

# Highly Oriented Co Soft Magnetic Films on Si Substrates

Heng Gong, Wei Yang, Maithri Rao\*, David E. Laughlin\*, and David N. Lambeth

Department of Electrical and Computer Engineering, [hgong@andrew.cmu.edu](mailto:hgong@andrew.cmu.edu),

\*Department of Materials Science and Engineering,

Data Storage Systems Center, Carnegie Mellon University, Pittsburgh, PA 15213-3890

## ABSTRACT

Thin Co and Co based alloy films with the face centered cubic (FCC) structure have been epitaxially grown on single crystal Si wafers by sputter deposition. Epitaxial orientation relationships have been determined by x-ray diffraction, x-ray pole figure scans and TEM. Magnetic properties have been characterized using vibrating sampling magnetometer (VSM), torque magnetometer and BH loop tracer. Soft magnetic properties have been observed for the pure Co films.

## INTRODUCTION

Soft magnetic thin films are widely used in magnetic storage applications and the control of the crystalline orientation and microstructure is desirable as this directly influences the anisotropy, coercivity and device functionality. Recently, very thin films of soft Co and Co based alloys have become important for sensor applications. In particular, Co and Co<sub>90</sub>Fe<sub>10</sub> alloys are currently used in GMR spin valve structures for ultra-high density magnetic recording. Hence, it is of considerable interest to fully understand the properties of soft Co films. Previously we have discussed highly oriented NiFe (permalloy) films epitaxially grown on Ag and Cu underlayer templates, which were grown on HF etched Si substrates by sputter deposition [1,2]. In this study, using similar template techniques, highly oriented Co and Co based alloy thin films with soft magnetic properties have been successfully prepared on HF etched Si substrates. From TEM and X-ray pole figure  $\phi$  scan measurements, it has been found that the FCC phase of Co grows and the epitaxial relationships were determined.

The film structures are sketched in Figure 1. Single crystal Si wafers with (111), (100) and (110) orientations were employed to induce the epitaxial growth of FCC Co films with the respective orientations. As we reported earlier, because of the 4:3 match between the Ag and Si lattice parameters, Ag grows epitaxially on Si(111), (100) and (110) with (111), (100) and (110) orientations, respectively [1-5]. In this study, epitaxial growth was also observed between Cu and Ag and between Co and Cu in all three orientations. Unlike the NiFe case, where a Cu buffer layer was indispensable to inducing the epitaxial growth of the NiFe film [2], it was observed that FCC Co can grow epitaxially directly on the Ag films. However, the structure containing a Cu intermediate layer may alleviate stress at the interface, especially when the



Fig. 1 Schematic drawing of the Co film structures epitaxially grown on Si wafers.

required Co film thickness is small, such as in a GMR spin-valve structure.

## EXPERIMENTAL

Si wafers were first ultrasonically cleaned in organic solvents and rinsed in deionized water. Clean wafers were then immersed in HF to remove the native SiO<sub>2</sub> and obtain a hydrogen-terminated surface. The etched wafers were blown dry using N<sub>2</sub> gas, then placed into the sputtering system. Samples were deposited by rf diode sputtering in a Leybold-Heraeus Z-400 sputtering system. The base pressure was below 5×10<sup>-7</sup> Torr. The Ar sputtering gas pressure was fixed at 10 mTorr and the sputtering power density was about 2.3 W/cm<sup>2</sup>. The substrate was kept at room temperature or pre-heated to about 260°C during sputtering. Samples which deposited at room temperature and the elevated temperature were prepared. The epitaxial structure of the films was studied by x-ray  $\theta/2\theta$  diffraction and pole figure  $\phi$ -scan, as well as TEM. The magnetic properties of the films were measured using a vibrating sampling magnetometer, torque magnetometer and BH loop tracer.

