Synthesis of Nd-Fe-B/Fe hybrid micro-magnets

Cite as: AIP Advances 9, 125139 (2019); doi: 10.1063/1.5130412
Presented: 5 November 2019 • Submitted: 3 October 2019 •
Accepted: 26 November 2019 • Published Online: 26 December 2019

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Note: This paper was presented at the 64th Annual Conference on Magnetism and Magnetic Materials.
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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,[1] microfluidics[2] and robotic swimmers used in wide range of applications from computer to biomedical industry.[3,4] In common practice, magnetic materials are incorporated into MEMS and other applications via physical/chemical vapor deposition, ergo in thin film form.[5–8] 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.[4] Surfactant assisted ball milling is a proven technique to synthesize anisotropic NdFeB powders in large quantities.[9,10] Maximum energy product achieved in the laboratory environment is limited to 60 MGOe for NdFeB magnets.[11] 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.[12–14] 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.[15–18] SU8 has been chosen for its rigidness, biocompatibility and resistant to chemicals.[19]

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

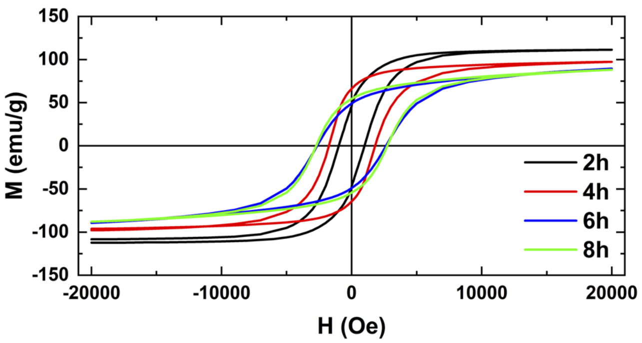

FIG. 1. Hysteresis loops of NdFeB powder milled for 2 to 8 hours.

acid. Jar was then loaded to the planetary Ball Mill (MTI Corporation SFM-1 Planetary Ball Mill) and milled for 2-8 hours (12 h for Fe) at 700 rpm. In surfactant assisted ball milling, the upper solution and the slurry contain the suspended nanoparticles and the flakes, respectively. Fe nanoparticles has been collected and dried from the upper solution. Synthesized nano thick NdFeB flakes were cleaned from extra oleic acid by centrifuging under pure ethanol four times. Various ratios of NdFeB flakes to Fe nanoparticles were mixed: 5 to 20 wt. %. Afterwards, SU8 and NdFeB/Fe were mixed thoroughly; 10, 20 and 30 wt. % of NdFeB/Fe. NdFeB/Fe loaded micro-pillars have been prepared by photolithography. Process is as follows: 100 mm wide Si/SiO_2 (100) wafers were spin-coated with SU8 photoresist followed by seriatim soft bake at 95°C, UV exposure though a mask and post exposure bake at 95°C. Photoresist was then developed with SU8 developer, leading to an array of square or circular micro features. Morphology and crystallographic analyses of the samples were performed using Jeol JSM 6010 SEM, JEOL JEM-ARM200CFEG UHR-TEM and light microscope. Magnetic measurements at room temperature were made with an EZ9 Microsense VSM. XRD measurements were made with a Bruker D2 diffractometer.

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.[9] 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.

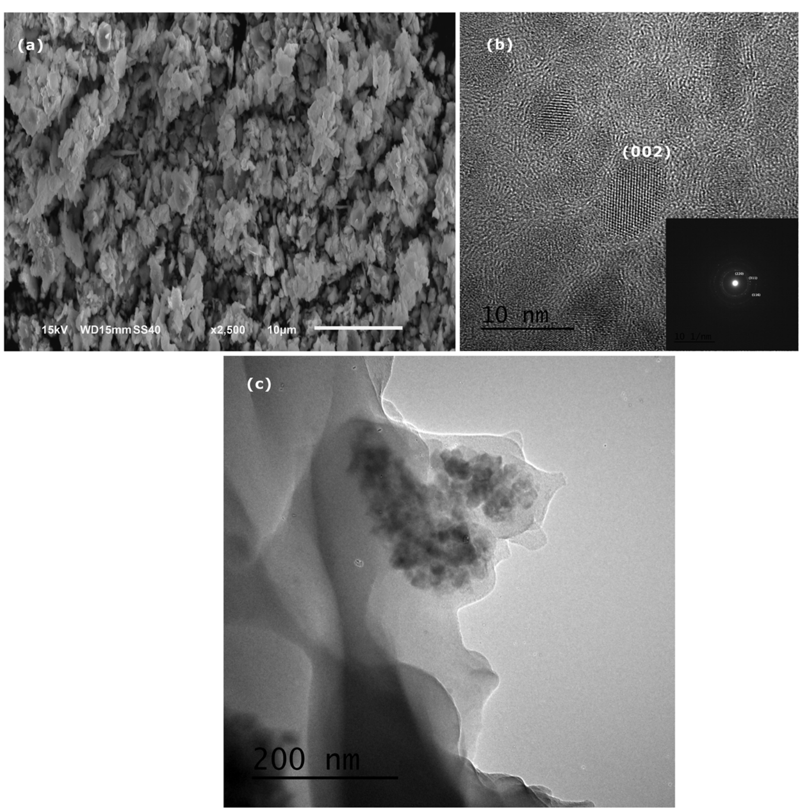

FIG. 2. a. SEM image of 6 h milled NdFeB flakes, b. HRTEM image of NdFeB nanoparticles and b inlet. corresponding SAED image, c. TEM bright field image of Fe nanoparticles.

