Hindawi Publishing Corporation
Journal of Nanomaterials
Volume 2013, Article ID 718365, 9 pages
http://dx.doi.org/10.1155/2013/718365

Research Article

High Temperature Magnetic Properties of Indirect Exchange Spring FePt/M(Cu,C)/Fe Trilayer Thin Films

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Received 29 March 2013; Accepted 15 July 2013

Academic Editor: Takeshi Seki

We report the investigation of temperature dependent magnetic properties of FePt and FePt(30)/M(Cu,C)/Fe(5) trilayer thin films prepared by using magnetron sputtering technique at ambient temperature and postannealed at different temperatures. $L1_0$ ordering, hard magnetic properties, and thermal stability of FePt films are improved with increasing postannealing temperature. In FePt/M/Fe trilayer, the formation of interlayer exchange coupling between magnetic layers depends on interlayer materials and interface morphology. In FePt/C/Fe trilayer, when the C interlayer thickness was about 0.5 nm, a strong interlayer exchange coupling between hard and soft layers was achieved, and saturation magnetization was enhanced considerably after using interlayer exchange coupling with Fe. In addition, incoherent magnetization reversal process observed in FePt/Fe films changes into coherent switching process in FePt/C/Fe films giving rise to a single hysteresis loop. High temperature magnetic studies up to 573 K reveal that the effective reduction in the coercivity decreases largely from 34 Oe/K for FePt/Fe film to 13 Oe/K for FePt/C(0.5)/Fe film demonstrating that the interlayer exchange coupling seems to be a promising approach to improve the stability of hard magnetic properties at high temperatures, which is suitable for high-performance magnets and thermally assisted magnetic recording media.

1. Introduction

The development of futuristic magnetic devices such as biasing nanomagnets and exchange coupled nanocomposite magnet in microelectromagnetic devices, and ultra-high-density magnetic storage media, strongly depends on progress of performance of hard magnetic thin films [1–3]. In order to improve the hard magnetic properties and to attain large thermal stability, single domain magnetic particles with high magnetic anisotropy energy are indispensable [4]. Hence, the single domain particles are of great interest, and extensive research work has been carried out recently to search for suitable thin films with magnetic alloys having large magnetic anisotropy energy from the standpoint of high density magnetic recording technology and permanent magnet applications [5–10]. It is well known that the permanent magnet, an essential component in modern technology, is an important magnetic material whose main application is to provide magnetic fluxes and to be used as a magnetic media. The key character is maximum high energy product, which is markedly dependent on remanent magnetization and coercivity (H_C). For instance, SmCo and NdFeB alloys with complex crystal structures are widely being used as permanent magnets with an idea of exchange coupling between the transition metal and rare earth atoms with strong uniaxial anisotropy [11, 12]. This provided a maximum energy of 55 MGOe for an NdFeB alloy, while its theoretical value is calculated to be 64 MGOe. Recently, $L1_0$ ordered equiatomic Fe(Co)Pt alloy is also considered as one of the most promising candidates for hard magnet applications because of its extremely high magnetic anisotropy energy ($K_u \sim 10^8$ erg/cc). Since high K_u materials can produce large H_C, attempts were shown to fabricate FePt based alloys in the form of chemically synthesized nanoparticles, isolated island particles, epitaxially grown single crystal films, and granular films [13–15]. Nevertheless, the maximum energy product in thin films is limited by M_S, and hence the permanent magnet with high-energy product is not available mainly because of a trade-off in obtaining large H_C and high M_S.

On the other hand, Coehoorn et al. [16] and Kneller and Hawig [17] proposed the exchange spring magnet, based on an interfacial exchange coupling of two nanostructured phases combining a hard magnetic material in order to provide large H_C and a soft magnetic material to provide high M_S, as a way to create a next generation high performance permanent magnet and magnetic recording media. Since then many investigations have been carried out using various hard (Sm-Co, FePt, and Co-Pt) and soft (Fe, Co, and Fe-Co, Fe_3Pt) ferromagnetic (FM) phases to achieve high energy product [15, 18–27]. It has been predicted theoretically that FePt based nanocomposite shows an energy product of 90 MGOe [28, 29]. However, a maximum energy product of 52.8 MGOe was achieved experimentally by Liu et al. [18] in FePt/Fe_3Pt nanocomposite thin film with rapid annealing process. A careful review on FePt films reveals that these films display large H_C (>30 kOe) in very thin island-like films and particulate films [5, 30, 31], but H_C drops down drastically when they become continuous [15, 32]. These results confirm that FePt films with high $L1_0$ ordering show very high H_C and the introduction of proper exchange coupling using soft magnets should improve the high energy product.