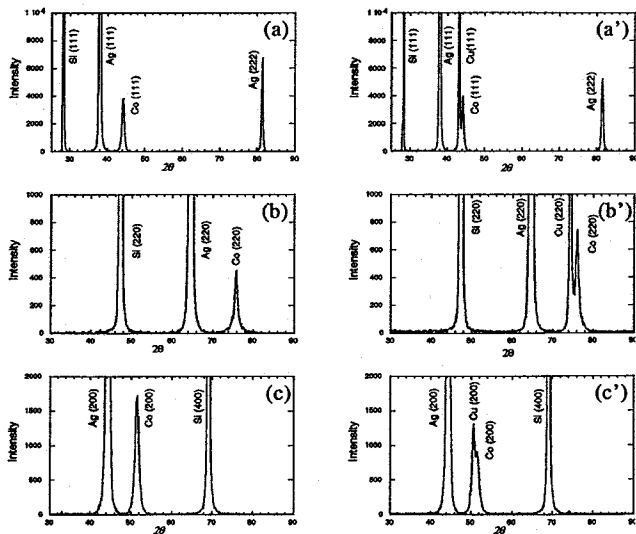


Fig. 2 X-ray diffraction patterns of Co/Ag films deposited on (a) Si(111), (b) Si(110) and (c) Si(100) substrates, as well as Co/Cu/Ag samples deposited on (a') Si(111), (b') Si(110) and (c') Si(100) substrates.

## RESULTS AND DISCUSSIONS

### Epitaxial Relationships

Figure 2 shows the x-ray diffraction patterns of samples deposited at 260°C, with and without the Cu intermediate layers. The film thickness of the Co and Cu layers were 50 nm, while the Ag film thickness was fixed at 75 nm for all samples. In  $\theta$ -2 $\theta$  x-ray diffraction patterns, only the FCC textures correspondent to the epitaxial relationships between each of the FCC layers were observed. Hence, in order to investigate the epitaxial orientation relationships in the plane, x-ray pole figure  $\phi$  scan measurements were employed. Figure 3 shows the measured  $\phi$  scan spectrum for samples with successive added layers to form the final structure of Co (50nm) / Cu (50nm) / Ag (75nm) / Si (111). As can be seen in Figure 3a, the Si{110} pole scan spectrum of the single crystal Si(111) substrate yields three diffraction peaks with 120° separation. They correspond to the three {110} poles in the Si (111) stereographic projection, which are also 120° apart. While three Ag{110} poles are expected for single crystal Ag(111), six peaks were observed in the Ag{220}-pole scan [Fig. 3(b)] of the Ag(111) film grown on Si(111). This suggests that there exist two twin-related orientations of Ag grains in the Ag(111) film. They may emerge at the Ag/Si epitaxial interface due to nucleation or result from Ag twinning during growth. In FCC metals, the (111) plane is the most common twinning plane, and the twinning direction is  $[1\bar{1}2]$ . Three of the peaks, the first, third and fifth, appear at the same  $\phi$  positions as the three peaks in Si{110}-pole spectrum, confirming the parallel relationship between the Si $\{1\bar{1}0\}$  and Ag $\{1\bar{1}0\}$  directions. Fig. 3(c) and 3(d) show the Cu and Co {220} pole scan of the (111) Cu and Co layers, respectively. Since the Ag is twinning we expected the Cu and Co show six peaks. These six peaks were observed for each layers. The peaks were at the same positions with Ag{220} pole peaks, indicating epitaxial growth between each layers. Finally, the orientation relationships of the epitaxial growth on Si (111) can be

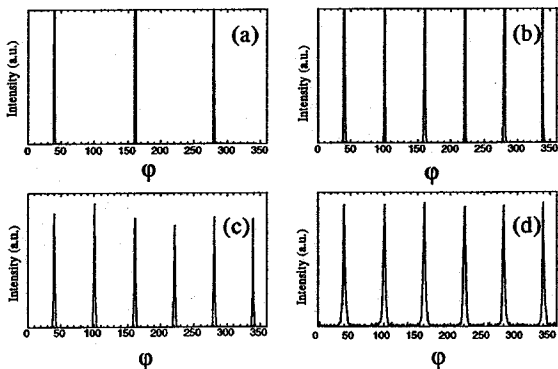


Fig. 3.(a) Si{110}-pole, (b) Ag{220}-pole, (c) Cu{220}-pole, and (d) Co{220}-pole  $\phi$ -scan spectra of a Co (50nm)/Cu (50nm)/Ag(75nm)/Si(111)-HF film.

determined as:

$$\text{Co (111) } [11\bar{2}] \parallel \text{Cu (111) } [11\bar{2}] \parallel \text{Ag (111) } [11\bar{2}] \parallel \text{Si (111) } [11\bar{2}].$$

Further measurements showed that, for samples without the Cu layer, the epitaxial relationships are the same with those involving Cu layers.

