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SmCo-based MFM Probes with High Switching Fields

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Abstract:

MFM probes with high switching fields have been synthesized by depositing Cr/$SmCo_5$/Cr trilayers onto commercial Si AFM probes. The maximum switching field values of the probes (between 4T and 5T for probes annealed at 550°C for 5 min) is lower than the coercivity measured on similar trilayers (6.8 T) deposited on a Si substrate. Reducing the annealing time of the probes results in a lower switching field while increasing the annealing time leads to a drop in the magnetization of the probes. Such ultra-high switching field MFM probes are of interest for characterizing a range of materials including hard magnetic materials in the remanent state or under an external applied magnetic field and magnetic transducers under excitation.

Introduction:

Magnetic force microscope (MFM) is a standard tool for characterizing sub-micrometer magnetic domains [1,2]. Uncontrolled switching of magnetization at the tip of an MFM probe due to the stray field from the sample under observation and/or an external magnetic field is an issue to be resolved since it may produce unreliable and misleading data. This is especially important when characterizing the domain structure of hard magnetic materials either in their remanent state [3] or under an externally applied magnetic field. [4,5] Among commercially available MFM probes, so-called "high coercivity" probes have switching field values of around 0.5 T (e.g. Asylum

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Research - ASYMFMHC-R2). Several research articles have reported efforts to develop high switching field probes. FePt coated carbon nano-cone probes with switching fields comparable to commercial probes (~0.5 T) were attached to tip-less cantilevers [6]. Switching fields of over 0.5 T has been achieved by coating AFM tips with CoPt layers followed by annealing at 700° C for 4 h (higher switching fields of around 1 T were achieved when such films were deposited on flat substrates). [7] In 2008 [8] Amos et al. demonstrated enhanced spatial resolution in standard Si AFM probes coated with an FePt (10 nm) / MgO (8nm) / FePt (10 nm) trilayers, which had coercivities of the order of 1 T. The enhanced spatial resolution was attributed to antiparallel coupling of the two hard magnetic layers. The same group reported similar values of coercivity for FePt based probes made by a rather elaborate procedure involving focused ion beam etching of a Si AFM probe to produce a plateau, followed by deposition of the magnetic layer on the plateau and subsequent FIB etching to produce a sharp tip [9]. In 2010, Amos et al. slightly enhanced the coercivity of FePt films deposited on Si probes, to between 1.1 and 1.2 T, by using CrRu and MgO seed layers (CrRu/MgO/FePt) and by optimizing the deposition conditions (temperature and pressure). [10] They also demonstrated the interest for these probes by imaging perpendicular magnetic recording write head under various excitation conditions. In 2018, Neu et al. used the focus ion beam method to cut triangle-shaped tips out of epitaxial $SmCo_5$ films with a coercivity of 2.5 T, and then attached them to force cantilevers with the aid of a nanomanipulation tool, for MFM imaging. [11] However, the apparent switching field of the tip was observed to be sample dependent, with a value of above 2 T reported when imaging a bulk Sm_2Co_{17} magnet but a reduced switching field value of 0.7 T when a Co/Pt multilayer thin film was imaged. [11] In this study, we exploit our recent development of high coercivity ($\mu_0 H_C$=6.8 T) polycrystalline SmCo films [12,13] to produce ultra-high switching field MFM tips using commercial AFM probes.

Experimental:

Triode sputtering was used to deposit Cr (10 nm) / Sm-Co (20 nm – 40 nm) / Cr (10 nm) [12,13] trilayers onto commercial Si AFM probes. The AFM probes are purchased from Olympus; Al coated OMCL-AC200T and bare OMCL-AC200TN. The base pressure in the sputtering chamber was $2x10^{-7}$ mbar and high purity Ar was used to produce the plasma, at a pressure of $1.4x10^{-3}$ mbar. A DC voltage of -900 V was applied to a target of total surface area 9 cm × 9 cm, glued to a Cu base plate. The hard magnetic layers of the probes were deposited from a base alloy target of nominal composition $SmCo_5$ with a 2 cm × 2 cm Sm foil bonded onto the surface of the base target. Cr buffer and capping layers were deposited to prevent interdiffusion with the substrate and oxidation, respectively. Note that though no power was supplied to the substrate holder heater during deposition, the probe temperature rose to a value of about 260°C, due to heating from the plasma, and the transfer of kinetic energy from impinging species. Probes were post-deposition annealed at 550°C for times in the range 2.5 - 30 minutes in a tube furnace under a vacuum of 10^{-8} mbar. The AFM probes were mounted on a specially designed sample holder which could be attached to the substrate holder of the sputtering system.

