

Current Perspectives

Micro-patterning of NdFeB and SmCo magnet films for integration into micro-electro-mechanical-systems

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ARTICLE INFO

Article history:

Received 15 May 2008

Available online 5 October 2008

Keywords:

NdFeB films

SmCo films

Integration

MEMS

ABSTRACT

The integration of high-performance RE-TM (NdFeB and SmCo) hard magnetic films into micro-electro-mechanical-systems (MEMS) requires their patterning at the micron scale. In this paper we report on the applicability of standard micro-fabrication steps (film deposition onto topographically patterned substrates, wet etching and planarization) to the patterning of 5–8 μm thick RE-TM films. While NdFeB comprehensively fills micron-scaled trenches in patterned substrates, SmCo deposits are characterized by poor filling of the trench corners, which poses a problem for further processing by planarization. The magnetic hysteresis loops of both the NdFeB and SmCo patterned films are comparable to those of non-patterned films prepared under the same deposition/annealing conditions. A micron-scaled multipole magnetic field pattern is directly produced by the unidirectional magnetization of the patterned films. NdFeB and SmCo show similar behavior when wet etched in an amorphous state: etch rates of approximately 1.25 μm/min and vertical side walls which may be attributed to a large lateral over-etch of typically 20 μm. Chemical-mechanical-planarization (CMP) produced material removal rates of 0.5–3 μm/min for amorphous NdFeB. Ar ion etching of such films followed by the deposition of a Ta layer prior to film crystallization prevented degradation in magnetic properties compared to non-patterned films.

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1. Introduction

There are many potential applications for high-performance micro-magnets in magnetic micro-electro-mechanical-systems (MEMS) [1]. However, the emergence of magnetic MEMS has been hindered by the challenge of integrating high-quality magnets using techniques compatible with today's MEMS technologies [2]. One of the most promising reports to date, from the viewpoint of integration, concerns the fabrication of Co₈₀Pt₂₀ magnets (cylinders of height 2 μm and diameter 5 or 10 μm, $\mu_0 H_c = 0.5$ T, $(BH)_{\max} = 52$ kJ/m³) by electro-deposition at room temperature [3]. The successful integration of RE-TM magnets into devices, for which much higher coercivities and energy densities may be expected, should provide a significant boost to the field of magnetic MEMS. To achieve this, two challenges are faced. The first of these is the preparation of thick film magnets (1–100 μm) at reasonable deposition rates over relatively large surface areas. High-rate triode sputtering has been

shown to be suitable for the preparation of thick films of both SmCo [4] and NdFeB [5] alloys and we have recently used this technique to prepare high-energy product 5 μm thick films of these materials on Si substrates (SmCo₇—140 kJ/m³, NdFeB—400 kJ/m³) [6,7].

The second challenge to be solved is the lateral structuring of the films at the micron scale. The use of Si substrates greatly facilitates lateral structuring using standard micro-technology processes. However, the use of these standard processes for the structuring of rare-earth-based alloys, in particular steps based on chemical attack, is challenging because of the poor resistance of these alloys to oxidation. Features of size 5 × 5 μm² up to 100 × 100 μm² were wet-etched into 1 μm thick NdFeB films sputtered onto Al₂O₃ substrates using salpetric acid [8]. Reports exist on both the dry and wet etching of SmCo films deposited on Al₂O₃ substrates [9,10]. Wang et al. [9] compared the etching rates achieved using different Ar-based plasmas and found that addition of Cl₂ to an Ar plasma led to a 10-fold increase in etching rate (a maximum value of 0.7 μm/min was reported) compared with a pure Ar plasma. Budde et al. [10] reported an increase in etching rate of SmCo from 0.02 μm/min for dry etching to 12.5 μm/min for wet etching. A major concern is whether

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coercivity is altered during structuring. Budde et al. [10] referred to the magnetic properties of the non-patterned films but not to those of the wet-etched films, while Wang et al. [9] only gave the magnetic properties of films etched at very low rates. Lemke et al. [8] reported that no significant change was observed in the coercivity of their patterned NdFeB films, which however, had relatively low coercivities to begin with (0.2 T).

