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ABSTRACT
There is an overgrowing interest in the synthesis of high performance micro magnets due to their wide application areas in MEMS, microfluidics, and micro-robotics. In addition, they possess great potential to become a model system for the development of next-generation hard magnets. In this study, NdFeB/Fe hybrid micro-magnets have been synthesized by a photolithography technique. NdFeB micro and Fe nano powders in the form of nano-thin flakes have been synthesized by one step surfactant assisted planetary ball milling. The fabricated nano-thin flakes with different lateral sizes have been obtained by varying the milling time. Room temperature coercivities of up to 3.1 kOe for NdFeB has been achieved after milling for 8 h. Photoresist and powders of NdFeB and Fe have been mixed at various powder-to-resist ratios. SU-8 has been chosen as the photoresist due to its resistance to chemicals and its suitability to be utilized in MEMS technology. Micro islands of SU-8/NdFeB/Fe have been prepared according to the manufacturer’s recommendations for pure SU-8. The hybrid micromagnets in pillar form have been successfully produced. The evolution of magnetic, structural and microstructural properties of these micro magnets will be reported. Successful fabrication of hybrid micro-magnets using the methodology prescribed in this work can pave the way for the development of next-generation MEMS.

I. INTRODUCTION
High performance nano and micro magnets are the key parts of MEMS, microfluidics and robotic swimmers used in wide range of applications from computer to biomedical industry. In common practice, magnetic materials are incorporated into MEMS and other applications via physical/chemical vapor deposition, ergo in thin film form. However, this morphology hinders with the magnetic properties of the material. Yet another method to incorporate magnetic materials is loading them into curable polymers. This method is preferable since specifically developed powders of ferromagnetic alloys with best magnetic properties for permanent magnet production could be used, ergo better magnetic performance could be achieved for applications. Among different kinds of ferromagnetic materials, NdFeB is the most prominent of all due to its high energy product. Surfactant assisted ball milling is a proven technique to synthesize anisotropic NdFeB powders in large quantities. Maximum energy product achieved in the laboratory environment is limited to 60 MGOe for NdFeB magnets. In order to enhance the magnetic energy product, magnetically soft material should be introduced in to the system, which will result in exchange coupling between soft and hard ferromagnets. In this study, NdFeB powder, in the form of nano thick and laterally micron sized flakes, and Fe nanoparticles (∼20 nm) was mixed with the photosensitive resist, SU8, to synthesize NdFeB/Fe loaded micro magnets by photolithography technique. Recently, successful compatibleness of SU8 with nano and micro particles has been shown. SU8 has been chosen for its rigidity, biocompatibility and resistant to chemicals.

II. EXPERIMENTAL
NdFeB flakes and Fe nanoparticles were synthesized by planetary ball milling. NdFeB chunks have been manually grinded down to 300 μm. Starting powder for Fe is Merck 103815.1000. Prepared powder was loaded in a milling jar (Vanadium Steel) alongside with various size steel balls (10:1 ball to powder ratio), hexane and oleic acid.
In order to synthesize NdFeB flakes, 300-micron size powder is ball milled under heptane and oleic acid for 2-8h. It has been observed that the coercivity increases by increasing the milling time and the maximum room temperature coercivity of 3.1 kOe has been reached after 6 hours of ball milling. [Figure 1] Coercivity increase is attributed to grain size refinement thus fewer defects are expected per particle. Further milling to 8 h did not show any considerable increase in coercivity (3.2 kOe) and a slight increase in remanence was observed. Powders milled for 6 h has been used for further experiments due to the better morphology observed in the synthesized flakes. It should also be noted that, as the milling of the powder continues, its polycrystallinity (number of randomly oriented grains) increases thus magnetization at the maximum applied field (2 T, substantially lower than anisotropy field, $H_A=7T$) drops.