Test samples used to assess the probes were prepared by thermomagnetic patterning (TMP) of an out-of-plane textured NdFeB thick film. NdFeB film is chosen due to the high stray field values. [3] In the TMP process, a previously magnetized NdFeB film is subjected to reverse applied magnetic field while shining a high-energy pulsed laser onto the film surface through a mask with a given pattern. The magnetization in parts exposed to the laser are reversed due to a drop in coercivity of the NdFeB film with increasing temperature [14], thus the pattern is decorated on the hard magnetic surface. Details of the synthesis of NdFeB thick films and thermomagnetic

patterning have been reported elsewhere. [15,16] Such test structures were previously used to study specific aspects of MFM imaging of hard magnetic films [3] and also to quantitatively study magnetic interactions between a micro-magnet array and individual magnetic micro-particles. [17] The test structure used here consisted of stripes of reversed and non-reversed zones of an out-of-plane magnetized NdFeB film, with stripe widths 3 µm and 7 µm, respectively. A schematic of the TMP process is shown in Figure 1.

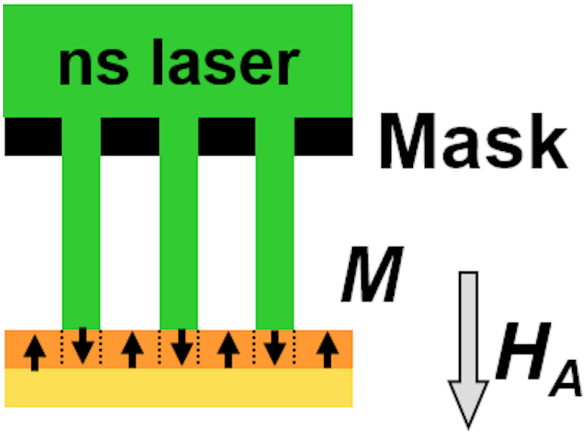

Figure 1. Schematic of the Thermo-Magnetic patterning (TMP) process used to prepare test structures to characterize the switching field of MFM probes.

In a typical test, the following protocol has been applied: a maximum field of +8 T was applied perpendicular to the cantilever of the MFM probe under vacuum in a superconducting coil and then the probe was used to produce an MFM image of the test sample. Following this, the probe was remounted in the superconducting coil, a reverse magnetic field was applied to the MFM probe and then the magnetic structure was rescanned in the MFM. This step was repeated using reverse fields of increasing intensity (at intervals of 1T), and flipping of the MFM contrast

associated with the reversed and non-reversed zones of the test sample was observed. The area scanned changed from one measurement to the next by no more than 10µm. MFM imaging was performed with a Veeco NSV microscope. Scan parameters are given in table 1.

Lift	**Amplitude**	**Scan rate**	**Lines × points**	**Height during first pass**	**Spring Constant**
100 nm < z < 150nm	~ 50 nm	0.5 Hz	128 × 512	30 nm - 35 nm	9 N/m

Table 1. Scan parameters.

Results and Discussions:

Initially, Cr 10 nm/SmCo 20 nm/Cr 10 nm trilayers were deposited on commercial aluminum-coated Si AFM tips, which were then annealed at 550 ºC for 30 min. The Al layer on such probes serves as a reflective layer used to follow the cantilever displacement. Deposition of the trilayer and subsequent annealing did not lead to bending of the cantilever (Fig. 2 a), however closer examination of the probe revealed that the Al coating had melted (Fig . 2. b, c), and presumably diffused into the Sm-Co layer.