Up to now most of the attempts in exchange spring have been executed by coupling the soft FM phase directly to the hard FM phase and studied the effects of bilayer and multilayer structures and various heat treatment process on the improvement of properties of nanocomposite magnets. Recently, interlayer exchange coupling using two FM films, hard and soft magnets, separated by a thin nonmagnetic layer, resulting in an indirect exchange coupling between the FM layers, was reported to construct an exchange spring [33]. The advantages of such coupling are (i) coherent interface is not necessary, and (ii) interface diffusion may not have serious effects on the exchange coupling. Besides, to use as a desirable magnet and for the application of high density magnetic recording media, both intrinsic (high M_S, high Curie temperature, and high K_u) and extrinsic (high energy product, high H_C, thermal stability, and corrosion stability) properties should be characterized. However, no systematic investigations on the indirect exchange spring have been reported. Since the fundamental magnetic properties of FePt films are very sensitive to chemical ordering and microstructure, thermal stability of FePt particles against temperature is also expected to show strong dependence on chemical ordering. Nevertheless, only a few reports have been published on the role of chemical ordering on the thermal stability and high temperature magnetic properties of FePt based single domain particles [34, 35] and their applications in indirect exchange spring nanocomposite magnets. Therefore, in this work, we have studied (i) the effect of chemical ordering on the high temperature magnetic properties of FePt polycrystalline films fabricated on low cost substrates, (ii) the formation of interlayer exchange coupling in indirect exchange spring, and (iii) their effects on the stability of magnetic properties at high temperature, by fabricating FePt(30 nm)/M(x nm)/Fe(5 nm) nanocomposite thin films with Cu and C as a nonmagnetic layer for the first time.

2. Materials and Methods

FePt films with various thicknesses ranging from 10 to 50 nm were prepared by sputtering a high purity Fe target with Pt pellets on it on low cost substrates. This gives a homogenous single layer FePt film, and composition of FePt was optimized by adjusting number of Pt pellets on Fe target. Thermally oxidized Si and Si(111) wafers were used as substrates. All the films were deposited at ambient temperature and postannealed at high temperatures (450 and 550°C) for 45 minutes under a high vacuum to induce face centered tetragonal (*fct*) structure. The base pressure of the chamber was better than 3×10^{-5} Pa and the high purity argon of 10 mTorr for FePt and Fe, 20 mTorr for C and 8 mTorr for Cu was flown during the sputtering. A series of FePt(30 nm)/M(x nm)/Fe(5 nm) (M = Cu and C, and x = 0, 0.25, 0.5, 1, 2, and 4 nm) trilayer films were deposited by sputtering FePt, M, and Fe targets at ambient temperature and postannealed at 550°C. The nominal thicknesses of the FePt, M, and Fe films were controlled based on precalibrated sputtering rates of the FePt, M, and Fe, respectively. Crystal structure and film structure were examined by X-ray diffraction (XRD) using a Rigaku TTRAX diffractometer with Cu-K_α radiation (λ = 1.5405 Å) and transmission electron microscopy (TEM). Compositions, estimated using energy dispersive X-ray spectroscopy attached to a scanning electron microscope (SEM, Leo 1430VP), were about $Fe_{49}Pt_{51}$ for all the films. Room temperature and temperature dependent magnetic properties were analyzed by using vibrating sample magnetometer (VSM, Model: Lakeshore 7410, USA) in the temperature range 300 K to 800 K by measuring magnetic hysteresis (M-H) loops at different temperatures.

3. Results and Discussion

3.1. Magnetic Properties of FePt Films. To optimize the magnetic properties and to study the thermal stability of single layer FePt films, M-H loops were measured at room temperature and at high temperatures. Figure 1 depicts the variations of $H_C(T)$ for FePt films with 30 and 50 nm thicknesses grown on oxidized Si substrate at ambient temperature and annealed at two different temperatures (450 and 550°C). $(M\text{-}H)_T$ loops measured at different temperatures for FePt(50 nm) film annealed at 450°C are displayed in inset of Figure 1. It is clear from the figure that room temperature magnetization of FePt film obtained at 20 kOe field is around 530 emu/cc, which is quite low as compared to its bulk value (M_S-1140 emu/cc). While the exact mechanism responsible for the pronounced loss in saturation magnetization in FePt film is unclear, the observed results can be attributed to one of the following reasons: (i) the observation of nonsaturated hysteresis curve for the applied field of 20 kOe, (ii) size dependent chemical ordering in FePt films with different thicknesses [36–38], (iii) incomplete structural phase transition from face-centered cubic (*fcc*) structure in as-deposited film to *fct* structure in annealed films [39], (iv) reduced magnetization on the