III. RESULTS AND DISCUSSIONS

In order to synthesize NdFeB flakes, 300-micron size powder is ball milled under heptane and oleic acid for 2-8h. It has been observed that the coercivity increases by increasing the milling time and the maximum room temperature coercivity of 3.1 kOe has been reached after 6 hours of ball milling. [Figure 1] Coercivity increase is attributed to grain size refinement thus fewer defects are expected per particle. Further milling to 8 h did not show any considerable increase in coercivity (3.2 kOe) and a slight increase in remanence was observed. Powders milled for 6 h has been used for further experiments due to the better morphology observed in the synthesized flakes. It should also be noted that, as the milling of the powder continues, its polycrystallinity (number of randomly oriented grains) increases thus magnetization at the maximum applied field (2 T, substantially lower than anisotropy field, $H_A=7T$) drops.

![Figure 1. Hysteresis loops of NdFeB powder milled for 2 to 8 hours.](image1)

![Figure 2. a. SEM image of 6 h milled NdFeB flakes, b. HRTEM image of NdFeB nanoparticles and b inlet. corresponding SAED image, c. TEM bright field image of Fe nanoparticles.](image2)
NdFeB flakes have been successfully synthesized after 6h of planetary ball milling. (Figure 2 a) Average thickness and the lateral sizes of the flakes are 100 nm and 1 μm, respectively. In addition to flakes, nanoparticles with an average size of 10 nm has been observed in the upper solution. (Figure 2 b) Crystal structure of the nanoparticles is confirmed to be the 2:14:1 FCT phase of NdFeB by selected area electron diffraction. (Figure 2 b and inlet). Surfactant assisted planetary ball milling of 10 μm Fe powder for 12 h yield Fe flakes in slurry (data not shown) and Fe nanoparticles with 20 nm average size in upper solution. (Figure 2 c)

Before mixing powders with SU8, exchange-coupling performance between NdFeB flakes and Fe nanoparticles has been evaluated. 5-20 wt. % of Fe nanoparticles were mixed thoroughly, and hysteresis loops have been measured. Although slight increase in saturation magnetization was observed at higher concentrations, kink in the demagnetization curve was also spotted indication of poor coupling. (Figure 3) Inhomogeneous coverage of NdFeB flakes with Fe nanoparticles could be the reason behind the poor coupling of the magnetically hard and soft grains.\(^\text{14}\) NdFeB flakes with 5 wt.% Fe nanoparticles have been used for further experiments.

Micro pillars of NdFeB/Fe loaded SU8 (10 – 30 wt.% powder) has been prepared by photolithography technique. Compared to the pure SU8 (Figure 4 a), 30 wt.% NdFeB/Fe loaded SU8 pillars’ outer surface is rippled thus the pillars are deformed due to the weight of the powder. However overall shape of the pillars is acceptable to be used in aforementioned applications.

Particle distribution inside the micro pillars for 10, 20 and 30 wt. % loadings were observed by light microscope. While powders are concentrated on the outer edges of the pillars for the 10 wt.% loading, powders start to accommodate the infrastructure of the pillars for the 20 wt.% (Figure 5 a and b) Best loading rate is 30 wt.% as seen by the Figure 5 c. At higher loading rates, powder and SU8 becomes immiscible, ergo photolithography was impossible.

Coercivity is conserved after the encapsulation of powders into the SU8 matrix. (Figure 6) Although the micro magnets synthesized...
here lacks the magnetic properties achieved in previous works, micro magnets with considerably high room temperature coercivity were successfully synthesized.

IV. CONCLUSION

In conclusion, NdFeB/Fe loaded SU8 micro magnets with high room temperature coercivity of 3.1 kOe have been successfully synthesized by photolithography. Surfactant assisted planetary ball milling was used to produce NdFeB flakes with high aspect ratio and Fe nanoparticles with an average size of 20 nm. Exchange coupling performance between NdFeB flakes and Fe nanoparticles were studied. Up to 30% loading of NdFeB/Fe powder is achieved. Synthesized micro magnets could be used in next generation MEMS, sensors and microfluidic systems. Moreover, this system could be used as model systems to study particle-particle interactions. There is a great window of opportunity for improvements consequently further developments are in order.

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