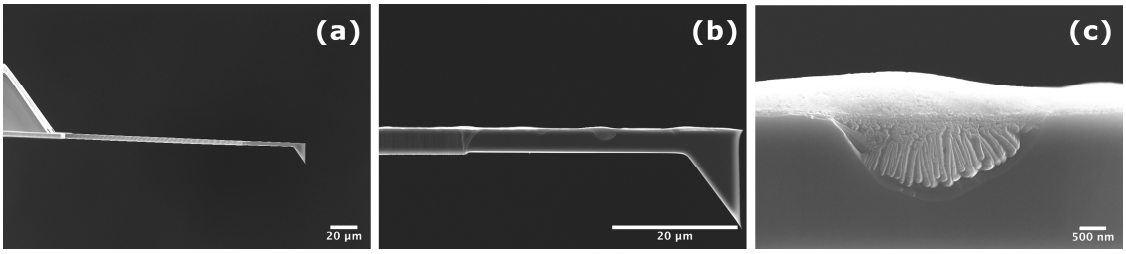

Figure 2. a) SEM image of a commercial aluminum coated AFM probe on which a Cr/SmCo/Cr trilayer was deposited, following annealing at 550°C for 30 min, b) and c) close up images of the tip of the probe, revealing that the Al layer had melted.

Following this, bare Si AFM probes, with no Al coating (OMCL-AC200TN). were covered with Cr 10 nm/SmCo 20 nm/ Cr 10 nm trilayers (Figure 3 a) and then subjected to post-deposition annealing (550 ^{o}C for 5 min.). A test MFM scan made with the probe indicated that the tip was not ferromagnetic. We tentatively attribute this to diffusion between the Si probe and the deposited layers, leading to localized melting at the apex of the tip (Figure 3 b). Loss of the SmCo phase due to diffusion would be critical for probes with very thin magnetic coatings. Doubling the nominal thickness of the hard layer (Cr 10 nm/SmCo 40 nm/ Cr 10 nm) produced tips that showed ferromagnetic behavior.

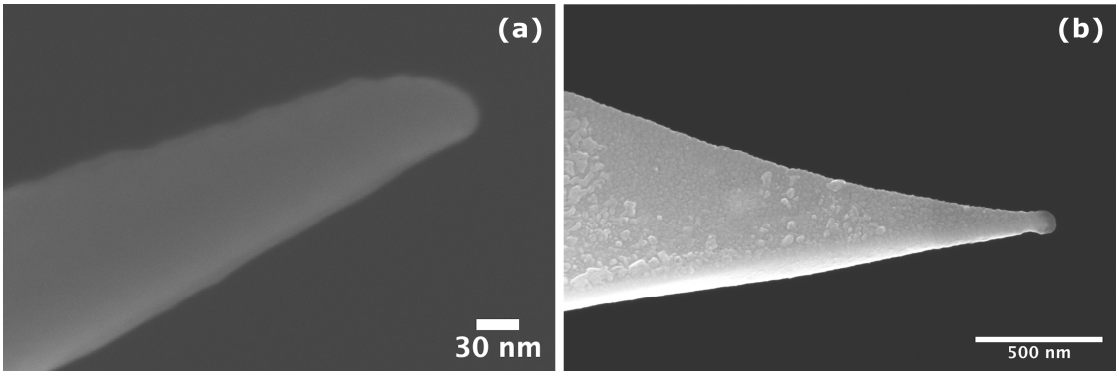

Figure 3. SEM image of a bare Si AFM probe coated with a Cr 10 nm/SmCo 20 nm/ Cr 10 nm trilayer; a, before and b, after annealing at 550°C for 5 min.

MFM images of the test structure made with a (Cr 10 nm/SmCo 40 nm/ Cr 10 nm) coated probe annealed at 550 ^{o}C for 5 min. are shown in Figure 4. All images are made in the remanent state, following ex-situ probe exposure to fields of variable strength. Magnetic contrast is observed at two different length scales. Firstly, coarse structures of width a few microns, which correspond to the thermo-magnetically written striped pattern. The reversed and non-reversed zones are respectively dark and bright dark, in the image made following probe saturation in +8 T. Secondly, there is fine contrast within these written patterns, on a scale of a few hundred nanometers, which results from $Nd_2Fe_{14}B$ grains in the TMP sample which are misoriented with

