# Thick Multilayered Micromachined Permanent Magnets With Preserved Magnetic Properties

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Abstract-This paper presents microfabricated permanent magnets possessing a multilayer structure that preserves the high magnetic energy density of thinner magnetic films, while simultaneously reducing average residual stress of the films and achieving a significant magnetic thickness. Many magnetic microelectromechanical systems (MEMS) devices heavily rely on the availability of thick (a few tens to hundreds of micrometers, high-energy-density permanent magnet components able to be deposited in a fully integrated and CMOS-compatible manner (process temperature less than 450 °C). However, it is observed that increasing magnetic film thickness typically causes a concomitant decrease in magnetic properties such as maximum energy density and increased mechanical instability (cracking and delaminating, due to the increased elastic strain energy stored in the films as magnetic volume increases), both of which limit the maximum total magnetic energy of these small-scale integrated magnets. The microlaminated permanent magnet presented here utilizes sequential multilayer electroplating in which the alternating layers of relatively thin magnetic films (CoNiP, micrometer range) and non-magnetic materials (Cu, a few tens of nanometer to micrometer range) are electrodeposited in a multilayer fashion realizing thick laminated permanent micromagnets with improved total magnetic energy as compared with their non-laminated counterparts. Low interface roughness has been demonstrated to play an important role in preserving the component magnetic thin layer properties in the multilayer configuration. [2015-0300]

*Index Terms*—CoNiP, electrodeposition, magnetic microelectromechanical systems (MEMS), multilayer, permanent magnet.

# I. INTRODUCTION

MICROMACHINED permanent magnets play an important role in magnetic MEMS devices such as compasses, micromotors, microphones, and relays, due to their ability to generate magnetic fields (after magnetization) in the absence of external energy sources. However, incorporation of such micromagnets in MEMS systems is often challenging, mainly due to the lack of large-volume,

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high-energy-density  $((BH)_{max})$  permanent magnet components that are able to be deposited in a fully-integrated and CMOS-compatible manner. Such micromagnets, simultaneously possessing desirable magnetic properties as well as large volumes, are essential in applications requiring significant magnetic flux (e.g., magnetic energy harvesting), volumetric magnetization (e.g., micromachined compasses), and magnetostatic force (e.g., magnetic MEMS relays). In the previously mentioned applications, properties such as total magnetic energy, rather than solely magnetic energy density, are required. In such applications, thin films (less than a micrometer) though possessing superior energy densities, are less applicable for such magnetic MEMS systems [1]–[4].

The most intuitive approach to increase the total magnetic properties of these films is to increase their volume; typically, for a given footprint, this translates to increasing their thickness, e.g., by depositing films for longer periods of time or at higher rates. However, two issues may arise with this simple strategy. First, it is observed that the magnetic properties of thin films do not simply scale with increasing thickness. Magnetic properties have been reported to deteriorate with continued film growth [5]-[8], resulting in magnetically weakened permanent micromagnets. Secondly, in general, hard magnetic films possess high residual stress [9], [10]. For a film with a given level of residual stress, there exists a maximum depositable film thickness (inversely proportional to the square of the residual stress of the film) at which spontaneous film delamination occurs [11]. The magnetic and mechanical deterioration of the thicker permanent magnetic films may limit their utility in the applications described above [12].

To address these issues, we propose laminated permanent micromagnets as illustrated in Figure 1. Rather than continuously grow the magnetic film to achieve a large volume and tolerate the reduced  $(BH)_{max}$  that accompanies that larger volume (Figure 1(a)), the stacking of multiple thin magnetic films with preserved  $(BH)_{max}$  is investigated as an approach to achieve large overall magnet thicknesses while simultaneously retaining maximum magnetic energy density (Figure 1(b)). In order to realize the concept of laminated structures comprising stacked thin films, two approaches are possible. The first exploits self-assembly or guided-assembly [13]. Individual layers of functional thin films are released from the substrate where favorable forces such as surface tension, magnetic, and/or electrostatic forces are harnessed to assemble individual layers that are optionally subsequently further bonded together. Although this approach benefits from fabrication simplicity, it may be less applicable in devices requiring precise inter-

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Fig. 1. Illustration of the concept of laminated hard micromagnets (not drawn to scale). (a) The magnetic properties of thick electrodeposited magnetic films tend to decrease with thickness; (b) Conceptually, stacking individual films can produce thick magnets with preserved properties; (c) Implementation of stacking in-situ using sequential multilayer electrodeposition.

layer or magnet-substrate alignment. The alternative approach proposed here exploits sequential multilayer electrodeposition [14]. Individual layers of relatively thin film magnets are electrodeposited in a multilayer fashion to achieve a laminated permanent magnetic structure in a stacked configuration. One important benefit of this approach is that, as shown by previous studies [9], [15]–[17], by incorporating proper auxiliary thin film layers with lower or opposite-signed stress than that of the functional thin films in the stacked layer, the average residual stress can be reduced to enhance the mechanical stability of the films.