In this paper we report on a study of three different processes ((i) film deposition onto patterned substrates, (ii) wet etching and (iii) planarization) for the micro-patterning of RE-TM films. The influence of the processing steps on the magnetic properties of the hard magnetic material is presented.

2. Experimental details

Patterned Si/SiO₂ substrates containing trench motifs with individual trenches of depth 6 μm , length 500 μm and trench/wall widths on the scale of 5–100 μm were prepared by standard micro-fabrication processes: deposition of 6 μm of SiO₂ on Si followed by photo-lithography through a mask containing the trench motif and finally dry etching of the SiO₂. The magnetic (NdFeB, SmCo) and buffer/capping (Ta, Cr) layers were deposited using DC triode sputtering at rates of the order of 18 $\mu\text{m}/\text{h}$ [6,7], onto patterned Si/SiO₂ wafers and standard Si wafers. The NdFeB films were deposited at temperatures $\leq 400^\circ\text{C}$, and were thus amorphous [7]. The SmCo films were deposited in both the amorphous (no-substrate heating) and crystallized state ($T_{\text{sub}} = 400^\circ\text{C}$) [6]. **Post-deposition annealing of amorphous films was carried out at 750°C for 10 min under secondary vacuum (10^{-6} mbar) either in-situ (full wafer) or ex-situ on samples of typical surface area $5 \times 5 \text{ mm}^2$.** Film thicknesses were measured using a Dektak profilometer. Magnetic measurements were made at 300 K on a Vibrating Sample Magnetometer (VSM) with a maximum magnetic field of 8 T. Observations were made with an FEG-SEM (Gemini 1530).

3. Results and discussion

3.1. Micro-patterning of RE-TM films

3.1.1. Deposition of RE-TM films onto patterned substrates

SEM images of fractured cross-sections of pieces of {Ta(100 nm)/NdFeB(8 μm)/Ta(100 nm)} and {Cr(100 nm)/SmCo(8 μm)/Cr(100 nm)} films, deposited onto non-heated, patterned Si/SiO₂ substrates, are shown in Fig. 1a and b. **It was found that NdFeB fills comprehensively and continuously the patterns even for trenches with an aspect ratio of 1.2 (6 μm deep, 5 μm wide).** Though SmCo fills the trenches, the film structure is different at the trench corners, appearing granular and less dense than the rest of the film (Fig. 1c). Note that because the substrates were not heated during deposition, the as-deposited films are amorphous. Deposition of SmCo in the crystallized state, achieved by heating the substrate to 400°C during deposition [6], did not lead to an improvement of the trench corners. Nor did a decrease in the sputtering rate from 18 to 10 $\mu\text{m}/\text{h}$, achieved by decreasing the target potential from 900 to 300 V. The difference in trench filling behavior of the NdFeB and SmCo alloys is not presently understood.

The amorphous as-deposited films were annealed at a temperature of 750°C for 10 min to crystallize the hard magnetic Nd₂Fe₁₄B and SmCo₇ phases [6,7]. Post-deposition annealing of the entire 100 mm patterned wafers with NdFeB layers did not lead to film fracture or peel-off. In contrast, annealing of the

