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**Stability of Coupling in Exchange Spring and Exchange Bias Systems****RECEIVED****DEC 08 2000****OSTI**

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**Abstract**

We have studied the magnetic stability in exchange bias and exchange spring systems prepared via epitaxial sputter deposition. The two interfacial exchange coupled systems, Fe/Cr(211) double superlattices consisting of a ferromagnetic and an antiferromagnetic Fe/Cr superlattice that are exchange coupled through a Cr spacer, and Sm-Co/Fe exchange-spring bilayer structures with ferromagnetically coupled hard Sm-Co layer and soft Fe layer, were epitaxially grown on suitably prepared Cr buffer layers to give rise to different microstructure and magnetic anisotropy. The magnetic stability was investigated using the magneto-optic Kerr effect during repeated reversal of the soft layer magnetization by field cycling up to  $10^7$  times. For uniaxial Fe/Cr exchange biased double superlattices and exchange spring bilayers with uniaxial Sm-Co, small but rapid initial decay in the exchange bias field  $H_E$  and in the remanent magnetization is observed. However, the exchange spring bilayers with biaxial and random in-plane anisotropy in the Sm-Co layer shows gradual decay in  $H_E$  and without large reduction of the magnetization. The different decay behaviors are attributed to the different microstructure and spin configuration of the pinning layers.

The exchange coupling between magnetic materials often provides the foundation for controlling magnetic devices or for creating new magnetic materials. For example, the *exchange bias* effect that occurs at the interface between a ferromagnet (F) and an antiferromagnet (AF) pinning layer is being used to stabilize and bias magnetoresistive (both AMR<sup>1</sup> and GMR<sup>2</sup>) heads for high-density magnetic recording, and the *exchange-spring* magnet nanostructures consisting of exchange coupled soft ferromagnets pinned to hard ferromagnetic phases are candidates for the next generation of high-energy-product permanent magnet materials.<sup>3,4</sup> The robustness of interfacial exchange coupling will undoubtedly affect the viability of various device and materials engineering schemes. Instabilities have recently been reported in tunneling magnetoresistance (TMR) junctions of the hard reference layer against magnetization decay during field cycling of the adjacent soft layer;<sup>5</sup> and even though permanent magnets are typically operated at a point to maximize the energy product, in a dynamic application the magnet might experience a strong demagnetizing field which could cause a catastrophic collapse of the magnetic induction.<sup>6</sup>

In this paper we present a comparison of the magnetic stability of a novel exchange bias structure and exchange spring heterostructures after multiple switching cycles. Our exchange bias system is an Fe/Cr 'double superlattice' consisting of a ferromagnetic Fe/Cr superlattice and an antiferromagnetic superlattice coupled through a Cr spacer.<sup>7</sup> Fe/Cr double superlattices behave like conventional exchange bias systems, but with more AF/F coherent interface and controllable coupling strength. For the exchange spring system we use Sm-Co/Fe bilayer structures grown epitaxially on

different substrates to yield uniaxial, biaxial and random in-plane anisotropies. The epitaxially grown Sm-Co films have coercivity values of up to 4 T at room temperature. The switching field and remanent magnetization of both systems were measured by the magneto-optic Kerr effect during repeated reversals of the soft layer magnetization by field cycling. The magnetic stability after  $10^7$  cycles is reported and the effects of microstructure and interfacial spin configuration of the pinning layers are discussed.