respect to the out-of-plane direction. [17] The overall contrast of the coarse patterns flips between application of a reverse magnetic field on the probe of - 4T and - 5T, while the contrast of some of the finer features is blurry at fields as low as -1T. This behavior reflects a distribution in switching field values in the SmCo film deposited on the MFM probe. It may also reflect a combination of contributions from very short-range interactions involving SmCo grains of presumably lower coercivity at the tip of the probe and longer-range interactions involving SmCo grains of presumably higher coercivity on the rest of the probe. Blurring of the contrast of the coarse features may be due to a combination of the flipping of lower coercivity grains at the tip of the probe and modifications of phase shift from one MFM to the next, due to slight changes in the tip properties (e.g. contamination with glue used to attach the MFM probe to the superconducting coil sample holder, which would disturb mechanical excitation from the microscope and so the probe oscillation response in phase and amplitude). Although the distribution in coercivities complicates interpretation of phase contrast for sub-micrometer domains, the probes developed here are nevertheless suitable for characterizing changes in high coercivity coarse hard magnetic patterns.

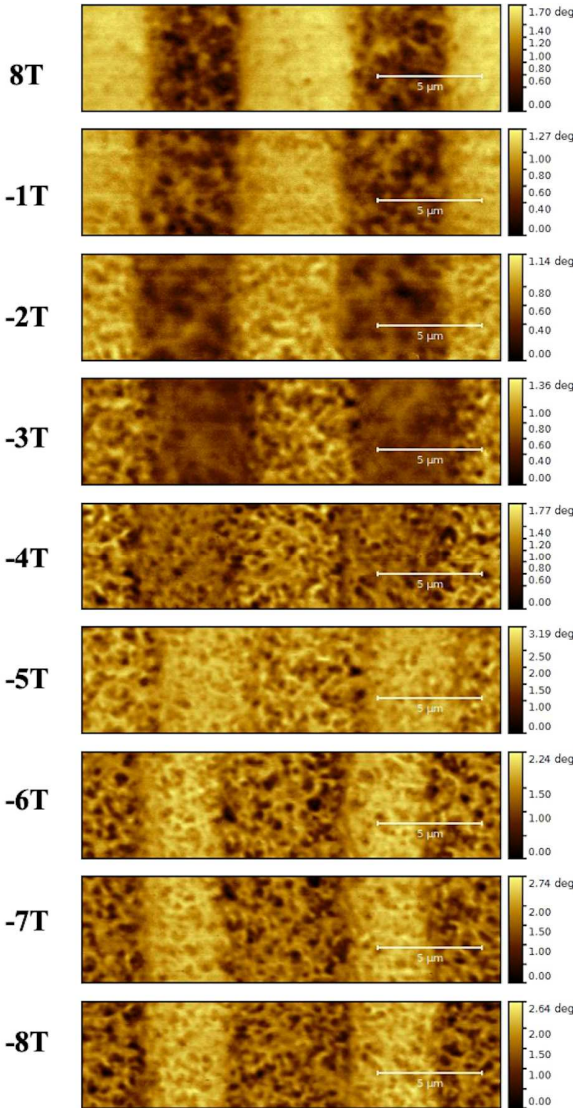

Figure 4. MFM images of a test TMP structure made using a 10 nm Cr/ 40 nm Sm-Co/10 nm Cr coated tip annealed at 550°C for 5 min. All images are made in the remanent state, following the ex-situ exposure of the tip to a magnetic field of a given direction and intensity.

In order to further analyze the effect of annealing time, probes were subjected to annealing at 550°C for 5 min – 30 min. Similar maximum switching field values in between 4T and 5T has been observed from the MFM images taken with the probes annealed for 10 min. (Figure 5)