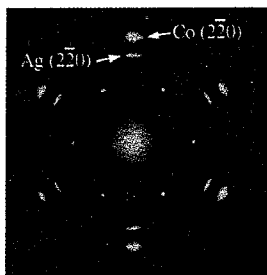
The same technique was employed to characterize the epitaxial growth of the Co films grown on Si (110) and Si (100) substrates. The epitaxial relationships were determined to be:

$$\begin{aligned} &\text{Co (100) } [001] \parallel \text{Cu (100) } [001] \parallel \text{Ag (100) } [001] \parallel \text{Si (100) } [001], \\ &\text{Co (110) } [001] \parallel \text{Cu (110) } [001] \parallel \text{Ag (110) } [001] \parallel \text{Si (110) } [001]. \end{aligned}$$

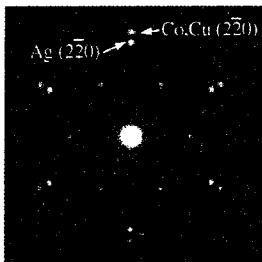
To further confirm the epitaxial nature of the Co films, TEM was employed. Figure 4(a) shows a TEM selected area diffraction (SAD) pattern of the (111) zone axes of the Co and Ag layers from a Co (50nm) / Ag (75nm) / HF-etched Si(111) sample. Epitaxial growth is clearly indicated. Fig. 4(b) shows the corresponding SAD pattern of the (111) zone axes of the Co, Cu and Ag layers of a Co (50nm) / Cu (50nm) / Ag (75nm) / HF-etched Si(111) sample. The reflections from the Cu and Co nearly overlap due to their closely matched lattice parameters. The high quality epitaxial relationship of Co (111) [220]  $\parallel$  Cu (111) [220]  $\parallel$  Ag (111) [220] is evident from this diffraction pattern.

### Magnetic Properties

To investigate the magnetic anisotropy of the FCC Co phase, in-plane torque measurements were performed on samples prepared on each of the three substrate orientations. Due to the crystal symmetry, it can be shown that the torque due to the first order anisotropy energy constant,  $K_1$ , vanishes for cubic (111) textured films. Furthermore, due to the second order anisotropy constant,  $K_2$ , the torque curves have a six fold symmetry with an amplitude of only  $K_2/18$ . For our (111) textured thin films we observed no torque distinguishable from the background noise. Figure 5(a) shows the torque curve measured within the film plane of a



(a)



(b)

Fig. 4 TEM Selected area diffraction pattern (SADP) of the (111) zone axes of the (a) Co, Ag layers for a Co / Ag / HF etched Si(111) sample, (b) Co, Cu, Ag layers for a sample with structure of Co / Cu / Ag / HF etched Si(111).

sample with (100) orientation. For this orientation there is no torque contribution due to  $K_2$ . Hence, a nearly single harmonic shape with  $90^\circ$  period was observed. This is consistent with the theoretical result for a cubic single crystal material, where the torque is calculated to be [6]:

$$L = -(K_1/2) \sin 4(\theta - 45^\circ) \quad (1)$$

and  $\theta$  is the angle from a [110] direction. For our preparation conditions we obtained  $K_1 = -2.3 \times 10^5$  erg/cc. The negative value indicates the easy axes to be along the  $\langle 111 \rangle$  directions. Figure 5(b) shows the measured in-plane torque curve for a (110) oriented Co film. Here both  $K_1$  and  $K_2$  should play a significant role in determining the torque. The general shape of this curve can be simulated by choosing the ratio of  $K_2/K_1$  to be 2.2. However, when noting the value for  $K_1$  from the (001) oriented sample this seems like an unreasonably large value for  $K_2$ . From other studies [5] of uni-crystalline hcp Co grown on (110) Si templates it was observed that the first order anisotropy energy can be degraded considerably via processing conditions. Hence, if the films prepared on these three substrate orientations may suffer from different degrees of strain or lattice defects it is possible that the results obtained may differ for the different orientations. While our results were very consistent for a number of films prepared under similar conditions we wish to explore greater variations in preparation conditions.