The Fe/Cr double superlattice and the Sm-Co/Fe exchange spring bilayers were grown epitaxially by dc magnetron sputtering. In all cases, Cr buffer layers 200-Å thick were deposited under suitable conditions to provide appropriate growth templates. For the Fe/Cr double superlattice, the Cr buffer layer was deposited onto a single-crystal MgO(110) substrate at 400 °C in an Ar pressure of 4 mTorr to achieve the (211)-oriented epitaxial growth. The substrate was then cooled to 100°C and the double-superlattices were grown in 2 mTorr of Ar as  $[\text{Fe}(14 \text{ Å})/\text{Cr}(11 \text{ Å})]_{20}/\text{Cr}(20 \text{ Å})/[\text{Fe}(50 \text{ Å})/\text{Cr}(20 \text{ Å})]_5$  with a 50-Å Cr capping layer. The 11-Å Cr and 20-Å Cr give rise to antiferromagnetic and ferromagnetic coupled Fe/Cr superlattices, respectively; and the 20-Å Cr between the two superlattices provides ferromagnetic inter-superlattice coupling. For the exchange spring bilayers, (100)-, (211)-oriented, and (100)-textured but in-plane random Cr buffer layers were deposited onto single-crystal MgO(100), (110) and glass substrates, respectively. After depositing the Cr buffer layer, the Sm-Co layers were epitaxially grown at 400 °C, followed by the deposition of an 800-Å Fe layer and a 100 Å Cr capping layer. The thickness of the Sm-Co layer was varied from 50 to 800 Å.

The exchange spring behavior of the Sm-Co/Fe bilayers and the exchange bias characteristics of the Fe/Cr double-superlattice were confirmed via minor loop

measurements using a Quantum Design SQUID magnetometer equipped with a 7-Tesla magnet and a magneto-optic Kerr effect (MOKE) setup with a 2-T magnet. The Sm-Co/Fe bilayers showed shifted minor loops and recoil curves that recovered full remanence; and the Fe/Cr double-superlattice had a exchange bias field of  $\sim 35$  Oe and loop width of  $\sim 10$  Oe. With the MOKE system, the magnetic stability after multiple field cycles was measured from the changes in switching field and intensity of the minor loops. The samples were initially magnetized to  $+7$  T along the easy axis direction and then the reverse field was cycled from 0 Oe to  $-260$  Oe for up to  $10^7$  times at about 66 Hz using an air-gap coil. No magnetizing field was applied during the measurements and field cycles. To make sure that changes in measured signal are due to repeated switching of the free layer and not to a natural aging, the time dependence of  $H_E$  and the magnetization were measured. The Sm-Co( $50 \text{ \AA}$ )/Fe( $800 \text{ \AA}$ ) film was kept in a field of  $-250$  Oe for more than two days after being magnetized at  $+7$  T. The remanent magnetization was measured periodically. To minimize the number of switching, only a few minor loops were measured. Even after  $2 \times 10^4$  seconds, which is comparable to the time needed to measure the switching stability to  $10^7$  cycles, no significant decay was found.

By using appropriate Cr buffers, we have prepared two exchange coupled systems with four different kinds of microstructure and magnetic anisotropy. The Cr(211) template yields an in-plane uniaxial anisotropy in Fe/Cr superlattices along the  $\text{MgO}[0\bar{1}1]$  direction<sup>8</sup>. The epitaxial Sm-Co films grown on Cr(211) and (100) buffers on single crystal MgO substrates are  $(1\bar{1}00)$ - and  $(11\bar{2}0)$ -oriented with in-plane uniaxial and biaxial anisotropies, respectively;<sup>9</sup> whereas the ones grown on (100)-textured Cr

buffer on glass are  $(11\bar{2}0)$ -oriented but with randomly distributed easy axes in the film plane, similar to the Pitsch-Schrader epitaxial relation in longitudinal recording media<sup>10</sup>.

Figure 1 shows the change in exchange bias with the number of field cycles  $N$  for an Fe/Cr double-superlattice and uniaxial, biaxial and random in-plane Sm-Co(800 Å)/Fe(800 Å) bilayers. The exchange bias  $H_E$  is defined as the shift of the minor loop from zero field.  $H_{E0}$  is obtained from the first loop, and  $\Delta H_E = (H_E - H_{E0})/H_{E0}$ . Two kinds of decay characteristics are seen. The two uniaxial systems—the Fe/Cr double superlattice and the uniaxial bilayer—both show a rapid initial decay in  $H_E$  and then stabilize. The decay is strong (~ 3%-9%) for the first 10 cycles. On the other hand, the biaxial and the in-plane random Sm-Co/Fe bilayers show a much slower decay in  $H_E$ , which does not stabilize even after  $10^7$  field cycles. In the following, we will focus on the uniaxial and the biaxial Sm-Co/Fe bilayers for the two kinds of decay behaviors.

