structures. It is based on total external reflection of X-rays from surfaces and interfaces that are amorphous, crystalline, or liquid, having layer thicknesses of 5Å to 400nm and a surface roughness of 0 to 20Å.

Deposition Mode Effects on Density



*Theoretical Bulk Density

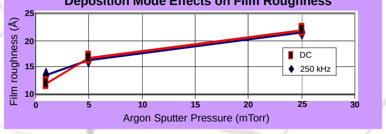
Key Findings: From the graph at left

- The densities of films grown in each of the two modes, PDC and continuous DC sputtering were evaluated using X-ray reflectivity. It is shown that at three different pressures, continuous DC operation produced slightly denser films. This dovetails with the resistivity data that shows continuous DC films have slightly less resistance.

Key Findings: From the graph at right

- The films exhibit nearly the same roughness for continuous and pulsed DC for a given argon sputter pressure.
- The roughness increases with sputter pressure.

Deposition Mode Effects on Film Roughness



Conclusions:

- Moderate to high levels of stress induce non-spherical bowing when using thin wafers. This leads to an underestimation of film stress when applying Stoney's equation.
- Pulse frequency has a minor effect on the residual stress of Ni films with the greatest differences occuring below 15 mTorr.
- Extrinsic stress from heat built up during sputter deposition is shown to be approximately 10-25% of the total residual stress.
- The resistivity of DC sputtered nickel films is slightly less than PDC sputtered coatings and resistivity increases with sputter pressure.
- Resistivity increases slightly with an increase in power supply frequency at the sputter pressure of 5 mT.
- The density of continuous DC sputtered films is 4.4% (ave.) greater than PDC sputtered films at three different pressures.
- Film roughness is comparable for pulsed and continuous DC operation.
- Film roughness increases with sputter pressure.

Acknowledgements/References

The author wishes to acknowledge the assistance of Mark A. Rodriguez and Jeffery G. Cederberg for data collection.

References:

1) J. Lin, J.J. Moore, W.D. Sproul, B. Mishra and Z. Wu, Thin Solid Films 518 (2009) 1566.
2) G.G Stoney, Proc. Roy. Soc. Ser A, 82 (1909) 172.
3) H. Kersten, G. M. W. Kroesen and R. Hippler, Thin Solid Films 332 (1998) 282-289.
4) L.B Freund, J.A Floro and E. Chason, Appl. Phys. Lett. 74 (1999) 1987.
5) G. W. C. Kaye and T. H. Laby, Tables of Physical and Chemical Constants, 14th ed., Longman, London, 1973, p. 31.

Equipment used:

- **Sputtering chamber-** KDF Vacuum and Electronics 744i
- **Power supplies -** Advanced Energy 10Kw Pinnacle Plus power supplies
- **Thickness measurements –** Dektak 150 (Veeco) profilometer**,** Mitutoyo Model ID-F125E
- **Contactless resistance measurement tool-** Lehighton Electronics Model 1510B-SA
- **Stress Measurements-** Tencor FLX 2320
- **Wafer Profiler (Bow) measurements-** Solarius Laser Scan
- **Density-**Scintag powder diffractometer using copper radiation, a incident beam mirror (with 50 micron slit), solid state germanium detector (Peltier cooled)

This work was supported by the Laboratory Directed Research and Development program at Sandia National Laboratories. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000.