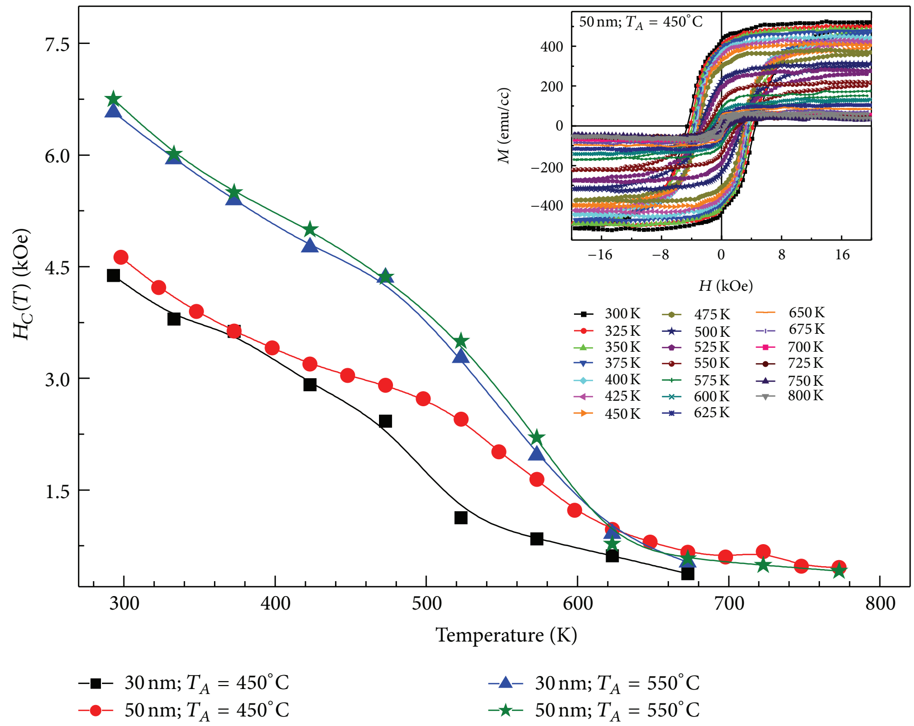

FIGURE 1: (Color online) Variations of coercivity with temperature in the temperature range 300 K and 800 K for the FePt films prepared on thermally oxidized Si substrates at ambient temperature and postannealed at different temperatures. Inset: Typical M-H loops obtained at different temperatures between 300 K and 800 K for FePt(50 nm) film annealed at 450°C.

surfaces [38], and (v) the formation of magnetically deadlayer at the substrate film interface or possible oxidation of the FePt surface during post annealing process, resulting an overall reduction in FePt film thickness [40]. Nevertheless, the observed room temperature magnetization in the presently investigated films is in agreement with the earlier reports ($M_S \sim 610$ emu/cc) on similar system (FePt(50 nm)) [39]. In addition, H_C of FePt films at room temperature shows a strong dependence of annealing temperature, which can be attributed to an increase in chemical ordering of $L1_0$ phase. The improvement in the chemical ordering was also confirmed from structural analysis (not shown here) that (i) the ratio of integrated intensities between the superlattice (001) peak and the fundamental (002) peak increases and (ii) the peak position of the (001) superlattice peak ((002) fundamental peak) shifts slightly to higher angles with increasing post annealing temperature. Another feature observed from Figure 1 is that $H_C(T)$ shows a substantial thickness dependent for the samples annealed at low temperature, while the samples annealed at high temperature follow a similar trend for different film thicknesses. This can be attributed to size dependent chemical ordering in FePt films with different thicknesses [36–38]. It is generally understood that H_C variation in FePt films is not only affected by the ordered volume fraction [41, 42], but also by a microstructural factor, that is related to the ordered volume fraction and that scales similarly. Defects, such as grain boundaries and phase boundaries, in the magnetic materials can form pinning sites, which impede the domain walls and leading to an enhancement in H_C. In addition, the boundaries between the ordered and disordered regions might also represent domain wall pinning sites and responsible for high H_C. In order to understand the effect of post annealing on the thermal stability of FePt films, $H_C(T)$ data are analyzed using Sharrock's equation [43–46], given as

$$H_r(t,T) = H_o\left\{1 - \left[\left(\frac{k_BT}{E_b}\right)\ln\left(\frac{f_o t}{\ln 2}\right)\right]^n\right\}, \qquad (1)$$

where H_r is remanent coercivity dependent upon field exposure time t and temperature T. H_o is intrinsic coercivity without thermal agitation, k_B is the Boltzmann's constant, f_o is attempt frequency, t is time of hysteresis measurement, and E_b is energy barrier at zero field. The value of n is taken as 2/3 by assuming slightly unaligned particles [47, 48]. The attempt frequency f_o and the hysteresis measurement time are taken as 10^{10} Hz and 1 second, respectively. Figure 2 depicts the applicability of Sharrock's equation on $H_C(T)$ data for FePt films. The fitting resulted the energy barrier values of 1.40 eV and 1.61 eV for FePt films of 30 and 50 nm annealed at 450°C, and 1.96 eV and 2.02 eV for FePt films of 30 and 50 nm annealed at 550°C, respectively. This reveals the corresponding thermal stability (the ratio between the energy barrier (E_b) to thermal energy (k_BT) at room temperature) of about 54 and 63 for FePt films of 30 and 50 nm annealed at 450°C and 76 and 78 for FePt films of 30 and 50 nm annealed at 550°C, respectively. These results confirm that the values of energy barrier increase considerably with the annealing

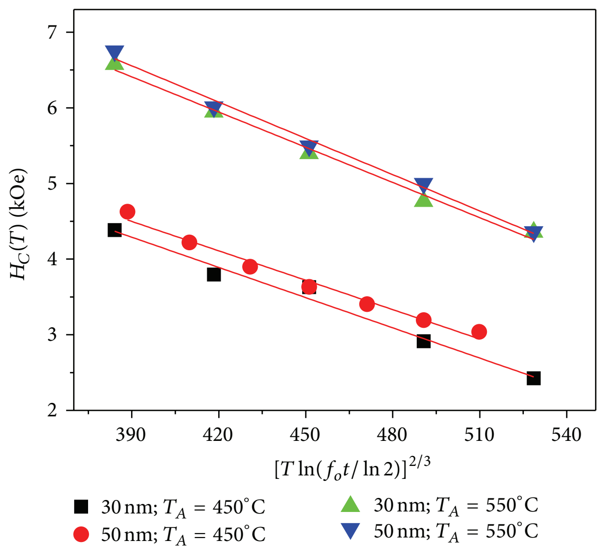

Figure 2: (Color online) Coercivity as a function of $[T\ln(f_o t/\ln 2)]^{2/3}$ for the FePt films annealed at different temperatures.

temperature. Since the FePt films annealed at 550°C show enhanced properties, we have taken FePt(30 nm) film for the study of indirect exchange spring by fabricating the FePt and Fe with different nonmagnetic interlayers.