Previously, utilizing sequential multilayer electrodeposition, we have demonstrated laminated soft magnetic films as magnetic cores in the application of DC-DC power conversion systems to suppress eddy current loss while at the same time achieving large overall core thicknesses for high power handling capability [18], [19]. In comparison, the goal of the thin film lamination of permanent magnets in this work is to retain the superior thin film permanent magnetic properties at large magnetic thickness such that improved total magnetic energy can be achieved in these laminated micromagnets as compared with their non-laminated counterparts. In considering sequential electrodeposition for laminated magnet formation, it should be noted that magnetic properties (magnetic moments, anisotropy, coercivity and domain structure) of thin films are influenced by surface/interface roughness [20]-[24]. A layer-by-layer growth inevitably uses the underlying layer as an effective substrate, the surface roughness of which could influence the properties of the subsequently-deposited layer. Further, this effect could accumulate as the number of layers increases, negatively affecting the preservation of thin film properties. Hence, an interlamination layer (e.g. copper, Figure 1(c)) electrodeposited in an alternating fashion with the magnetic layers, is investigated as an approach to "reset" the deposition surface and alleviate these effects.

# II. DESIGN AND OPTIMIZATION

# A. Material Selection

The proposed laminated hard micromagnets are comprised of two types of component layers: (i) the *magnetic layer* a hard magnetic material; and (ii) the *interlamination layer* a nonmagnetic layer deposited for the purpose of "resetting" the growth of high-energy-density magnetic films, and acting as a substrate for the deposition of subsequent magnetic layers.

Candidate materials for the magnetic layer should be compatible with electrodeposition and preferably possess a controllable direction of magnetic anisotropy. Electrodeposition is of interest not only due to its relative economy and low operating temperatures, but more importantly, for the relatively rapid deposition rates achievable, allowing achievement of substantial overall thickness. For example, the deposition rate in this work is around 8 nm/s. In comparison, high rate sputtering of NdFeB thick films is 5 nm/s [25] and conventional sputtering speed is less than 0.1 nm/s [26]. The multilayer structure (Figure 1) has magnetic films with a high aspect ratio of in-plane dimension to thickness, resulting in a high in-plane magnetic shape anisotropy. A magnetic material with controllable magnetocrystalline anisotropy could potentially be engineered to align the shape anisotropy (associated with shape of the thin film) and magneto-crystalline anisotropy (associated with preferred crystalline orientation), thereby boosting overall magnetic performance.

Example suitable hard magnetic materials include RE (rare earth) magnetic alloys (e.g. NdFeB and SmCo), equiatomic Pt-TM (Transition Metal) alloys (e.g. FePt L1<sub>0</sub> and CoPt L1<sub>0</sub>), Co-rich hexagonal alloys (e.g. CoNiP, CoNiMnP, CoPtP) and others [1], [2]. RE magnetic alloys often seen in bulk-scale applications could be integrated in MEMS with top-down fabrication approaches [27] but with restrictive processing conditions [8]. Equiatomic Pt-TM alloys with attractive performance commonly require either a high temperature deposition environment or a high temperature post-process annealing. Among the Co-rich hexagonal alloys, CoNiP not only can be readily electroplated without precious metals but also can be conveniently tuned by bath compositions [28] and electroplating parameters [29] to yield in-plane magnetocrystalline anisotropy with significant in-plane maximum energy density.

In addition to being electrodepositable (to support a subsequent magnetic layer deposition), and possessing low or opposite-signed residual stress (to reduce the average stress of the deposited films), it was hypothesized that the interlamination layer material should have excellent planarization properties such that the surface conditions of the interlamination layer after deposition would largely mimic those of an original sputtered Cu seed layer. In this case, the accumulating surface roughness of thicker magnetic layers is reduced or reset due to the planarizing properties of the interlamination layer, potentially resulting in preserved magnetic properties. Cu plated from a commercial copper bath (Grobet, Clean Earth Cu-mirror solution) containing brighteners and levelers was chosen as the interlamination material for its low surface roughness [18].

 TABLE I

 Electrodeposition Conditions for CoNiP Thin Films

Items	Quantity
NiCl <sub>2</sub> •6H <sub>2</sub> O	0.2 (M)
$CoCl_2 \bullet 6H_2O$	0.206 (M)
$NaH_2PO_2 \bullet H_2O$	0.028 (M)
NaCl	0.7 (M)
$H_3BO_3$	0.4 (M)
Saccharin	0.0048 (M)
Temperature	20-25°C
<b>Current Density</b>	20 mA/cm <sup>2</sup>
рН	2.2
Agitation	None

