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Recyclying of Nd-Fe-B alloy for fabrications of Three-Dimensional Magnetic Micro-components

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Abstract*— *This paper presents a new fabrication process for producing magnetic microcomponents. The process is based on micro powder injection moulding technology, while the micro moulds are produced using microelectromechanical systems (MEMS) technology. The process involves (1) fabrication of polydimethylsiloxane (PDMS) micro moulds from SU-8 masters, which are produced using UV photolithography process; (2) mould filling with magnetic powder and demoulding; and (3) sintering of moulded components in vacuum. The proposed process has been used to sinter Nd-Fe-B microcomponents successfully without using binder system. This research proposes a new approach to fabricate 3-D magnetic microcomponents to meet the needs in applications where magnetic field required.

Keywords*— *Magnetic microcomponents, MEMS, PDMS micro moulds, SU-8 moulds, Nd-Fe-B, hydrogen decrepitation

I. Introduction

Permanent magnets experienced a rapid development throughout the 20th century, with the maximum energy product (a figure of merit for permanent magnets) rising from < 10 kJm-3 to > 400 kJm-3. Advances in processing and the discovery of new materials have led this development. The most recent, and highest maximum energy product, permanent magnet material was discovered in 1984 [1,2] and is based on a ternary alloy: NdFeB. A higher maximum energy product means that a smaller permanent magnet can be used to perform a certain function, such as generating torque in a motor. Thus the introduction of NdFeB based permanent magnets has allowed, for example, the miniaturisation of hard disk drives where permanent magnet motors are used to spin the disk and scan the head across the surface of the disk.

Conventionally sintered NdFeB permanent magnets are produced by powder metallurgy. The alloy is exposed to hydrogen; causing hydrogen decrepitation (HD) of the alloy and the formation of a powder with ~100µm particle size, which is further milled to ~5µm particle size. The particles are single crystals, which have a preferred direction of magnetisation, that can be aligned in a magnetic field and pressed to give a green compact that is sintered under vacuum at ~1080°C for 1 hour. The alignment process is essential for producing the optimum magnetic properties; the maximum energy product of a fully aligned magnet can be up to four times greater than an isotropic, unaligned magnet.

There have been many reports (a recent review can be found in reference 3) of potential applications of permanent magnets in MicroElectroMechanical Systems (MEMS), sometimes referred to as MAGnetic Micro-Actuators and Systems (MAGMAS). These applications include micro-motors, micro-switches and micro-generators. Almost all of these reports use magnets produced by wire electro-discharge machining from large blocks of NdFeB or SmCo magnets. This is not suitable for producing a large number of components so alternate routes are being sought, including screen printing [4], sputtering [5] and pulse laser deposition [6]. As far as the authors are aware the work presented here is the only work to attempt the sintering of near net shape microcomponents from NdFeB powder.

II. Experimental Procedure

A. Mould Fabrication

The fabrication process to produce magnetic microcomponents using Nd-Fe-B alloy powder starts from making master moulds. The master mould is relatively rigid, of high precision and usually fabricated using MEMS technology, such as deep reactive ion etching (DRIE) and X-ray exposure on SU-8 photoresist. SU-8 UV lithography has gained much progress in recent years. 1 mm thick micropatterned master mould was fabricated through UTSP. More details can be found in references.[7-8] Figure 1 & 2 show images of 1mm thick SU-8 microgears in SU-8 master and PDMS soft moulds.

The gear is 1 mm in height and 2.9 mm in diameter, with two through holes in the middle. The SU-8 master moulds have very smooth surfaces that can be replicated to the negative moulds.[9]