3.2. FePt/M/Fe Trilayer System. To investigate the indirect exchange spring in FePt film and to study their temperature dependent magnetic properties and the thermal stability, FePt(30 nm)/M(x nm)/Fe(5 nm) trilayer films with x = 0, 0.25, 0.5, 1, 2, and 4 nm, and different intermediate layers using C and Cu were fabricated on Si(111) substrates. Figure 3 shows the XRD patterns of trilayer films with different Cu and C thicknesses deposited at ambient temperature and postannealed at 550°C. It is evident from the figure that superlattice diffraction line of (001) is observed around 2θ = 24°, and all the films are $L1_0$ ordered with *fct* structure after post annealing. The other unlabeled diffractions peaks are due to the Si substrate. The relative integrated intensities of (001) and (111) peaks reveal that the films have a weak preferred orientation to [001], that is, in the perpendicular direction to the film plane [15, 49]. Note that none of the XRD patterns in fact showed any diffraction peak of Fe layer or any information about the Fe and the nonmagnetic interlayers. Figure 4 shows the room temperature *M-H* loops of FePt(30)/M(x)/Fe(5) trilayer films. For the films with no nonmagnetic interlayer, *M-H* loop exhibits a clear kink in second quadrant due to an incoherent or separate magnetic reversal process suggesting that there is a weak FM coupling or no coupling between the hard and soft layers [22, 33]. With increasing Cu layer thickness, the kink observed in bilayer film reduces gradually up to 4 nm without a much change in M_S values. In addition, the magnetic field required for saturating the films (H_{sat}) and H_C decreases significantly with increasing the Cu layer thickness. On the other hand, the introduction of C interlayer interestingly exhibits different properties. (i) With increasing the C to 0.25 nm, the kink

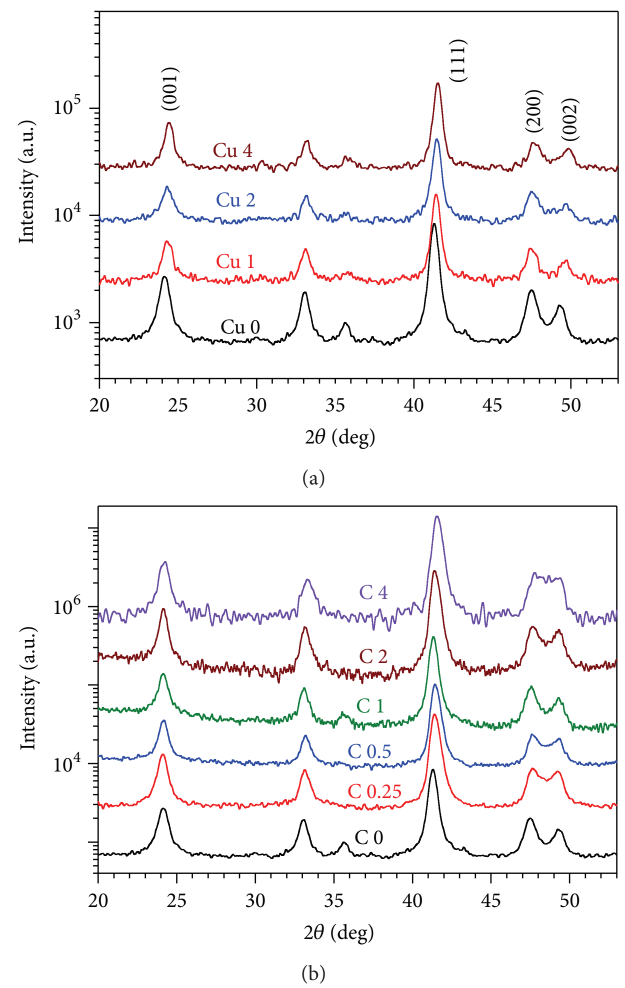

Figure 3: (Color online) XRD patterns for FePt(30 nm)/M(x nm)/Fe(5 nm) trilayer films with M-Cu (a) and C (b) and x = 0–4 prepared on Si(111) substrates at ambient temperature and postannealed at 550°C.

in the second quadrant slightly reduces, but H_{sat} decreases noticeably. (ii) When the C thickness is increased to 0.5 nm, the kink in the loop disappears, and *M-H* loop exhibits a single hysteresis with almost rectangular shape. This confirms a strong interlayer FM exchange coupling between the FePt and Fe layers through C interlayer. (iii) Remarkably, M_S value is increased to 990 emu/cc, which is much higher than that of pure FePt single layer films obtained in the present investigation. (iv) On further increasing the C layer thickness up to 4 nm, the shape of the *M-H* loop and values of M_S remain same, but the values of H_C and H_{sat} decrease gradually. To understand the effect of interlayer thickness on the magnetic parameters, the values of H_C, M_S, and H_{sat} were extracted from the loops and depicted in Figure 5 for FePt(30)/M(x)/Fe(5) trilayer films. It is observed that H_C and H_{sat} decrease largely for the initial increase in C thickness up to 1 nm and exhibits gradual decrease for the higher C thickness. On the other hand, M_S values increases quickly to 990 emu/cc for C thickness up to 1 nm and remains almost constant for larger C thicknesses. In the case of Cu