# B. Optimization of Electrodeposition Condition

Park et al. [28] showed that the magnetic properties (in-plane/out-of-plane (BH)max and anisotropy) of CoNiP were strongly influenced by NaH<sub>2</sub>PO<sub>2</sub> concentrations in the bath. Kirkwood et al. [29] demonstrated a strong correlation between applied current density and c-axis orientation (perpendicular/longitudinal) to the film plane. The CoNiP electrodeposition conditions (i.e., chloride plating bath and deposition parameters) for this paper were adopted from the above-mentioned papers and optimized for multilayer deposition to achieve significant longitudinal (in-plane) magnetic anisotropy and energy density. The parameters are summarized in Table I. The deposition pH was chosen to be 2.2 since at this level, CoNiP thin films show simultaneously the highest in-plane coercivity and squareness [28]. The NaH<sub>2</sub>PO<sub>2</sub> (phosphorous source) concentration was chosen to be 0.028 M, as the deposited films not only shows strong hcp (100) peak from XRD analysis indicating a well-defined c-axis in the in-plane direction, but also exhibit high maximum magnetic energy density. Further increasing the NaH<sub>2</sub>PO<sub>2</sub> concentration would change the preferred orientation from hcp (100) to hcp (002) [28]. A current density of 20 mA/cm<sup>2</sup> was chosen for an optimized combination of in-plane anisotropy (below which a decrease of hcp (100) shows up concurrently with an increase of hcp (002)) and in-plane coercivity (above which coercivity drops sharply). NaCl and boric acid operated as supporting electrolyte and pH buffer, while saccharin was used for the purpose of reducing deposit stress [28]. No agitation was applied and a Ni sheet was used as an anode for Ni ion replenishment. Due to the lack of Co ion replenishment as plating continued, in order to ensure compositional uniformity of each magnetic film layer in the multilayer structure, the bath volume was adjusted such that the consumption of Co was less than 1% of the total dissolved Co ions in the bath for every batch.

# III. EXPERIMENTAL

# A. Fabrication Sequence

Figure 2 illustrates the fabrication sequence of the laminated magnets. An insulating layer of silicon dioxide  $(1 \ \mu m)$  was



Fig. 2. Fabrication process flow (not drawn to scale): (a) solvent cleaning of a 4-inch wafer; (b) deposition of insulating layer of silicon dioxide and seed layer of Ti/Cu/Ti; (c) deposition and patterning of thick photoresist mold with its dimension shown in (d); (e) stripping of top layer of Ti before electroplating commenced; (f) robotically-assisted sequential multilayer electrodeposition of CoNiP and Cu; and (g) removal of the photoresist mold.

deposited on both sides of a solvent-cleaned 4-inch wafer by plasma enhanced chemical vapor deposition (PECVD). A seed layer comprising copper (500 nm) sandwiched by titanium (30 nm) layers was then formed using DC sputtering (Figure 2(a), (b)). The top Ti layer not only reduces possible oxidation of the Cu seed layer which serves as the substrate for the initial CoNiP layer growth, but also helps to enhance the adherence of the photoresist mold. A thick photoresist (NR 21-20000P, Futurrex, Inc.) mold was then patterned consisting of an array of circles, each 16 mm<sup>2</sup> in area (Figure 2(c), (d)). The top layer of Ti within the mold was stripped in dilute hydrofluoric acid (2% vol.) just before electroplating commenced (Figure 2(e)). For single layer films, CoNiP films were electrodeposited (with conditions detailed in Table I) using a DC current source. For multilayer films, robotically-assisted sequential multilayer electrodeposition (refer to [30] for details) comprising alternating CoNiP and Cu layers was carried out (Figure 2(f)) in a dual bath system with the customized CoNiP bath (Table I) and the commercial Cu bath (using a Cu anode with a current density of 20 mA/cm<sup>2</sup>). Individual component layer thicknesses were controlled by the electrodeposition time utilizing Faraday's law of electrolysis. For samples conducting stress measurement, no photoresist molds were used and film deposition occurred directly on 4-inch Si wafers with oxide and seed layer as described above.

# B. Characterization Method

After deposition, the photoresist mold was stripped (Figure 2(g)) and the cross-sectional morphology of the resulting micromagnets was characterized by a scanning electron microscope (Zeiss Ultra60 FE-SEM) equipped with energy-dispersive X-ray spectroscopy (EDX). Surface roughness analysis was performed using atomic force microscopy (Bruker Icon AFM) operating in tapping mode. Crystalline structure, grain size and preferential orientation of the films were examined by X-ray diffraction (XRD, Rigaku Geiger-Flex D/Max-B diffractometer) in the Bragg-Brentano geometry with Cu K<sub> $\alpha$ </sub> radiation. Peak profile analysis was performed using X'Pert HighScore Plus and Igor Pro multi-peak fitting software packages. Magnetic properties were examined by vibrating sample magnetometry (VSM, LakeShore,



Fig. 3. Typical in-plane and out-of-plane hysteresis loops of single layer  $1-\mu$ m-thick CoNiP films.



Fig. 4. A typical XRD spectrum of single layer 1-µm-thick CoNiP films.