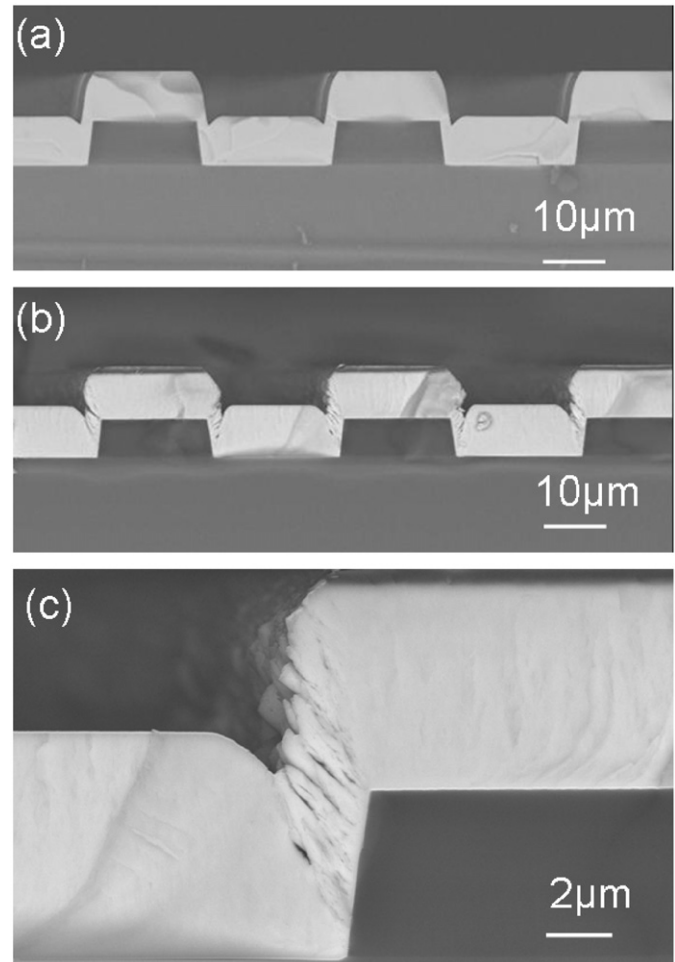


Fig. 1. SEM cross-sectional images of RE-TM films deposited onto patterned wafers (this particular section has trench and wall widths of 20 μm): (a) NdFeB, (b) SmCo and (c) zoom of a trench corner for the SmCo film.

amorphous SmCo films deposited on the patterned wafers does lead to film peel-off. Peel-off occurs via fracture below the SmCo/substrate interface (i.e. some SiO₂ remains adhered to the SmCo film). **When deposited directly in the crystallized state ($T_{\text{sub}} = 400^\circ\text{C}$), peel-off from patterned SmCo films do not occur in the trenched regions, but only in the continuous planar regions between different motifs.**

Film peel-off is attributed to internal stresses and the build up of strain due to the difference in thermal expansion coefficients of the magnetic layer and the Si wafer. While Si has a thermal expansion coefficient of $2.6 \times 10^{-6}/\text{K}$, values in the range of $6 \times 10^{-6}/\text{K}$ – $12 \times 10^{-6}/\text{K}$ have been measured for RE-TM alloys at 750°C [11]. The fact that NdFeB films have distinctly less tendency to peel-off than SmCo₇ films may be attributed to two particular features. The first is that Nd₂Fe₁₄B has a lower thermal expansion anisotropy than SmCo₅ (SmCo₇ is closely related to the SmCo₅ phase) [11]. The second is the fact that Nd₂Fe₁₄B is an Invar system. A large positive magnetovolume anomaly develops in the ferromagnetic state, below 592 K, which reduces very significantly the material's thermal expansion. The difference in behavior of the two material systems may also be attributed to a difference in internal stresses. Indeed these material systems have quite different microstructures when prepared in thick film form [6,7]. A study of the mechanical properties of these films is underway.

3.1.2. Wet etching of RE-TM films

Wet etching trials on non-capped 5 μm thick RE-TM films were carried out on continuous planar films in the amorphous state. This is because it is expected that amorphous films should be less prone to oxidation than crystalline films, due to the absence of grain boundaries which can act as paths for oxygen diffusion. In addition, inhomogeneous etching due to the difference in etching rates of grains and grain boundaries is thus avoided. Optical lithography was used to define mm sized patterns in a resist layer deposited on the RE-TM film. Removal rates of approximately 1.25 $\mu\text{m}/\text{min}$ were achieved for both NdFeB and SmCo wet etched at room temperature in an acidic bath containing $(\text{NH}_4)_2\text{S}_2\text{O}_8 \cdot \text{H}_2\text{O}$ and H_2SO_4 . A large lateral over-etch of typically 20 μm occurred in both materials. The side walls of the etched films are relatively vertical (Fig. 2). Budde had also reported vertical flanges for wet-etched SmCo films, which he attributed to large lateral over-etching [9]. Note that patterns as small as a few tens of μm in size could in principle, be defined this way.