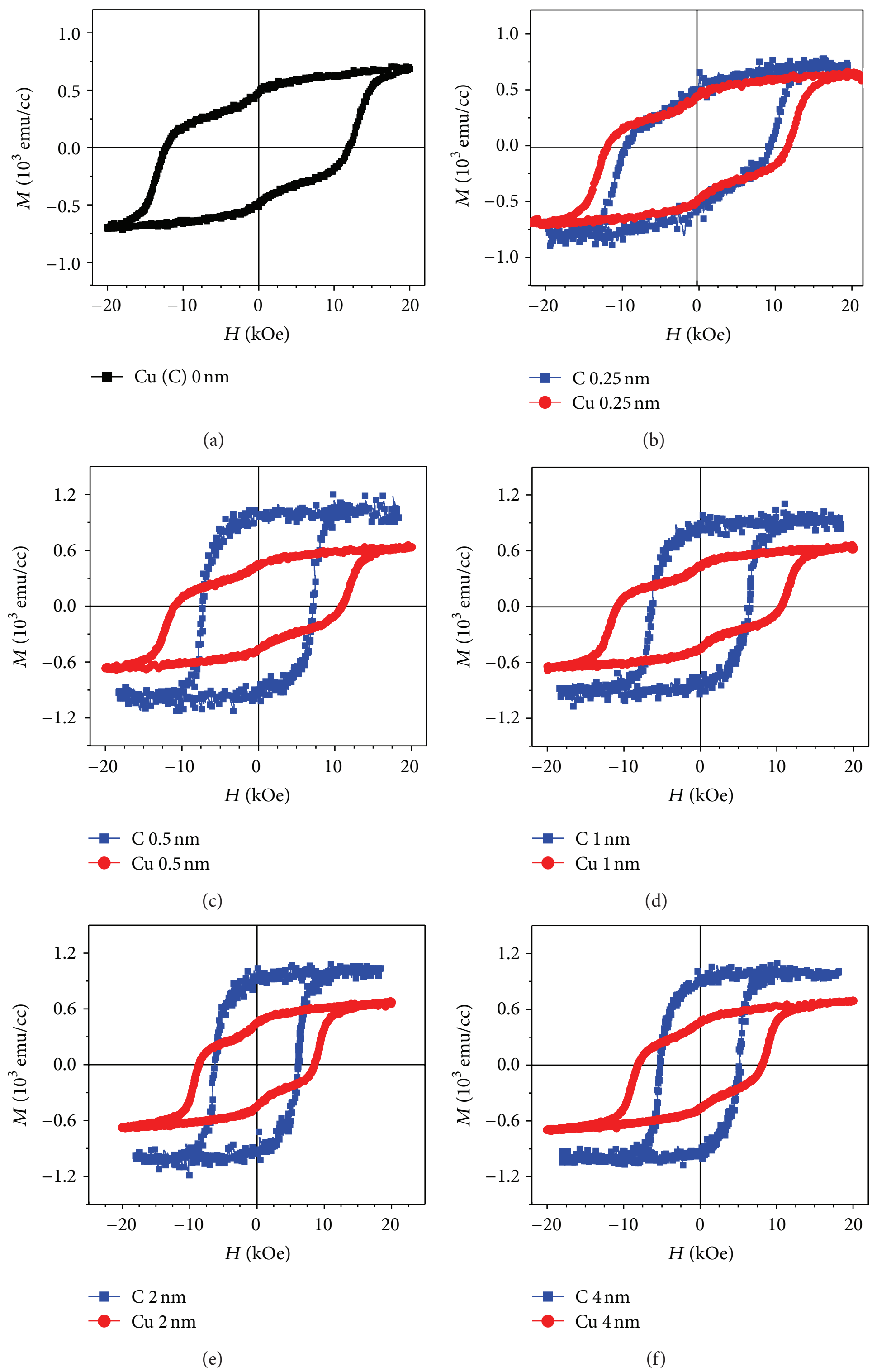

FIGURE 4: (Color online) Room temperature M-H loops of FePt(30 nm)/M(x nm)/Fe(5 nm) trilayer films with M-Cu and C and $x = 0$–4 nm prepared on Si(111) substrates at ambient temperature and postannealed at 550°C.

interlayer, both H_C and H_{sat} exhibit a slow decrease up to 1 nm and a larger change for higher thicknesses. In addition, M_S decreased initially up to 0.5 nm and started increasing with increasing the Cu thickness.