The different decay behaviors in the uniaxial and the biaxial samples could be accounted for by noting the differences in microstructure and spin configurations at the interface of the pinning layers. High-resolution electron microscopy study<sup>11</sup> shows that, due to local departure from the nominal stoichiometry during growth, the uniaxial Sm-Co film has  $\text{SmCo}_3$ ,  $\text{Sm}_2\text{Co}_7$  and  $\text{SmCo}_5$  polytypoids with stacking faults parallel to the hcp basal plane. These different phases have different magnetic anisotropy and share a common easy axis. Upon field cycling, regions with lower magnetic anisotropy become reversed, giving rise to the rapid initial decay in  $H_E$ . However, the planar stacking faults provide effective domain-wall pinning which prevents further propagation of reversed domains, and  $H_E$  reaches saturation. The biaxial Sm-Co, on the other hand, has a bicrystalline microstructure with grains separated by incoherent boundaries. The grains

have their easy axes in either MgO [001] or [010] directions. The strong inter-granular coupling results in clusters of grains that have effective easy axes along either MgO [011] or MgO  $[0\bar{1}1]$  directions. It is likely that during repeated field cycling, the magnetization of some of the grains relax into their local preferred directions. Since this process involves only a  $45^\circ$  rotation, the decrease in the coupling with the Fe layer would be small. This is evidenced by the smaller change of  $H_E$  for the biaxial samples than for the uniaxial samples. The high density of twin boundaries could account for the very slow decay processes.

Figure 2 shows the decay behavior of the uniaxial and biaxial exchange spring samples with different Sm-Co thicknesses. For the uniaxial samples, the one with 800 Å Sm-Co shows strongest initial decay of  $H_E$  and the rate of decay decreases with decreasing Sm-Co thickness. Thicker Sm-Co layer has larger roughness due to the island growth mode<sup>9</sup>. The increased inhomogeneity in the coupling with the Fe layer for thicker SmCo layers could result in a stronger initial decay in  $H_E$ . On the other hand, the decay of the nucleation field  $H_N$  in the biaxial samples is less sensitive to the Sm-Co layer thickness. This again is consistent with the fact that the density of twin boundaries in biaxial Sm-Co is thickness-independent.

Unlike the other uniaxial exchange spring samples with thicker SmCo layers, the bias field of a bilayer sample with 50 Å Sm-Co decreases linearly with the number cycles. To understand this behavior, we show in Figure 3 the minor loops of the bilayer film after 1,  $10^6$ , and  $10^7$  field cycles. It is seen that not only does the magnitude of the magnetization decrease, the squareness of the loop decreases as well. Since the MOKE signal is dominated by the top Fe layer which should remain constant if the Fe layer is



saturated at  $-80$  Oe and  $-180$  Oe. The decrease is due to the gradual demagnetization of the thin Sm-Co layer which causes the Fe magnetization to rotate away from the initial bias direction.<sup>12</sup> Since the SmCo thickness,  $50\text{\AA}$ , is smaller than the thickness of a domain wall, the Sm-Co would be susceptible to irreversible switching by reversal of the coupled Fe layer.<sup>5</sup>

#### 4. SUMMARY

The magnetic stability of two novel interfacial exchange coupled systems was investigated. For Fe/Cr double-superlattice and exchange spring bilayers with uniaxial Sm-Co, the exchange bias field  $H_E$  shows strong initial decay and then becoming stable as in a training effect. However, for the biaxial samples,  $H_E$  decays gradually and does not reach saturation. The different decay behaviors are attributed to the different microstructure and spin configuration of the pinning layers. The strong initial decay in uniaxial samples are due to the reversal of local inhomogeneities whereas the gradual decay in biaxial samples are due to slow relaxation metastable spin configuration.