Model730). No correction for demagnetization effect was applied to the data presented throughout the paper. It should be noted that the magnetic volume (i.e., the sum total of all magnetic layers, omitting the interlamination layers) was used for the estimation of magnetic properties measured by VSM throughout this paper for both single-layered magnets and microlaminations. For thicker single-layered micromagnets, epoxy encapsulations were applied to the films immediately after deposition of the films in order to temporarily enhance the mechanical stability for magnetic property measurement due to the large strain energies of these thicker single-layer films. Film stress measurements based on curvature of 4-inch Si wafers bearing films were carried out using a stylus profiler (KLA P7 3D/stress profiler).

# IV. SINGLE LAYER CONIP FILMS

Figure 3 shows typical in-plane and out-of-plane magnetic hysteresis loops, measured by VSM, of a single layer 1- $\mu$ m-thick CoNiP thin film deposited using the conditions discussed above. As desired, the magnetic easy axis lies in the in-plane direction with an average coercivity of 50 kA/m and maximum magnetic energy density of 23 kJ/m<sup>3</sup>. EDX results showed that these films had a composition (atomic %) of 78 Co%, 13% Ni and 9% P. Both the thin film magnetic performance and the compositions are similar to the values reported in the literature [28], [29]. XRD analysis of these 1- $\mu$ m-thick films is shown in Figure 4 and indicates that the deposited CoNiP films exhibited hcp structures, including



Fig. 5. Maximum magnetic energy density as a function of film thicknesses of single layer CoNiP films.

hcp (100), (002), (101), (110) and (112) peaks, corresponding well with the literature [28], [29], [31]. As a result of the optimized bath composition and electroplating conditions, comparatively strong hcp (100) and weak (002) peaks were evident as compared with CoNiP powder diffraction diagrams reported in the literature [27], indicating a well-defined hcp (100) texture with c-axis orientated mainly parallel to the film plane.

Multiple permanent magnetic alloy systems (e.g. CoPt, CoNiMnP) show a decrease of maximum magnetic energy density with increasing deposited film thickness [5]–[8]. A similar trend was observed in the CoNiP alloy system, which is shown in Figure 5. The energy product  $(BH)_{max}$  decreases monotonically as the film thickness increases. In particular, the maximum energy density value decreased sharply between film thicknesses of 1-10  $\mu$ m, showing two "knee points" with the first at approximately 1 $\mu$ m and second at approximately 10  $\mu$ m. The data shown in Figure 5 could be used as a basis for determining the "stacked" individual thin film thickness and the resultant number of layers needed for the assembly of a laminated structure. In subsequent multilayer electrodeposition magnet stacks fabricated in this work, the individual magnetic layer thickness is set to 1 $\mu$ m.

# V. LAMINATED CoNiP MICROMAGNETS

Stacks of laminated CoNiP micromagnets have many geometric parameters, necessitating the introduction of descriptive nomenclature. The individual functional component (magnetic) layer thickness is defined as  $t_{CoNiP}$  and the corresponding individual interlamination layer thickness is  $t_{Cu}$ . The total *magnetic* thickness  $t_M$  is defined as the sum of individual functional component layer thicknesses ( $t_M = \sum t_{CoNiP}$ ). Similarly, the total *interlamination* layer (or interlayer for short) thickness  $t_I$  is defined as the sum of individual interlamination layer thicknesses ( $t_I = \sum t_{Cu}$ ). The magnetic thickness will always be less than or equal to the total thickness T which is defined as the sum of all functional and interlayer thicknesses ( $T = t_M + t_I$ ). Obviously, in a case of a single



Fig. 6. Cross-sectional SEM images of (a) a  $[CoNiP(1\mu m)/Cu(1\mu m)]_{10}$  microlamination and (b) an enlarged view.

layer magnetic film, the magnetic thickness and the total thickness are equal  $(t_M = T)$ . Fill factor  $\gamma$  is defined as the ratio of  $t_M$  to T ( $\gamma = t_M/T$ ). We introduce the notation [CoNiP( $t_{CoNiP}$ )/Cu( $t_{Cu}$ )]<sub>n</sub> to represent the proposed microlamination structure, indicating that in the structure, CoNiP layers and Cu layers were deposited in an alternating fashion, with the very first layer deposited being CoNiP with thickness  $t_{CoNiP}$  (directly on seed Cu), followed by a Cu layer with thickness  $t_{Cu}$ . We define this first CoNiP layer and the first Cu layer to be the 1<sup>st</sup> pair. Subsequent pairs are defined similarly, so that the n<sup>th</sup> pair is comprised of the n<sup>th</sup> CoNiP layer and the n<sup>th</sup> Cu layer. Hence, a [CoNiP( $t_{CoNiP}$ )/Cu( $t_{Cu}$ )]<sub>n</sub> lamination comprises n pairs, and 2n structural layers.

