Stress dependence of soft, high moment and nanocrystalline FeCoB films

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Soft, high moment materials are crucial for the magnetic data recording industry in applications such as write poles and soft underlayers. Ever increasing areal densities have pushed the requirements for materials exhibiting saturation magnetizations above 2 T. In this work we investigate nanocrystalline FeCoB which exhibits a saturation magnetization of ~ 2.2 T. FeCoB exhibits a magnetostriction of $> 50 \times 10^{-6}$ which suggests that the magnetoelastic anisotropy is important. Nanocrystalline FeCoB films are rf diode sputtered as a function of Ar pressure. As the pressure is increased, film stress increases from compressive to tensile with a critical pressure of ~ 12 mTorr. TEM and XRD show that pressure has no significant effect on microstructure, however, at pressures above a critical pressure, the FeCoB films change from magnetically isotropic to magnetically uniaxial with low coercivity. This is due entirely to the magnetoelastic anisotropy which must be considered as the industry begins to utilize magnetic alloys with nonzero magnetostriction. © 2002 American Institute of Physics. [DOI: 10.1063/1.1449454]

INTRODUCTION

Novel high moment, soft magnetic materials are a critical enabling technology for the magnetic data storage industry to continue extending areal recording density and data rate. Such materials are employed both as write poles for heads and as soft underlayers for perpendicular media. As the industry moves away from the traditional NiFe alloys to the high moment Fe-based alloys, it is important to understand that these materials exhibit finite and often large values of magnetostriction. It is therefore imperative to develop processes for sputtering high moment alloys which take into account the magnetoelastic anisotropy and, hence, the sign and magnitude of both the film stress and magnetostriction.

Previous magnetic and microstructural studies have been done on the addition of boron to an $\text{Fe}_{65}\text{Co}_{35}$ sputtered film deposited onto an amorphous SiO_2 layer on a Si substrate.¹ The work focused on $(\text{Fe}_{65}\text{Co}_{35})_{90}\text{B}_{10}$ which achieves soft magnetic properties via an amorphous transition and exhibits a moment of 2–2.1 T. In contrast to the amorphous $(\text{Fe}_{65}\text{Co}_{35})_{90}\text{B}_{10}$, the current work will focus on nanocrystalline $(\text{Fe}_{65}\text{Co}_{35})_{90}\text{B}_{10}$ obtained by the use of a crystalline NiFeCr seed layer.

EXPERIMENT

FeCoB films were rf diode sputtered at room temperature from an FeCoB target with the composition $(Fe_{65}Co_{35})_{90}B_{10}$. The films were deposited using a NiFeCr buffer layer and a NiFeCr cap which was deposited via dc magnetron from a NiFeCr target. NiFeCr\FeCoB\NiFeCr structures were deposited on oxidized Si (100) substrates. Both NiFeCr and FeCoB were deposited using UHP Ar as the process gas with NiFeCr being deposited at 4 mTorr and the FeCoB being deposited in the range from 3 to 20 mTorr. With the exception of the process pressure, all other process parameters, including process time, were held constant during the deposition of the FeCoB. This resulted in FeCoB films which ranged in thickness from 1850 to 2250 Å. Magnetic hysteresis measurements were made on an SHB model 109 BH loop tracer. Magnetostriction constants were evaluated in a 50 Oe field using a Lafouda tester. Film stress was measured in a KLA Tencor model FLX-2908 thin film stress measurement system. Structural measurements were performed utilizing a Philip's X'Pert Pro XRD with a Cu source and x-ray mirror as well as a JEM-2000EX II TEM.

RESULTS/DISCUSSION

It was found by high angle XRD that when (Fe₆₅Co₃₅)₉₀B₁₀ was deposited onto a crystalline NiFeCr seed layer that the FeCoB film had a large (110) bcc peak. This is contrary to FeCoB of the same composition deposited onto an amorphous Si oxide layer.¹ Apparently the crystalline NiFeCr helps to nucleate the crystalline FeCoB phase onto it. The (110) peak shifts to smaller *d*-spacings with increasing sputtering pressure as observed in Fig. 1. The reasoning for this peak shift will be discussed in more detail later in the paper. The bcc FeCoB phase is strongly oriented with the (110) planes parallel to the surface and the full width at half maximum (FWHM) of the rocking curves are shown in Fig. 1. Although there is not a strong dependence of the (110)fiber texture on sputtering pressure, it is observed that with increasing pressure there is a slight decrease and then a very gradual increase of the FWHM of the rocking curves. Additionally, the FWHM of the $2\theta/\theta$ (110) scans are also plotted in Fig. 1. The broadening of this peak would be a signature of a decrease in grain size or an increase in the distribution of *d*-spacings both of which are indicative of a transformation from crystalline to amorphous. However, there seems to be very little variation of the broadening with sputtering pres-

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FIG. 1. (110) *d*-spacing, (110) $2\theta/\theta$ peak's FWHM and (110) rocking curve's FWHM as a function of sputtering pressure.

sure which points to the pressure not having a strong influence on the crystalline to amorphous transition of FeCoB.