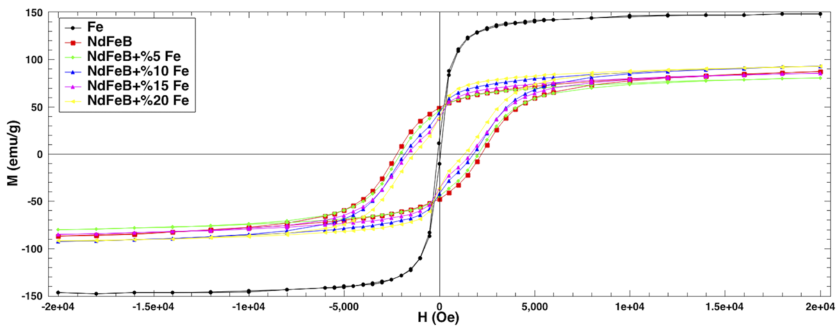

FIG. 3. Hysteresis loops of Fe, NdFeB, and NdFeB:Fe mixtures; 5-20 wt. %.

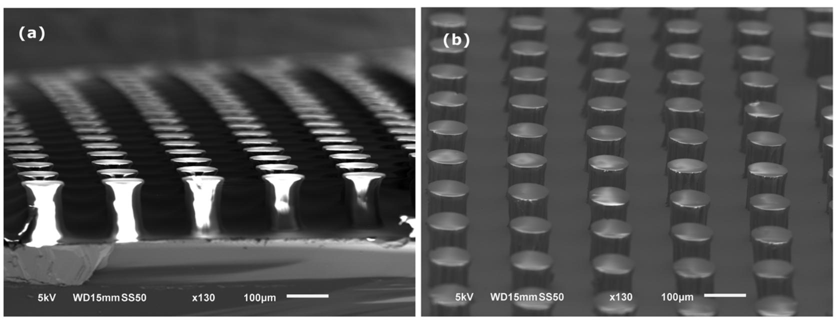

FIG. 4. SEM image of 50 µm diameter pillars of a. SU8, b. 30% NdFeB/Fe powder loaded SU8.

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.[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

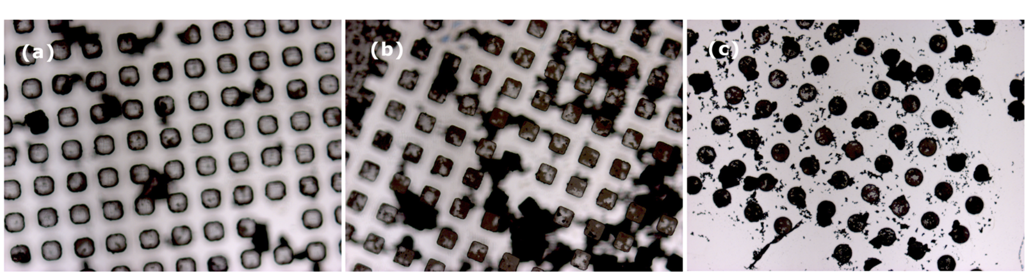

FIG. 5. Light Microscopy images of a. %10, b. %20 and c. 30% NdFeB/Fe loaded SU8 pillars.

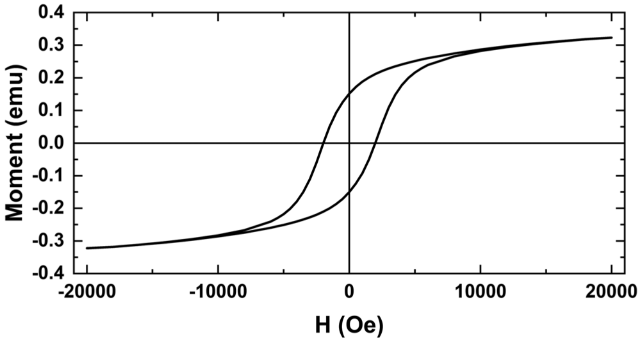

FIG. 6. Hysteresis loop of 30% NdFeB flakes loaded SU8 micro magnets.

here lacks the magnetic properties achieved in previous works,[5,8] 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 plenatry 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.

ACKNOWLEDGMENTS

Authors would like to thank Dr. Gülcan Çorapcıoğlu (Sabancı University Nanotechnology Research and Application Center), and Prof. Dr. Ilker Küçük (Uludağ University) for TEM and VSM studies, respectively. This work was supported by TUBITAK projects: 118F319 and 116C080.

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