#### A. Fabrication Results

A typical cross-sectional SEM image of the fabricated magnetic microlamination is shown in Figure 6(a), featuring a  $[CoNiP(1\mu m)/Cu(1\mu m)]_{10}$  microlamination. A short selective Cu wet etch using an acidified thiourea solution [32] was performed to create contrast between the layers; the brighter, protruding layer and darker, receding layer are CoNiP and Cu, respectively (see Figure 6(b)). EDX results verified that there is no significant compositional fluctuation between the bottommost and topmost individual magnetic layers, and that the layer composition fraction resembles the single-layer case.

# B. Effect of Substrate/Interface Roughness on Resultant Magnetic Properties

In the fabricated micromagnets, with the exception of the first CoNiP layer which was formed on the seed Cu, all other CoNiP layers were formed on their respective underlying Cu interlamination layers. Therefore, every deposited Cu interlayer served as the effective substrate/seed for the subsequent CoNiP layer. Studies reveal that surface, substrate and interface roughness have crucial implications for the physics of thin films and multilayer structures [20]–[24]. The surface/interface condition of the Cu interlayer is hence crucial to the realization of the magnetic microlamination concept. In order to explore the underlying role of this interlayer, controlled experiments investigating the interface roughness



Fig. 7. Typical AFM scans of top layer Cu in  $[\text{CoNiP}(1\mu\text{m})/\text{Cu}(t_{Cu})]_5$  microlaminations with  $t_{Cu}$  equals (a) 0  $\mu$ m; (b) 0.25  $\mu$ m; (c) 0.7  $\mu$ m; (d) 1  $\mu$ m and for reference, AFM scans on (e) seed Cu as well as (f) 1  $\mu$ m single layer CoNiP.



Fig. 8. Top Cu layer roughness as a function of Cu interlayer thicknesses in  $[\text{CoNiP}(1\mu m)/\text{Cu}(t_{Cu})]_5$  microlaminations.

were conducted in  $[\text{CoNiP}(1\mu\text{m})/\text{Cu}(t_{Cu})]_5$  microlaminations with fixed CoNiP layer thickness  $(1 \ \mu\text{m})$  and various Cu layer thicknesses  $(t_{Cu} \text{ from } 0\text{-}1 \ \mu\text{m})$ . The number of pairs was chosen as five for an amplified effect (assuming that for insufficiently thick Cu layers the roughness would accumulate as the number of layers increases).

Figure 7(a)-(d) show some of the representative AFM scans of the very top layer (5<sup>th</sup>) of Cu in [CoNiP(1 $\mu$ m)/Cu( $t_{Cu}$ )]<sub>5</sub> microlaminations with various Cu interlayer thicknesses. The scan size is 15  $\mu$ m by 15  $\mu$ m. Figure 7(e), (f) provide the reference scans of the seed Cu and 1  $\mu$ m single-layer CoNiP surface topography, respectively. Detailed roughness data (arithmetic average  $R_a$  and root mean squared  $R_q$ ) is summarized in Figure 8. The lamination data were bound by two limits: the upper limit is associated with the 5  $\mu$ m single-layer CoNiP; and the lower limit is associated with



Fig. 9. A typical XRD spectrum of a  $[CoNiP(1\mu m)/Cu(0.7\mu m)]_5$  microlamination.

the seed Cu. As Cu interlayer thickness increases, both  $R_a$  and  $R_q$  decrease and plateau when  $t_{Cu}$  approaches 1  $\mu$ m. Two observations were made from Figures 7 and 8: (i) the 5  $\mu$ m single-layer CoNiP layer has the largest roughness (as expected); and (ii) increasing the Cu interlayer thickness has a monotonically increasing planarization effect, reducing the surface/interface roughness towards that of the seed Cu (which is expected to be the lower limit).

The corresponding crystalline structure, grain size, and preferential orientation of the [CoNiP( $1\mu$ m)/Cu( $t_{Cu}$ )]<sub>5</sub> microlaminations with various Cu interlayer thicknesses were examined by X-ray diffraction. A typical scan with large  $2\theta$ range  $(35-100^\circ)$  featuring a  $[CoNiP(1\mu m)/Cu(0.7\mu m)]_5$ microlamination is shown in Figure 9. The profile shown in Figure 9 exhibits neither additional nor missing peaks when compared with the single layer CoNiP case (Figure 4). Also, among microlaminations with various Cu interlayer thicknesses, the number and locations of the peaks remains unchanged (not shown, but can be partially seen from Figures 9 and 10). A finer scan with better signal to noise ratio was then carried out with smaller  $2\theta$  range (35°-55°) for various Cu interlayer thicknesses in order to precisely portray the peak of interest: hcp (100). The hcp (100) was the peak of interest not only because it represents the preferred inplane orientation but also because its large peak to background intensity ratio helps to reduce the possible fitting errors. The detailed representative finer scan profiles in Figure 10 show that as the thickness of the Cu interlayer increases, the intensity of the Cu (111) peaks (peak position at  $2\theta = 43.6^{\circ}$ ) increases along with a concurrent decrease in the CoNiP (100) intensity. The grain size in the direction normal to the film plane was estimated from peak hcp (100) using the Scherrer equation (1) [33].