3.1.3. Planarization of NdFeB films

Chemical–Mechanical–Planarization (CMP) can be used to eliminate topographical relief. This step was developed for NdFeB only, as the poor quality of the trench corners of SmCo deposited on patterned substrates and stress issues render them unsuitable for planarization. Five-micrometer thick NdFeB films were deposited onto non-heated planar Si substrates. Wet etching was used to topographically pattern the magnetic films. These patterned films were planarized to a final thickness of 1 μm using CMP. Film removal rates of 0.5–3 $\mu\text{m}/\text{min}$ were achieved by varying the composition of the polishing slurry.

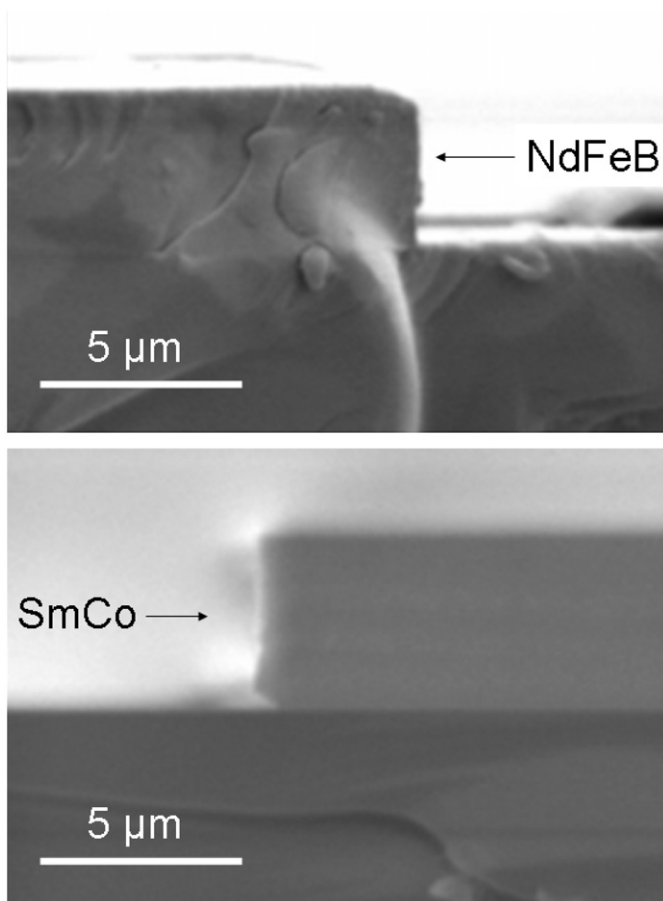


Fig. 2. SEM cross-sectional images of a fracture surface of wet etched RE-TM films.

3.2. Magnetic properties of micro-magnets

The extrinsic properties of a permanent magnet (coercivity, remanence, loop squareness etc.) depend on both the material's intrinsic magnetic properties and the magnet's microstructure (grain size, grain boundary quality, oxygen content, crystallographic texture etc.). As the latter may be influenced by how the magnet is prepared, the different steps involved in the micro-patterning of magnets may be expected to influence the micro-magnet's extrinsic properties. The magnetic properties of micro-magnets prepared using the various steps described above were thus evaluated.