Transmission electron microscopy (TEM) was done on a sample sputtered at 3 mTorr and one sputtered at 13 mTorr. Very little difference in the microstructures was observed. In Fig. 2 is shown a bright field image of the 13 mTorr sample for discussion. A peculiar contrast of elongated needles a few nanometers in diameter is observed and in certain regions neighboring needles show similar contrasts indicating similar orientations. The SAD patterns inset in Fig. 2 confirmed the strong (110) fiber texture that was observed via XRD. Additionally, a very weak and broad ring is observed near the d-spacing of (110) in regions where the strong (110) fiber texture causes crystalline spots to be absent. Presumably this is from some amorphous material. Dark field imaging was done using this amorphous ring, which gave uniform contrast across the thickness of the film and some stronger contrast in the regions between the needles discussed above. It is believed that amorphous material is separating the crystalline needles.

BH looper and VSM measurements verify that the films in this study exhibit a saturation magnetization of ~2.2 T. Figure 3 shows easy axis coercivity (H_c), uniaxial anisotropy (H_K), magnetostriction at 50 Oe (λ_{50}), and film stress vs process pressure for FeCoB. Figure 3 clearly shows that as the process pressure is increased from ~4 to ~20 mTorr, the film stress increases from a compressive ~-1200 MPa to a tensile ~+850 MPa passing through 0 stress at ~12 mTorr. This behavior is typical of most sputter deposited



FIG. 2. Bright field TEM image of FeCoB sputtered at 13 mTorr.



FIG. 3. Easy axis coercivity (H_C) , uniaxial anisotropy (H_K) , magnetostriction at 50 Oe (λ_{50}), and film stress vs process pressure for as-deposited FeCoB films.

films.² Figure 3 also shows that λ_{50} is positive and increases as a function of pressure. It is important to remember that this is magnetostriction at 50 Oe and not the saturation magnetostriction. The fact that λ_{50} has not become a constant indicates that the FeCoB is not saturated at 50 Oe at any of the sputtering pressures shown. We have measured, however, a λ_{50} that is large and positive (>50×10⁻⁶), which means that for FeCoB, the film stress must be tensile in order for the magnetoelastic anisotropy to be positive which forces the magnetization to lie in-plane. Figure 3 shows that as soon as the film stress becomes tensile, the films become soft and uniaxial.

A clearer way to look at this is shown in Fig. 4 which shows H_C , H_K , λ_{50} , and dispersion 50 vs film stress. Figure 4 clearly shows that FeCoB becomes soft and uniaxial only when the film stress and, hence, the magetoelastic anisotropy are positive. Remember that TEM showed no significant microstructural difference between FeCoB films sputtered at low, medium, and high pressures. XRD showed that the only difference between FeCoB films sputtered at various pressures was in the *d* spacing of the (110) planes. This can be explained by the changing film stress. As the sputtering pressure increases, in-plane film stress becomes more tensile re-



FIG. 4. Easy axis coercivity (H_c) , uniaxial anisotropy (H_K) , magnetostriction at 50 Oe (λ_{50}), and dispersion50 (disp50) vs film stress for as-deposited FeCoB thin films.

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sulting in a Poisson's ratio "contraction" in the *d*-spacings normal to the plane of the film which is what is measured by high angle XRD. Thus, the transition of FeCoB from an magnetically isotropic and high coercivity film to an uniaxial and low coercivity film is due entirely to the magnetoelastic anisotropy. H_C also appears to indicate that there is an optimum tensile stress where H_C is a minimum. This is shown better by the measured magnetic dispersion plotted in Fig. 4. The figure indicates that the soft magnetic properties are optimum in the range of ~200–500 MPa and begin to deteriorate at stresses higher than ~500 MPa again due to the magnetoelastic anisotropy.

CONCLUSIONS

In this work, we have highlighted several aspects of soft, nanocrystalline, high moment FeCoB. FeCoB compositions which would normally be amorphous, are nanocrystalline when deposited onto a NiFeCr seed layer. The fact that the nanocrystalline FeCoB can be made magnetically soft like the amorphous FeCoB suggests that the nanocrystalline FeCoB exhibits a grain size which is smaller than the exchange length of the material. Increasing the process pressure has no significant impact on microstructure as shown by XRD and TEM but significantly changes the film stress from compressive at low process pressures to tensile at high process pressures with the critical pressure at ~ 12 mTorr. Below the critical pressure, film stress is compressive and the films are magnetically isotropic. Once this critical pressure is exceeded and the films are in a state of tension, the nanocrystalline FeCoB exhibits soft and uniaxial magnetic properties. This uniaxial behavior is due entirely to the magnetoelastic anisotropy of the FeCoB which is a critical parameter to control when using magnetic materials which have large values of magnetostriction such as is the case with FeCoB.

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