$$D_{hkl} = K\lambda/(B_{hkl} \cdot \cos\theta) \tag{1}$$

Here  $D_{hkl}$  is the crystallite size in the direction perpendicular to the lattice plane (*hkl*), *K* is a dimensionless crystallite shape factor with 0.9 being a good approximation [33],  $\lambda$  is the wavelength of the X-ray,  $B_{hkl}$  is the full-width at halfmaximum (FWHM) of the peak (in radians), and  $\theta$  is the Bragg angle of the peak. For small nanocrystallites, the peak broadening from the specimen dominates other error sources such as instrumental and stress-induced broadening [34]. Neglecting these other broadening sources, the calculated grain



Fig. 10. Typical XRD spectra of  $[CoNiP(1\mu m)/Cu(t_{Cu})]_5$  microlaminations with various Cu interlayer thicknesses.



Fig. 11. Grain sizes of CoNiP films as a function of Cu interlayer thicknesses in  $[CoNiP(1\mu m)/Cu(t_{Cu})]_5$  microlaminations.

sizes as a function of interlayer Cu thicknesses are shown in Figure 11. The measured nanocrystalline grain sizes vary from 16 nm to 24 nm, corresponding well with the literature [29], [31]. By comparing with Figure 8, a close correlation between interface roughness and CoNiP layer grain size can be observed. Increasing the Cu interlayer thicknesses, and decreasing the interface roughness, reduce the grain sizes of the CoNiP magnetic layers by 31%. The dotted lines represent two limiting crystal size scenarios, with lower limit associated with 1  $\mu$ m single layer case and upper limit associated with the 5  $\mu$ m single layer case. The 1  $\mu$ m single layer case serves as the lower bound due to the fact that without multilayer roughness accumulation, the grain size of CoNiP should only be affected by the seed Cu, which is the smoothest interface seen from Figure 8. For the 5  $\mu$ m single layer, without any smoothing effect from interlamination Cu layer, its grain size is expected to be the largest. Note that the 5  $\mu$ m single layer case is essentially the same in structure as samples comprising a 0  $\mu$ m Cu interlayer. The slight difference in their grain sizes could be due to the fact that in the latter case, the



Fig. 12. (a) Maximum magnetic energy density; (b) coercivity; and (c) remanence as a function of Cu interlayer thicknesses in  $[\text{CoNiP}(1\mu\text{m})/\text{Cu}(t_{Cu})]_5$  microlaminations.

continuous grain growth seen in 5  $\mu$ m single layer is disrupted by removing and replacing the sample out of and into the plating bath every 1  $\mu$ m of deposition.

The corresponding magnetic of the properties  $[CoNiP(1\mu m)/Cu(t_{Cu})]_5$ microlaminations with various Cu interlayer thicknesses were characterized using VSM and the results are summarized in Figure 12. It can be seen that both energy density (Figure 12(a)) and coercivity (Figure 12(b)) curves follow an inverse trend with grain size (Figure 11) and surface roughness (Figure 8) plots: the increase of the Cu interlayer thicknesses (decrease of interface roughness and decrease of grain size) improves the coercivity (by 18%) and maximum magnetic energy density (by 29%). Comparatively, the remanence (Figure 12 (c)) is relatively unchanged (with deviation < 7%). Moreover, special attention should be given to the points of 0  $\mu$ m Cu interlayer in Figure 12. An important implication associated with these points is that solely interrupting electrodeposition periodically does not improve the performance of the micromagnet; incorporation of the planarizing interlayer is key to the success of the proposed process.

The interplay between grain size and coercivity of a crystalline magnetic material could be understood by a theoretical model proposed by Herzer [35]. This model suggests that the competition between local magnetic anisotropy energy and ferromagnetic exchange energy determines the magnetic properties of an assembly of grains. For large grains, magnetocrystalline anisotropy dominates and coercivity is proportional to 1/D where D is the grain size. For small grains, exchange interaction dominates and averages out locally fluctuating anisotropies so that only a small anisotropy net-effect exists. In the latter scenario, coercivity is proportional to  $D^6$ . The dividing gain size is equal to the ferromagnetic exchange length (2):

$$L_{ex} = \sqrt{A/K_1} \tag{2}$$

where A denotes the exchange stiffness and  $K_1$  is the magneto-crystalline anisotropy constant. For CoNiP, A is in the range of  $1.0 \times 10^{-6}$  to  $1.3 \times 10^{-6}$  erg/cm [36]  $(1.0 \times 10^{-11}$  to  $1.3 \times 10^{-11}$  J/m) and  $K_1$  is approximately 510 kJ/m<sup>3</sup> [2], [29]. The dividing grain size hence is calculated to be approximately 5 nm, smaller than the grain size range (16–24 nm) in our system, which explains the observed inverse correlation between the grain size and coercivity.