The observed results suggest that the interlayer FM exchange coupling between the FePt and Fe layers strongly depends on the type of materials used as interlayers and their thickness, and interface morphology [33, 50]. All the trilayer

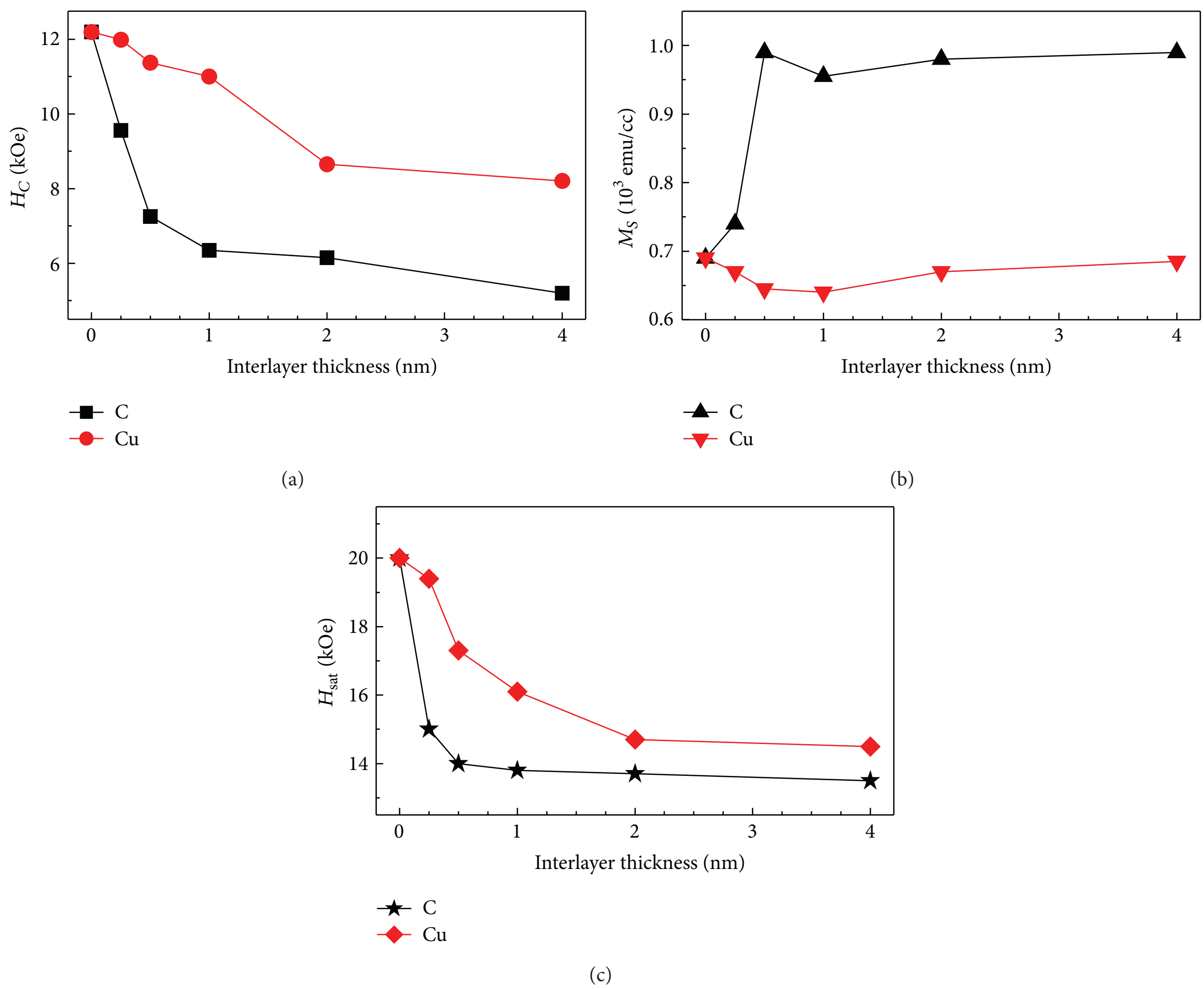

FIGURE 5: (Color online) Variations of (a) coercivity, (b) saturation magnetization, and (c) applied magnetic field required for saturating the films with different interlayer thicknesses of FePt(30 nm)/M(x nm)/Fe(5 nm) trilayer films with M-Cu and C and $x = 0$–4 nm prepared on Si(111) substrates at ambient temperature and postannealed at 550°C.

films were prepared at room temperature and postannealed at 550°C. It is known that the post annealing may cause a possible interdiffusion between the layers in multilayer films resulting a change in interface morphology [51]. Also, the growth morphology of FePt films with annealing may provide inhomogeneous interface morphology to the subsequent layers [50]. Since the trilayer films with Cu interlayer did not show a considerable change in the magnetic properties, this may be correlated to a possible inter-diffusion across the interlayers resulting an unclear interface between hard and soft layers [51], when the Cu thickness is less than 1 nm. Although the kink observed in the second quadrant weakens slowly due to a thick Cu interlayer at higher thickness, the formation of interlayer exchange coupling was not at all observed for Cu thickness up to 4 nm. On the other hand, the introduction of C between the hard and soft phases is expected to provide stable interfaces even after annealing [52]. Hence, the hard and soft phases are clearly separated by the C interlayer, which induces the interlayer exchange coupling resulting a coherent switching process and giving rise to a single hysteresis loop. The appearance of incoherent or separate magnetization reversal process in the bilayer films may be correlated to the growth morphology of FePt [50] or to the higher thickness of the soft layer [15, 22]. The interface analysis reported by Casoli et al. [50] on the FePt/Fe bilayer films reveals that (i) for homogeneous and pseudocontinuous FePt layers, Fe layers grow epitaxially on FePt resulting a strong exchange coupling, (ii) but the FePt film grown at high substrate temperature exhibits island-like morphology where the Fe shows a more disordered structure due to considerable amount of Fe grows directly on the substrate, and thus the system behaves as an incoherent reversal process for high Fe layer thickness. On the other hand, Rheem et al. [52] reported that the robust interlayer exchange coupling obtained in the FePt(30)/FeCo(10)/FePt(30) tri-layers is improved initially by introducing a thinner C/Ta interlayer, but eventually degraded at larger thicknesses. They also correlated that the degradation of the exchange coupling is mainly due to the formation of continuous TaC films between the hard and soft layers. Jiang et al. [33]