3.2.1. Magnetic properties following deposition onto patterned substrates

VSM measurements of RE-TM films deposited onto patterned substrates are compared with those of films deposited onto

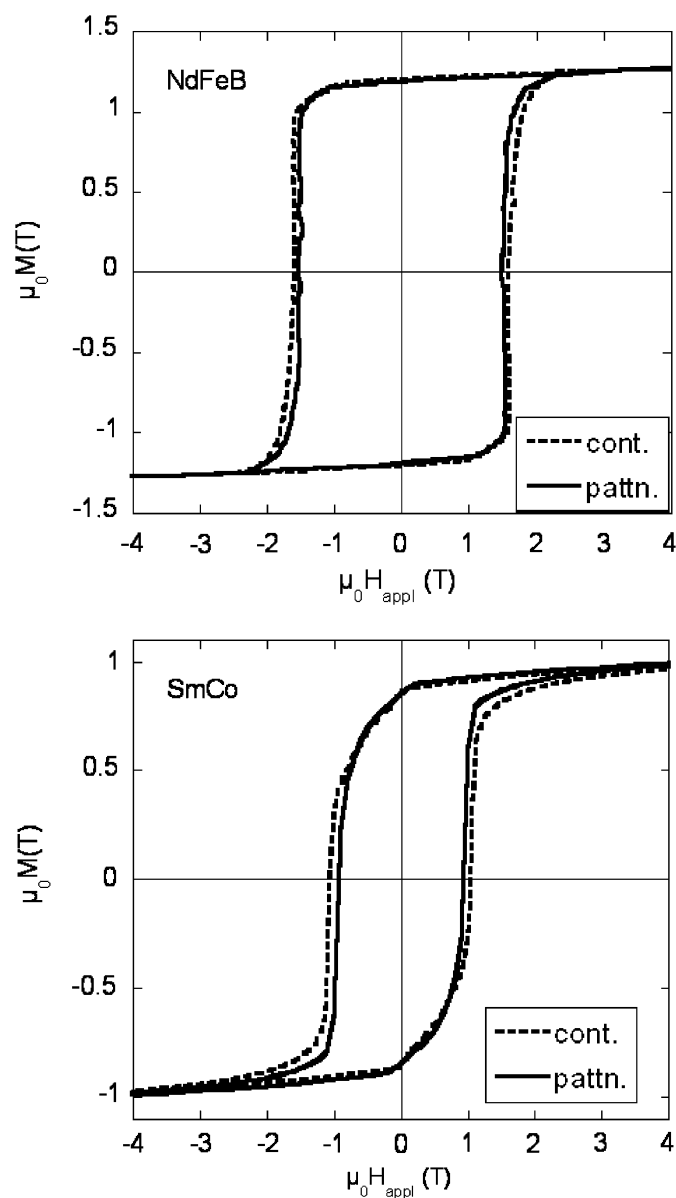


Fig. 3. Comparison of the hysteresis loops of non-patterned continuous (cont.) and patterned (pattn.) RE-TM films: NdFeB (top), SmCo (bottom). Note that the NdFeB films were measured out-of-plane and the loops corrected with a demagnetization factor $N = 1$ while the SmCo films were measured in-plane.

non-patterned substrates and processed under similar conditions (Fig. 3). More specifically, the NdFeB films were deposited amorphous at 400 °C and crystallized during a post-deposition anneal (750 °C/10 min), while the SmCo films were deposited in the crystallized state ($T_{\text{sub}} = 400$ °C). These deposition temperatures were chosen to induce crystallographic texture and thus maximize the remanent magnetization and energy product of the hard magnetic material [6,7]. It can be seen that, for a given material, the hysteresis loops of the patterned and planar films are very similar.

The patterned NdFeB films, which have an out-of-plane texture, were magnetized by the application of an out-of-plane magnetic field of 7 T. A polar Kerr image of a uniaxial Magneto-Optic-Indicator-Film (MOIF) [12] placed on top of the magnetized patterned NdFeB film is shown in Fig. 4. The black/white contrast corresponds to the up/down direction of magnetization in the MOIF. This image reveals that the patterned sections of the film produce micron-scaled multi-polar magnetic field patterns at the position of the MOIF with a pole pitch approximately equal to the pitch of the trench walls (note that the non-patterned sections of film between motifs do not produce enough stray field to saturate the MOIF). This is only a qualitative image and quantitative imaging using a planar MOIF is underway. The relevance of this result is that following a simple fabrication procedure (deposition+annealing of the magnetic layer), the trivial application of a uniaxial magnetic field can be used to produce a micron-scaled multi-polar field pattern. These micro-patterned films have been used to levitate diamagnetic beads [13], and have potential applications in lab-on-chip experiments.