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**FIGURE CAPTIONS**

- Fig. 1 The change in exchange bias  $\Delta H_E$  with numbers of field cycles  $N$  for an  $[\text{Fe}(14 \text{ \AA})/\text{Cr}(11 \text{ \AA})]_{20}/\text{Cr}(20 \text{ \AA})/[\text{Fe}(50 \text{ \AA})/\text{Cr}(20 \text{ \AA})]_5$  double superlattice and  $\text{Sm-Co}(800 \text{ \AA})/\text{Fe}(800 \text{ \AA})$  exchange spring bilayers with uniaxial, biaxial and random in-plane  $\text{SmCo}$  layer.
- Fig. 2 (a) The change in exchange bias field,  $\Delta H_E$ , with  $N$  for uniaxial  $\text{Sm-Co}/\text{Fe}(800 \text{ \AA})$  bilayers with different  $\text{Sm-Co}$  thicknesses. (b) The change in nucleation field,  $\Delta H_N$ , with  $N$  for biaxial  $\text{Sm-Co}/\text{Fe}(800 \text{ \AA})$  bilayers with different  $\text{Sm-Co}$  thicknesses.  $H_N$  is defined as the switching field in the demagnetization curve.
- Fig. 3 The minor hysteresis loops of the uniaxial  $\text{Sm-Co}(50 \text{ \AA})/\text{Fe}(800 \text{ \AA})$  bilayer after different number of field cycles.

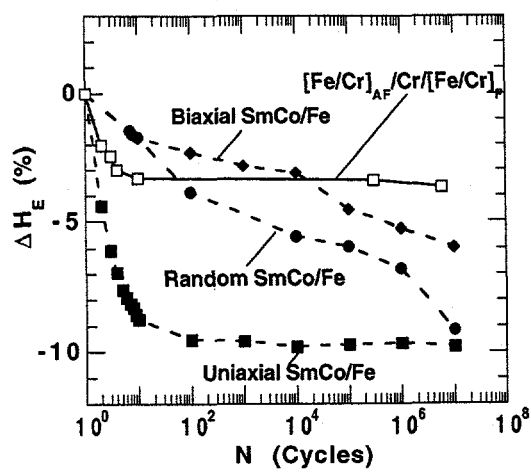


Fig. 1

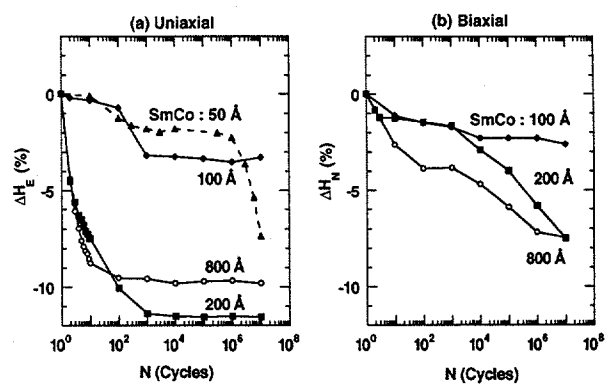


Fig. 2

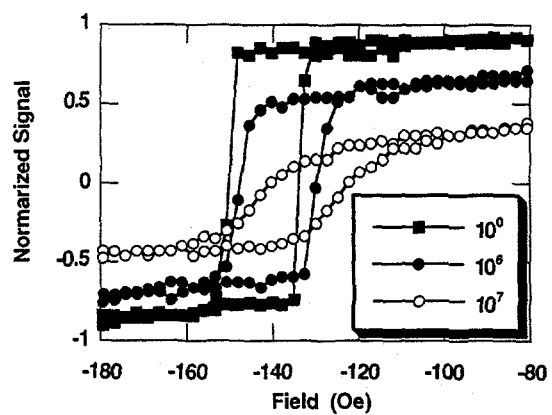


Fig. 3