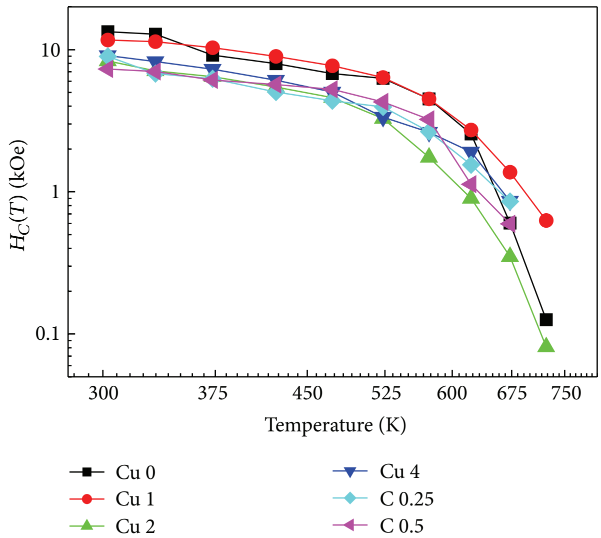

FIGURE 6: (Color online) Variations of coercivity with temperature for FePt(30 nm)/M(x nm)/Fe(5 nm) trilayer films with M-Cu (0, 1, 2, and 4) and C (0.25 and 0.5 nm) prepared on Si(111) substrates at ambient temperature and postannealed at 550°C.

reported the indirect exchange between FePt and Fe using Ru interlayer, where the introduction of a thin Ru layer improves the existence of weak FM coupling into strong FM exchange coupling. The observed results in the presently investigated FePt/C/Fe trilayers are in agreement with the results observed in FePt/Ru/Fe trilayer films [33]. However, it is important to mention that the formation of exchange coupling between FePt and Fe was observed even for the 4 nm thick C interlayer. This value is significantly larger than the value reported in FePt/Ru/Fe trilayer films. Herndon et al. [53] reported the effect of nonmagnetic (Al_2O_3) spacer layer thickness on magnetic interactions of self-assembled single domain iron nanoparticles separated by Al_2O_3 spacer layer, and the variation of observed magnetic properties has been attributed to three types of magnetic interactions: exchange, strong dipolar, and weak dipolar depending on the spacer layer thickness. This revealed a dominance of exchange type interaction between two ferromagnetic layers when the nonmagnetic spacer layer thickness is less than 6 nm. In order to form an interlayer exchange coupling [54], a ferromagnetic coupling between hard and soft layers with preferred orientation of magnetization without much interdiffusion across the interface is necessary [33]. Since the introduction of C provides a stable interface between FePt and Fe layers, the ferromagnetic coupling between them generates a possible interlayer exchange coupling even at 4 nm thick C interlayer. Nevertheless, a systematic investigation of interlayer thickness dependent exchange coupling over a wide range of interlayer thicknesses is required to obtain more insight of interlayer exchange coupling.

To understand the effect of interlayer exchange coupling between hard and soft phases on the $H_C(T)$ in FePt/M/Fe trilayers, $(M\text{-}H)_T$ loops were observed at different constant temperatures, and the extracted values of $H_C(T)$ are plotted as a function of temperature in Figure 6 for different Cu and C interlayers. $H_C(T)$ decreases gradually with increasing temperature up to 600 K as expected for a typical FM material. This is mainly due to the reduction in the magnetocrystalline anisotropy energy with increasing temperature. On further increasing the temperature above 600 K, $H_C(T)$ decreases largely due to instability of $L1_0$ ordered state, and finally they exhibit soft magnetic properties. In order to correlate the effect of interlayer exchange coupling on the magnetic properties, the effective reduction in $H_C(T)$ data for various interlayer thicknesses was analyzed carefully. It is revealed that in the temperature range between 300 K and 573 K, $H_C(T)$ decreases from 34 Oe/K for $x = 0$ to 25 Oe/K and 22 Oe/K for $x = 1$ and 2 nm, respectively, in FePt/Cu(x)/Fe films. Similarly, in FePt/C(x)/Fe films, H_C reduces from 34 Oe/K for $x = 0$ to 15 Oe/K and 13 Oe/K for $x = 0.25$ and 0.5 nm, respectively. The above results provide a clear evidence that the interlayer FM exchange coupling between the hard and soft phases through a nonmagnetic film plays a major control on the stability of the hard magnetic properties of FePt films at higher temperatures leading to a sluggish decrease in $H_C(T)$. However, a careful analysis of $H_C(T)$ study at different Fe layer thicknesses in FePt/C/Fe trilayer films would reveal a detailed information on the effects of interlayer exchange coupling and the role of different amount of soft phases on the FePt hard magnetic properties and their possible applications in permanent magnet and thermally assisted magnetic recording media.