3.2.2. Magnetic properties following etching and planarization

Rare earth elements have a high affinity for oxygen and it is well known that surface oxidation of RE-TM magnets leads to degradation in their magnetic properties [14]. Thus fabrication steps involving chemicals could be critical for the processing of RE-TM films. To assess the impact of the etching/planarization steps, the 1 μm thick amorphous NdFeB patterns produced were further processed under two different conditions: (#1) simple annealing and (#2) Ar ion etching during 5 min (to remove any damaged surface layer produced during the chemical attack) then deposition of a Ta capping layer then annealing. The annealing

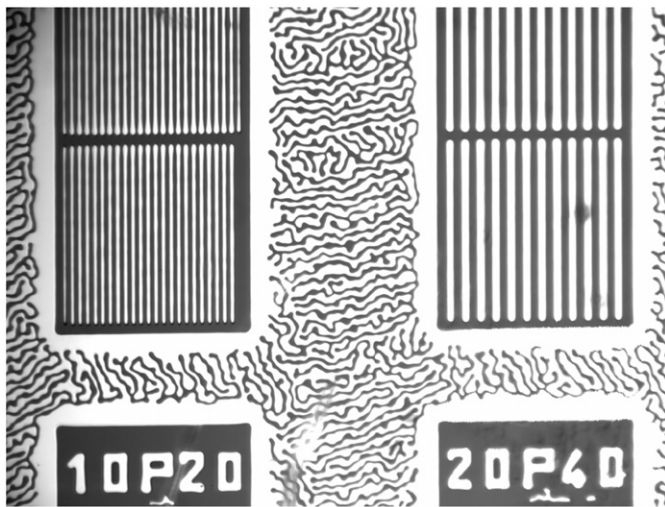


Fig. 4. Polar Kerr optical image of a Magneto-Optic-Indicator-Film (MOIF) placed on top of a patterned NdFeB film (this particular section contains two motifs, one with trench and wall widths of 10 μm and one with trench and wall widths of 20 μm , i.e. trench width/pitch of 10/20 and 20/40 μm , respectively).

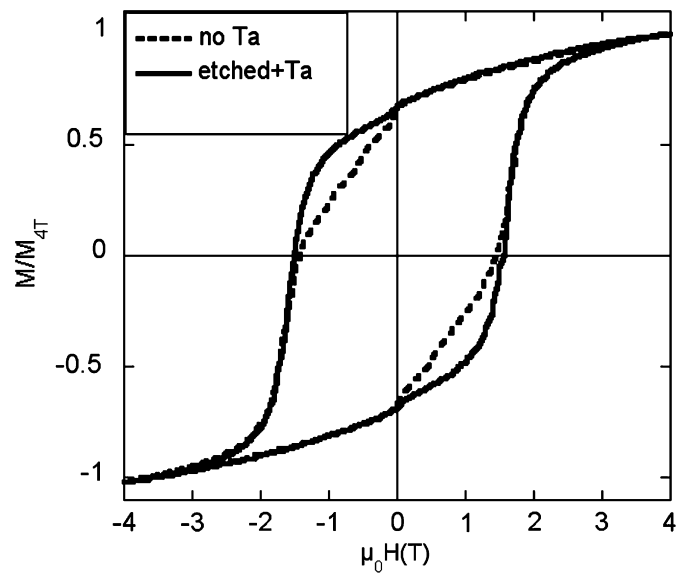


Fig. 5. Comparison of the hysteresis loops of 1 μm thick NdFeB films which were etched and planarized in an amorphous state and then further processed under different conditions: (#1) simple annealing (no Ta), (#2) Ar ion etching (5 min) then deposition of Ta capping layer (etched+Ta) then annealing.