# C. Residual Stress Reduction on Laminated Micromagnets

Many hard magnetic films exhibit increased residual stress as their magnetic hardness increases [9]. Additional stresses could be introduced during deposition. For example, in the case of CoNiP, it is believed that hydrogen evolution during hypophosphite (Phosphorus source in CoNiP) oxidation and the parallel chemical reduction of the metal ion is a cause of the tensile stress in the films [29]. Stress releasing methods in electroplated magnetic films have been discussed in the past, including (not limited to) controlling the DC current density, the concentration of stress-relieving additives in the electroplating bath [10], and the use of pulse-reverse plating technique [11]. While effective in stress reduction as these methods could be, it is well known that magnetic properties (magnetic anisotropy, remanence, coercivity and etc.) of the plated films are highly sensitive to the plating conditions such as the current densities and bath additives [9], [19], [29], [31]. For some applications where the magnetic properties are predetermined (e.g. in-plane magnetization with high remanence), stress reduction via variation of plating parameters is fairly constrained. One alternative approach, is to stack the desired thin film with other auxiliary thin films with compensating stress conditions (very low stress or negative-signed stress as compared with the residual stress of the films of interest) into bilayer [15], sandwich [9] or multilayer [16] configurations, such that the overall stress condition in the deposited films is improved. Several previous studies [11], [17] have shown that the average stress of a multilayer structure can be calculated (by Equation (3)) as an individual-layer-thicknessweighted average stress under the assumption of small total film thickness (as compared with substrate thickness) and that individual layer material operates in the linear elastic regime. In Equation (3),  $\sigma_f$ ,  $\sigma_{CoNiP}$ , and  $\sigma_{Cu}$  are the average residual stress in the multilayer film, in the CoNiP component layers and in the copper component layers, respectively; T, t<sub>CoNiP</sub>, and  $t_{Cu}$  are the same as defined previously.

$$\sigma_f = \frac{\sum \sigma_{CoNiP} t_{CoNiP} + \sum \sigma_{Cu} t_{Cu}}{T}$$
(3)

To verify the application of equation (3) to the  $[\text{CoNiP}(t_{CoNiP})/\text{Cu}(t_{Cu})]_n$  system, the residual stress of the deposited single layer and microlamination films were estimated using Stoney equation (4):

$$\sigma_f = \frac{1}{6R} \frac{E_s}{1 - \nu_s} \frac{t_s^2}{t_f} \tag{4}$$

TABLE II Measured Residual Stress of Various Films

Film type	Thickness (µm)	Measured stress* (MPa)
CoNiP, single Layer	1	170
CoNiP, single Layer	5	189
Cu, single Layer	1	-1.65
[CoNiP/Cu] <sub>5</sub> , microlaminations	[(1µm)/(1µm)]₅	82.0

\* Positive stress being tensile

where  $\sigma_f$  denotes the average film stress;  $t_s$  and  $t_f$  are thickness of the substrate and film, respectively; R is the measured radius of wafer curvature by a stylus profiler,  $E_s$  and  $v_s$  denote the Young's modulus and Poisson's ratio of the substrate, respectively. The results of the estimated residual stress of various films are listed in Table II. The relatively low stressed (1.65 MPa, compressive) Cu interlamination layer reduces the measured residual stress of the [CoNiP(1 $\mu$ m)/Cu(1 $\mu$ m)]<sub>5</sub> microlamination (82.0 MPa, tensile) to approximately half that of a 1 $\mu$ m single layer (170 MPa, tensile) and less than half that of a 5 $\mu$ m single layer (189 MPa, tensile). Note that the measured stress value of the microlamination is very similar (< 2.5%) to the one calculated using Equation (3).

# D. Highly Laminated Permanent Micromagnets

The advantages of the micolaminated permanent magnets over their non-laminated counterparts can be demonstrated in two scenarios: 1) the total magnetic thickness  $(t_M)$  of the permanent micromagnets is of concern, and 2) the total thickness (T) is of concern. To differentiate between the achievable energies of these two scenarios,  $E_1$  will be defined as the total magnetic energy per unit area in the former scenario, while  $E_2$  will be defined as the total magnetic energy per unit area in the latter scenario.