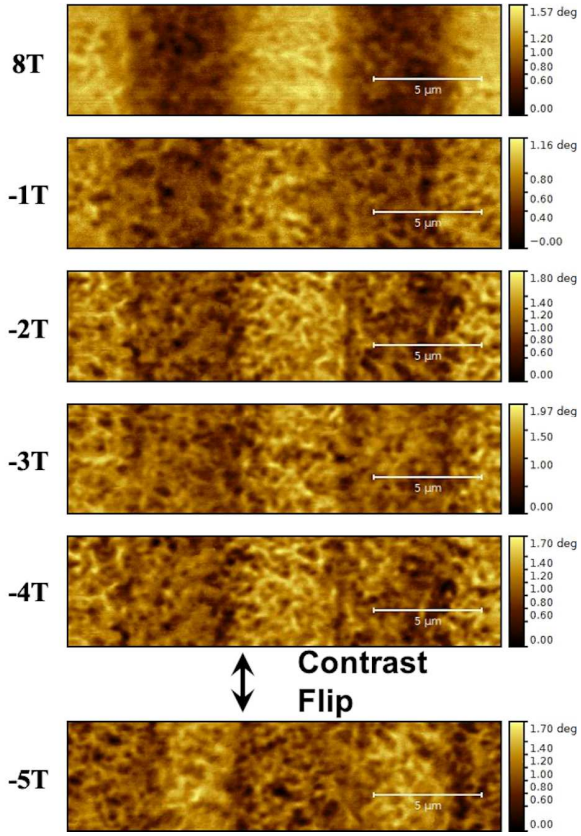

Figure 5. MFM images of a test TMP structure made using a 10 nm Cr/ 40 nm Sm-Co/10 nm Cr coated tip, annealed at 550°C for 10 min. All images are made in the remanent state, following the ex-situ exposure of the tip to a magnetic field of a given direction and intensity.

MFM images made with a (Cr 10 nm/SmCo 40 nm/ Cr 10 nm) coated probe annealed for 2.5 min, reveals that the shorter annealing time leads to a switching field value of less than 1 T (Figure 6a). We attribute this to incomplete crystallization of the hard magnetic SmCo phase. The influence of longer annealing times was also studied. The RMS phase shift associated with MFM image scans of (Cr 10 nm/SmCo 40 nm/ Cr 10 nm) coated probes annealed for times in the range 5-30 minutes, following saturation in a field of 8 T, are compared in Figure 6 b. The drop in phase shift for annealing times of 10 minutes and longer is indicative of a drop in the magnetic moment of the probes, possibly due to insufficient protection of the magnetic layer by the Cr

buffer, leading to interdiffusion with the Si probe, and/or a change in the spring constant due to annealing.

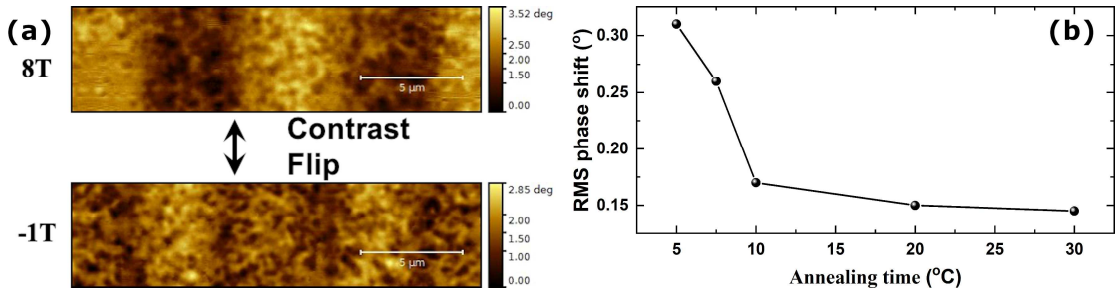

Figure 6. (a) MFM images of a test TMP structure made using a 10 nm Cr/ 40 nm Sm-Co/10 nm Cr coated tip annealed at 550°C for 2.5 min. (b) RMS phase shift associated with MFM image scans of (Cr 10 nm/SmCo 40 nm/ Cr 10 nm) coated probes annealed (550°C) for times in the range 5-30 minutes, following saturation in a field of 8 T.

For comparison, topography and MFM images (Figure 7) has been taken with commercially available high coercivity MFM probe (FePt coated MFM probe (2N/m) ASYMFMHC-R2). Contrast flip of the coarse patterns has been observed below -1T which agrees with the advertised coercivity value of 0.5 T.