Figure 6 shows the BH loop tracer hysteresis loops for two typical samples with (111) substrate orientation and compositional structures of Co(50nm)/Ag(75nm)/Si(111) and Co(50nm)/Cu(50nm)/Ag(75nm)/Si(111). These soft magnetic properties are for pure Co samples deposited both at room temperature (solid line) and  $260^\circ\text{C}$  (dashed line). Since no magnetic field was intentionally applied during the deposition process there was no angular dependence to that hysteretic properties. This was consistent with the lack of torque for this orientation. Coercivities for the other two orientations were slightly higher and as a function of angle the loops possessed properties corresponding to the observed torque data. Co alloy films with compositions of CoCr<sub>20</sub>Pt<sub>4</sub> and CoPt<sub>8</sub> were also prepared on similar substrate orientations via the same technique. Even though similar alloy compositions are commonly used for hard disk recording these films were also found to be the FCC phase as opposed to the hcp phase used for hard disk media. Here the coercivities were found to be greater than for the pure Co films, but were limited to approximately 200 Oe. For the high Cr content films the coercivity increases were found to be consistent with the lower magnetization values. Also, because of the long-range order of the epitaxial growth on the Si substrates the concept of grain boundary exchange

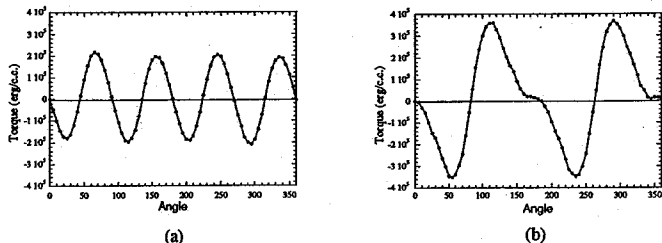


Fig. 5 Measured in-plane torque curves for (a) (100) oriented Co film; (b) (110) oriented Co film.

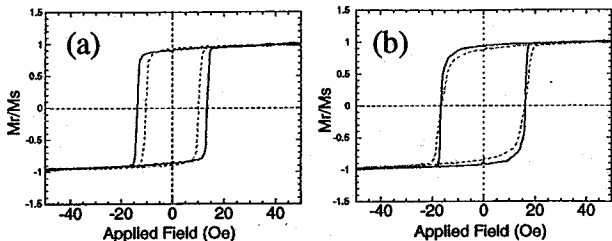


Fig. 6 Measured hysteresis loops for samples with structures of (a) Co(50nm) / Ag (75nm) / HF-Si(111); (b) Co (50nm) / Cu (50nm) / Ag (75nm) / HF-Si(111). The solid line is for samples deposited at room temperature, while samples deposited at 260°C are represented by dashed lines.

decoupling at grain boundaries due to chemical segregation is not applicable. This may enable considerable domain wall mobility and lower the coercivity. Further work on Co-alloys is in progress.

## SUMMARY

In this study, highly oriented Co and Co alloy thin films were epitaxially grown on single crystal Si substrates. The epitaxial relationships were determined by x-ray diffraction, x-ray pole figure  $\phi$  scans and TEM. The magnetic properties were determined and for the different substrate orientations significant differences were observed for the in-plane torque curves. Pure Co samples with (111) orientation showed nearly isotropic magnetic properties within the film plane and were found to possess reasonably soft magnetic properties.

## REFERENCES

- [1] D.N. Lambeth et al., *Mat. Rec. Soc. Symp. Proc.*, Vol. 517, 181(1998).
- [2] H. Gong, M. Rao, D.E. Laughlin, and D.N. Lambeth, *J. Appl. Phys.* 85, 5750(1999).
- [3] H. Gong, M. Rao, D.E. Laughlin, and D.N. Lambeth, *J. Appl. Phys.* 85, 4699(1999).
- [4] W. Yang, D.N. Lambeth, L. Tang, and D.E. Laughlin, *J. Appl. Phys.* 81, 4370(1997).
- [5] W. Yang, D.N. Lambeth, and D.E. Laughlin, *J. Appl. Phys.* 85, 4723(1999).
- [6] For example, see B.D. Cullity 'Introduction to Magnetic Materials' Page 221, Addison-Wesley Publishing Company, Inc. (1972).

## ACKNOWLEDGEMENT

This work is supported by the National Science Foundation under grant No. ECD-8907068. The U.S. government has certain rights in this material.