treatments were carried out at 750 °C for 10 min in a rapid heating furnace [15]. In-plane VSM measurements of these samples are compared in Fig. 5. It can be seen that good magnetic properties (single phase behavior, high coercivity ($\mu_0 H_C = 1.5$ T), good squareness for an isotropic sample) are achieved for the sample #2 which was cleaned and capped before annealing. Sample #1 shows approximately the same coercivity value but it has poor loop squareness indicative of a wide distribution in coercivity values of individual grains. Note that samples which were capped with Ta but not etched prior to annealing had a small amount of a soft magnetic phase (data not shown). It can be concluded that light etching serves to remove some damaged material and that the Ta capping layer protects the magnetic film from oxidation during the annealing step.

4. Conclusions

The applicability of some standard micro-fabrication steps has been tested for 5–8 μm thick rare earth transition metal (NdFeB and SmCo) films. It has been found that the deposition of films onto pre-patterned Si/SiO₂ substrates with trench motifs with individual trenches of depth 6 μm and trench/wall widths on the scale of 5–100 μm works better for NdFeB than for SmCo. While NdFeB fills comprehensively and continuously the trenches, the SmCo deposits are characterized by poor filling of the trench corners (i.e. full density is not achieved in these areas). The use of patterned substrates was found to improve the mechanical behavior of the RE-TM films. Film fracture did not occur during the post-deposition high temperature (750 °C) annealing treatment of amorphous NdFeB films deposited on patterned wafers. However fracture occurs upon annealing of amorphous SmCo films deposited on patterned wafers, which should thus be directly deposited in the crystallized state. The magnetic properties (hysteresis loops) of patterned NdFeB and SmCo films are comparable to those of non-patterned films prepared under the same deposition/annealing conditions. The unidirectional magnetization of such a topographically patterned film directly produces a micron-scaled multipole magnetic field pattern.

Wet etching of NdFeB and SmCo gave very similar results: etch rates of approximately $1.25\text{ }\mu\text{m}/\text{min.}$, large lateral over-etch of typically $20\text{ }\mu\text{m}$ and vertical side walls.

Chemical–Mechanical–Planarization was studied for NdFeB films. Material removal rates of $0.5\text{--}3\text{ }\mu\text{m}/\text{min}$ were achieved. Degradation in magnetic properties of the thus patterned NdFeB films, relative to continuous planar films, was prevented by the application of Ar ion etching of the film surface followed by the deposition of a Ta layer prior to film annealing.

The first fully integrated magnetic micro-switch was made with electrodeposited hexagonal close packed CoPtP magnets with relatively low values of coercivity and remanence ($\mu_0 H_c = 0.3\text{ T}$, $\mu_0 M_r = 0.3\text{ T}$) [16]. The integration of high-performance NdFeB magnets into a modified version of this micro-switch, by the consecutive application of the processing steps developed in this work, is presently being studied [17]. The applicability of these micro-fabrication steps to thicker RE-TM films needs to be established. Finally, it should be noted that the high processing temperatures needed for producing NdFeB ($550\text{--}750^\circ\text{C}$) films dictate that these films need to be integrated onto a given wafer before other temperature sensitive components. On the other hand, SmCo alloys, which are produced at relatively low temperatures ($350\text{--}400^\circ\text{C}$), may be processed “above IC” (integrated circuit).

Acknowledgments

This work was carried out in the framework of the ANR “Nanomag2” project. R.G. is grateful for a CNRS visiting research fellowship at Institut Néel.

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- [17] The fabrication of a fully integrated RF micro-switch using RE-TM magnets is being studied within the framework of the ANR (French National Research Agency) “Nanomag2” project, which is a collaborative action between CEA-LETI, G2Elab, Institut Néel and Alcatel Alenia Space.