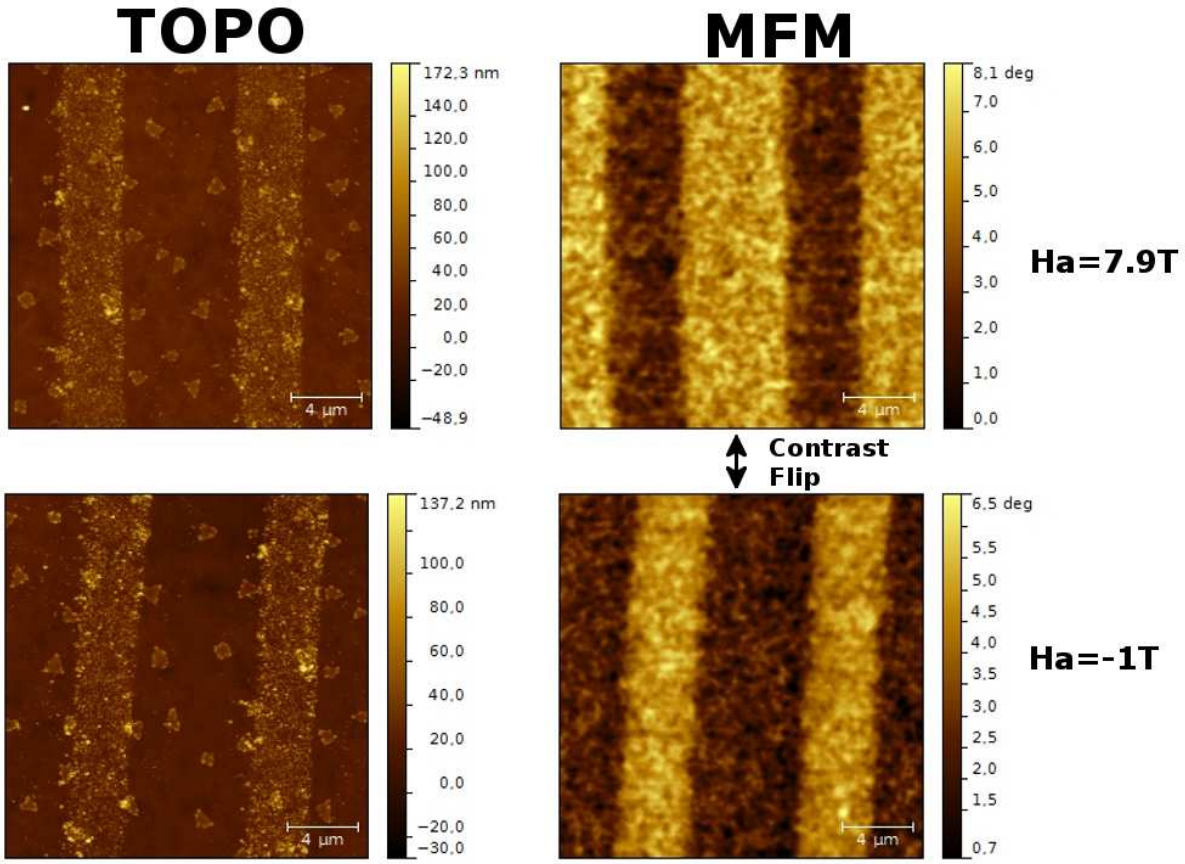

Figure 7. Topography and MFM images of the test film by using the commercial coercive FePt Probe magnetized under external field with certain magnetic field values.

Conclusions:

In conclusion, MFM probes having broad switching field distribution with a maximum value in between 4 T and 5 T have been made by coating bare Si AFM probes with a high coercivity SmCo layer, protected with Cr buffer and capping layers. The maximum switching field value is 10 times higher than commercial high coercivity MFM probes. Our fabrication approach is relatively simple and cheap, compared to other rather elaborate approaches reported in the literature. While the observed distribution in switching field values complicates interpretation of phase contrast for sub-micrometer domains, the probes developed here are suitable for

characterizing changes in high coercivity coarse hard magnetic patterns under the influence of an external magnetic field. Efforts will now be made to reduce the distribution in switching field values, so as to improve the spatial resolution of the probes.

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