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## Introduction – Magnetron sputtering

 Magnetron sputtering is applied to deposit magnetic thin films



- Conventional dc magnetron sputtering (dcMS) suffers from a low degree of ionization of the sputtered material
- High power impulse magnetron sputtering (HiPIMS) provides a highly ionized material flux, while being compatible with conventional magnetron sputtering deposition systems



## Introduction – HiPIMS

- High ionization of sputtered material requires very high density plasma
- In a conventional dc magnetron sputtering discharge the power density (plasma density) is limited by the thermal load on the target
- High power pulsed magnetron sputtering (HPPMS)
- In a HiPIMS discharge a high power pulse is supplied for a short period
  - Iow frequency
  - Iow duty cycle
  - low average power



Gudmundsson et al. (2012) JVSTA 30 030801

Power density limits

- $p_{t} = 0.05 \text{ kW/cm}^{2} \text{ dcMS}$  limit
- $p_{\rm t} = 0.5 \ {\rm kW/cm^2} \ {\rm HiPIMS} \ {\rm limit}$



## Introduction – Thin magnetic films





- To obtain magnetic hysteresis loops, we use a high sensitivity magneto-optical Kerr effect (MOKE) looper
- The **coercivity** *H*<sub>c</sub> is the field required to reduce the magnetic induction to zero and is read directly from the easy axis loops
- The **anisotropy field** *H*<sub>k</sub> corresponds to the internal field along the hard direction of magnetization which counterbalances the torque resulting from the anisotropy
- Magnetic anisotropy simply states that the magnetic properties depend on the direction in which they are measured



### Nickel

- Nickel is a ferromagnetic material of heavy 3d transition metal that crystallizes in the fcc structure
- HiPIMS and dcMS for depositions were made at substrate tilt angles ranging from 0° (substrate faces the target) to 70°
- The distance between target and substrate position is 25 cm and working gas pressure 0.6 Pa
- The substrates used were thermally oxidized Si(001) with an oxide thickness of 100 nm



After Cullity and Graham (2009). Introd

to Magnetic Materials, John Wiley

- The film density, deposition rate and surface roughness of Ni films deposited by HiPIMS and dcMS at 0, 35 and 70° tilt angles
- Increasing the tilt angle to 70° leads to a drop in density for both methods
- The dcMS deposition rate is roughly double the HiPIMS deposition rate
- HiPIMS deposited films show lower roughness than dcMS deposition for low tilt angle





- The films deposited by HiPIMS at 0, 35 and 50° tilt angle exhibit in-plane biaxial anisotropy
- The films deposited at 60 and 70° present an uniaxial behavior i.e. linear hard axis traces along the angle of incidence and square easy axis perpendicular to that
- Regardless of the anisotropy the coercivity H<sub>c</sub> of the film increases with increasing the tilt angle
- This is also true for the anisotropy field *H*<sub>k</sub> in the samples with uniaxial anisotropy



- The film deposited by dcMS at 10° tilt angle shows in-plane biaxial anisotropy
- Films deposited by dcMS at 35° and 70° tilt angles exhibit uniaxial anisotropy with hard axis along the angle of incidence and easy axis perpendicular to that
- In contrast with the HiPIMS results, dcMS deposited films present a reduction in H<sub>c</sub> with increase in tilt angle up to 35° and then it exhibits an increase



#### Nickel

- The coercivity of the Ni films are plotted as a function of the tilt angle
- Regardless of the type of anisotropy, along the easy direction of magnetization, H<sub>c</sub> of the HiPIMS deposited films increases with increasing tilt angle
- dcMS deposited films present a reduction in H<sub>c</sub> with increase in tilt angle up to 35° and then an increase with further increase in tilt angle



- The cross sectional SEM images of HiPIMS and dcMS deposited films under 70° substrate tilt angle
- The dcMS deposited film exhibits inclined columnar growth with column length extending through the entire film thickness – with 32° incline on the substrate
- The HiPIMS deposited film shows grains that are smaller than the film thickness – the grains do not show a well defined inclined growth