4. Conclusion

We have investigated the high temperature magnetic properties of pure FePt thin films and indirect exchange spring FePt/M/Fe trilayer through various interlayers at different interlayer thicknesses. It is perceived that the interlayer exchange coupling between the hard and soft magnetic phases through the nonmagnetic layer strongly depends on the interlayer materials and their thicknesses. When the C interlayer thickness was about 0.5 nm, a strong interlayer exchange coupling between FePt and Fe has been realized, and the saturation magnetization was largely improved. High temperature magnetic properties of the FePt/C/Fe trilayer films suggest that the stability of the hard magnetic properties of the FePt films are improved by the interlayer exchange coupling, as compared to the bilayer FePt/Fe films. The observed results provide a promising way in making exchange spring in future for the applications in permanent magnet and thermally assisted magnetic recording media.

Acknowledgments

One of the authors, A. Gayen, acknowledges the financial support from DRDO, New Delhi, in the form of research fellowship for the Ph.D. program. This work was financially supported by the DRDO, New Delhi, through a research project (ERIP/ER/0900363/M/01/1185). Infrastructure facilities provided by DST, New Delhi (SR/S2/CMP-19/2006, SR/FST/PII-020/2009) are gratefully acknowledged.

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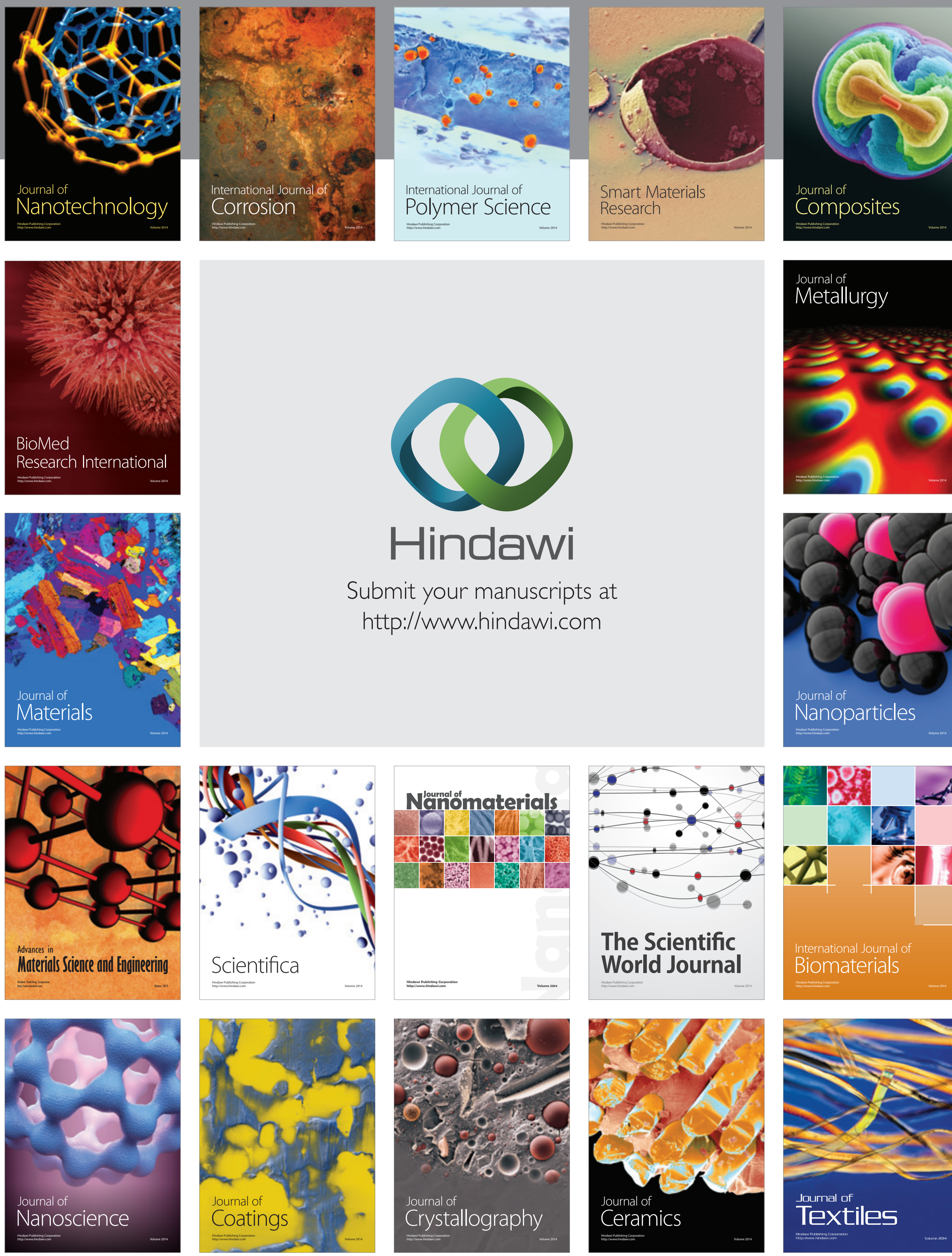
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