Due to the high residual stresses often present in hard magnetic films as described above [9], [10], together with the fact that total strain energy in the films increases with increasing film thickness, film delamination [11] could limit the achievable total magnetic thickness (and therefore energy) of these small-scale integrated magnets. In this case, total magnetic thickness  $(t_M)$  instead of total thickness (T) is the relevant/limiting thickness for comparing the magnetic properties of laminated and non-laminated magnets. The strategy of lamination design in this case is to 1) increase the mechanical stability (e.g. reduce average stress), and 2) retain the magnetic properties of the component magnetic thin film as much as possible. Both of these can be achieved by relatively thicker Cu interlayers (Table II and Figure 12). Here,  $[CoNiP(1\mu m)/Cu(1\mu m)]_n$  microlaminated magnets up to n=80 have been fabricated. Figure 13 compares the in-plane maximum magnetic energy density  $((BH)_{max})$ as a function of total magnetic thickness  $(t_M)$  for various  $[CoNiP(1\mu m)/Cu(1\mu m)]_n$  microlaminations and CoNiP single layer films. It is evident that the  $(BH)_{max}$  of the CoNiP is well-retained in the microlamination configuration up to a total magnetic thickness of 80  $\mu$ m, while a single 80  $\mu$ m



Fig. 13. Comparison of the in-plane maximum magnetic energy density as a function of the total magnetic thicknesses  $(t_M)$  for various  $[\text{CoNiP}(1\mu m)/\text{Cu}(1\mu m)]_n$  microlaminations and CoNiP single layer films.

thick CoNiP film shows substantial degradation of  $(BH)_{max}$ .  $(BH)_{max}$  as high as 16.2 kJ/m<sup>3</sup> was achieved even at a large magnetic thickness of 80  $\mu$ m, an approximately 30% improvement over single layered CoNiP films of the same magnetic thickness. Because magnetic thickness  $(t_M)$  is the limiting thickness, in this case, the maximum achievable total magnetic energy per unit area  $(E_1)$  can then be defined in Equation (5). Hence, Figure 13 indicates that, in a given footprint, the laminated micromagnets show a higher achievable total magnetic energy over their single-layered counterparts. Although substantially improved overall, the properties of the magnetic layers are not completely retained in the microlamination, potentially due to incomplete planarization (Figure 8), which is accentuated as the number of layers (n) increases. Further increasing the interlayer thickness could potentially further improve the performance while simultaneously further reducing the residual stress, at the expense of the process duration.

$$E_1 = t_M \cdot (BH)_{max} \tag{5}$$

For applications where the overall thickness T is constrained (e.g., in the case of embedded MEMS where the MEMS module resides inside a silicon trench), the optimization tradeoff between allowable fill factor  $(\gamma)$ , tolerable average residual stress and achievable  $(BH)_{max}$  should be considered in the lamination design. As the total thickness (T) is constrained, the reduction of the total interlayer thickness  $(t_I)$ , though retaining less individual layer magnetic properties, will be compensated by the increase of total magnetic thickness  $t_M(t_M = T - t_I)$  and hence may act to enhance the total achievable magnetic energy per unit area  $E_2$ . The strategy of lamination design in this case is to balance this above mentioned opposing effect of the increased fill factor  $(\gamma)$ . A few examples of the possible designs of the lamination configurations are shown in Figure 14, which compares the inplane fill factor modified maximum magnetic energy density  $(\gamma * (BH)_{max})$  as a function of total thickness (T) for various  $[CoNiP(1\mu m)/Cu(t_{Cu})]_n$  microlaminations and CoNiP



Fig. 14. Comparison of the in-plane fill factor modified maximum magnetic energy density  $(\gamma^*(BH)_{max})$  measured as a function of the total thicknesses (*T*) for various  $[\text{CoNiP}(1\mu\text{m})/\text{Cu}(1\mu\text{m})]_n$  microlaminations and CoNiP single layer films.

single layer films. The fill factor of each example can be seen in the secondary axis on right of the figure. Note that total thickness (*T*) instead of total magnetic thickness ( $t_M$ ) is the relevant/limiting thickness in this case, and the fill factor ( $\gamma$ ) of single-layered magnetic films is 100%. Because total thickness (*T*) is the limiting thickness, in this case, the maximum achievable total magnetic energy per unit area ( $E_2$ ) can be defined in Equation (6). As can be seen in Figure 14, in a given footprint, laminated micromagnets with a proper choice of fill factor could also outcompete their single-layered counterparts in terms of achievable total magnetic energy in the case where total thickness is constrained.

$$E_2 = T \cdot \gamma \ (BH)_{max} \tag{6}$$

#### VI. CONCLUSIONS

A microlaminated MEMS permanent magnet enabled by a fully-integrated, CMOS-compatible fabrication approach utilizing the technique of sequential multilayer electroplating has been successfully demonstrated. Due to the preserved individual component magnetic layer properties and reduced average stress possessed in these films, the thick, microlaminated magnets showed an improved total magnetic energy as compared with their non-laminated counterparts. The key to retain the superior magnetic properties of thin films in thick laminations is the low surface roughness of the interface between the magnetic layers, which in turn reduces the grain size and improves the coercivity of the magnetic component layers. Depending on the application of these permanent micromagnets in various MEMS devices, individual component layer (magnetic and non-magnetic) thicknesses and hence the fill factor could be adjusted to balance the achievable  $(BH)_{max}$ , tolerable average residual stress and allowable total thickness constrains if required. The demonstrated fabrication approach has the potential for application to other permanent magnetic material systems with higher intrinsic properties to further increase the total magnetic energy possessed in these micromagnets.

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