- HiPIMS deposited Ni films are magnetically softer than dcMS deposited films at the same tilt angle
- For both methods lower tilt angle gives biaxial anisotropy and it changes to uniaxial anisotropy at higher tilt angle
- The transition turning point for HiPIMS deposition is above 50° while it is less than 35° for dcMS



## Permalloy Ni<sub>80</sub>Fe<sub>20</sub> – polycrystalline



## Permalloy Ni<sub>80</sub>Fe<sub>20</sub> – polycrystalline

- Permalloy Ni<sub>80</sub>Fe<sub>20</sub> is a very well known ferromagnetic material and has over the years been used extensively in various industrial applications
- Fe-Ni alloys crystallize as fcc when the nickel fraction is larger than 30%
- We explore the microstructure and magnetic properties of Ni<sub>80</sub>Fe<sub>20</sub> thin films (37 nm) deposited by HiPIMS and dcMS on p-Si(001) with native oxide layer at 35° tilt deposition



## Permalloy Ni<sub>80</sub>Fe<sub>20</sub> – polycrystalline

- GIXRD patterns of HiPIMS deposited films at 100°C at different pressures
- The three main peaks are 44.217, 51.518 and 75.845° corresponding to (111), (200) and (220) planes, respectively – the dominant peak is (111) in all cases
- The (111) texture provides perpendicular anisotropy to the permalloy films as for fcc alloy structures the (111) direction is the easy magnetization axis



Kateb et al. (2018) J. Phys. D. 51 285005



## Permalloy Ni<sub>80</sub>Fe<sub>20</sub> – polycrystalline

- The results of the fitting of XRR data for dcMS and HiPIMS depositied films of 37 nm thickness
- HiPIMS deposition exhibits significantly lower average deposition rate than dcMS deposition
- HiPIMS deposition at room temperature can maintain high density for most of the cases explored
- In general, we see increased surface roughness with increasing working gas pressure



## Permalloy Ni<sub>80</sub>Fe<sub>20</sub> – polycrystalline

- The variation of the anisotropy field *H*<sub>k</sub> and coercivity *H*<sub>c</sub> with pressure for both dcMS and HiPIMS grown films
- The *H*<sub>k</sub> increases with increased pressure for films grown by dcMS at room temperature
- All other films have either a nearly constant *H<sub>k</sub>* (dcMS and HiPIMS at 100°C) or a delayed and slower growth as function of pressure
- From the inset we see there is a strong correlation between film density, H<sub>k</sub> and H<sub>c</sub> i.e. high film density maintains low H<sub>c</sub> and low H<sub>k</sub>



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## Permalloy Ni<sub>80</sub>Fe<sub>20</sub> – polycrystalline

- The MOKE response of the films deposited by dcMS and HiPIMS at room temperature measured along
  - (a) hard axis of the films
  - (b) easy axis of the films
- dcMS grown films always present higher saturation fields than their HiPIMS counterparts grown at the same pressure
  - (a) dcMS grown films dotted lines
  - (b) HiPIMS grown films dashed lines



## Permalloy Ni<sub>80</sub>Fe<sub>20</sub> – polycrystalline

- X-ray diffraction reveals that there is very little change in grain size within the pressure and substrate temperature ranges explored
- The film density, obtained by XRR measurements, can be related to magnetic properties such as anisotropy field (*H*<sub>k</sub>) and coercivity (*H*<sub>c</sub>)
- Depositions where adatom energy is high (like HiPIMS) produce dense films, while low adatom energy (like dcMS) results in void-rich films with higher H<sub>k</sub> and H<sub>c</sub>





- Ni<sub>80</sub>Fe<sub>20</sub> thin films were epitaxially deposited on single crystalline MgO(001)
- The vertical dashed lines show the peak position of bulk Ni<sub>80</sub>Fe<sub>20</sub> and MgO
- The fcc (002) peak is the only detectable Ni<sub>80</sub>Fe<sub>20</sub> peak indicating that the (002) planes of Ni<sub>80</sub>Fe<sub>20</sub> are very well aligned to that of the MgO substrate i.e. Ni<sub>80</sub>Fe<sub>20</sub>(001) || MgO(001)



Kateb et al. (2019) AIP Adv. 9 035308



- We note a slight left shift in the (002) peak in the HiPIMS deposited films
- We attribute the shift in the (002) peak to departure from the L1<sub>2</sub> Ni<sub>3</sub>Fe superlattice – (002) peak shift to the right indicates higher order and to the left indicates more disorder
- This is because the Ni-Fe bond is shorted than both Fe-Fe and Ni-Ni bonds
- In the Ni<sub>3</sub>Fe super lattice, all bonds are Ni-Fe



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- In the {200} pole figure, there is an intense spot at  $\psi = 0$  that verifies that the (002) plane is lying parallel to the substrate i.e. epitaxial relationship of Ni<sub>80</sub>Fe<sub>20</sub>(001) || MgO(001) for both dcMS and HiPIMS deposited films
- The extra dots that appear in the {111} pole figure of the HiPIMS deposited film belong to twin boundaries
- The existence of twin boundaries in the Ni<sub>80</sub>Fe<sub>20</sub> films is a signature of high deposition rate



- Hysteresis loops obtained by MOKE along the [100] and [110] directions
- The upper figures indicate a biaxial behaviour in the dcMS deposited film consisting of two easy axes along the [110] directions with  $H_c$  of  $\sim$ 2 Oe
- This is consistent with the  $\langle 111 \rangle$ direction being the easy direction of the Ni<sub>80</sub>Fe<sub>20</sub> crystal and the magnetization being forced in-plane along the  $\langle 110 \rangle$  directions due to shape anisotropy





- The HiPIMS deposited film shows very well-defined uniaxial anisotropy indicated by a linear hard axis trace without hysteresis and slightly rounded easy axis loop along the [100] directions
- The anisotropy field (*H<sub>k</sub>*) is 3.5 Oe, i.e. much lower than the value observed for polycrystalline films deposited by HiPIMS on Si/SiO<sub>2</sub> (11 – 14.5 Oe)
- The coercivity (*H*<sub>c</sub>) of 1.8 Oe is very close to that of polycrystalline films i.e. 2 2.7 Oe



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### Permalloy Ni<sub>80</sub>Fe<sub>20</sub> – epitaxy

 The AMR response to the rotation of in-plane saturated magnetization

$$AMR = \frac{\Delta \rho}{\rho_{ave}} = \frac{\rho_{\parallel} - \rho_{\perp}}{\frac{1}{3}\rho_{\parallel} + \frac{2}{3}\rho_{\perp}}$$

- The dcMS deposited films are thinner but the resistivities in the dcMS case are all lower than the HiPIMS ones, which contradicts the Fuchs model
- The lower resistivity of dcMS deposited can be explained in terms of higher Ni<sub>3</sub>Fe order achieved in the dcMS deposited film



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- The dcMS deposited epitaxial film has biaxial symmetry in the plane, with easy directions [110] as one might expect for a bulk fcc magnetic material
- The HiPIMS deposited epitaxial film exhibits different magnetic symmetry, uniaxial anisotropy with (100) as the easy direction and the film is magnetically soft and has an anisotropy field of only 3.5 Oe
- We attributed the uniaxial anisotropy to less ordered dispersion of Ni and Fe at the atomic level in the film deposited by HiPIMS due to high deposition rate of HiPIMS during the discharge pulse



## Summary



## Summary

- Nickel films exhibit in-plane biaxial anisotropy for small tilt angles while for larger tilt angles they exhibit uniaxial anisotropy
- The tilt angle where the transition occurs depends on the deposition method
- For polycrystalline Ni<sub>80</sub>Fe<sub>20</sub> films the coercivity *H*<sub>c</sub> depends on the film density and increases as the film density drops
- The dcMS deposited epitaxial film has biaxial symmetry in the plane, with easy directions [110] while the HiPIMS deposited epitaxial film exhibits uniaxial anisotropy with (100) the easy direction
- We attributed the uniaxial anisotropy to less ordered dispersion of Ni and Fe at the atomic level in the film deposited by HiPIMS due to high deposition rate of HiPIMS during the discharge pulse

## Thank you for your attention

The slides can be downloaded at

http://langmuir.raunvis.hi.is/~tumi/ranns.html
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