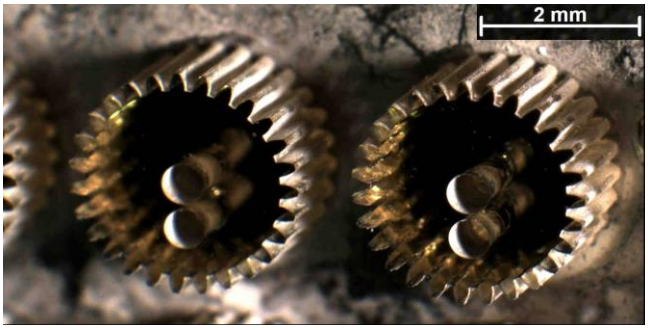

Fig. 1. SU-8 master moulds

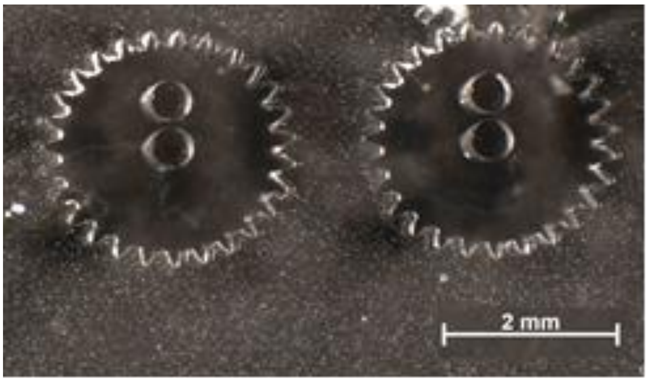

Fig. 2. Soft PDMS moulds replicated from SU-8 master

The microgear was used as the master mould and a negative soft mould was produced from it. A widely adopted soft moulding technique is using elastomer PDMS to pattern the micrometer and sub-micrometer sized structures. Further details about the PDMS moulds can be found in literature.[8,10-15]

B. Powder Preparation

The starting material was, in the form of sintered magnets with commercial composition given in table I. Their composition was obtained by inductive coupled plasma (ICP). Magnets were placed into stainless steel hydrogenation vessel, which was then evacuated using rotary pump to a pressure of 1×10-1 bar. The hydrogen decrepitation (HD) process was then carried out by admitting hydrogen at 25oC to a pressure of 2 bar, resulting in decrepitation of the sintered material.

TABLE I. ICP MEASUREMENTS OF THE TYPICAL COMPOSITION.

Nd at %	Pr at %	Dy at %	B at %	Nb at %	Al at %	Fe at %
13.98	0.06	0.63	6.44	0.36	0.69	77.81

C. Micro Mould Filling Methods

TABLE II. MOULD FILLING METHODS USED IN THIS EXPERIMENTS.

Method	Filling Process
A	Pour powder slurry on PDMS
B	Sink PDMS mould into the slurry
C	Repeat method B on a bulk magnet
D	Tamp powder into SU-8 mould
E	Fill up rubber tube with powder

The magnetic powders are then prepared in two types, i.e. with and without binder, to fill up the micro moulds. Table II shows the different types of mould filling methods with simple explanation.

For methods A ~ C, the powders and binder were mixed with acetone to form slurry. The mixing process was carried out for 2 hours using a magnetic stirrer(Hanna Instruments HI180H/D). When the adhesive binder is mixed with the powders in acetone, it is diluted and its adhesion is lost temporarily. When the mixture is dry, the adhesive binder regains its bonding characteristics so the moulded components can maintain their patterns. No additional debinding process was required, as the binder evaporates in-situ during the sintering process in the furnace.

Then the cavity of patterned micro mould was filled up with the powder. The following methods were experimented in the mould filling process: A, pouring the powder premix on to the moulds under gravity; B, immersing PDMS mould into the mixture; C, process method B on a bulk of magnet, so the powder will compacted into the patterned mould; D, fill up dry powder into SU-8 mould and tamp down gently; E, fill up rubber tube with powder and place mould on top of them and compact tube under isostatic pressure. Extensive experiments have been carried out to fill the micro moulds using the five methods. It is found that methods A and B tend to leave some holes unfilled in the moulds and cracked during sintering. Cracks are avoidable when green structures have thick substrate like E. Method C results lined up powder to vertical direction all over rather then filling up the mould. Handling magnetic powder within magnetic field was not handy. Also, produced green structure has rough surface profile. Method D is unlikely using SU-8 mould rather than soft PDMS mould. In this method, when powder is compacted into the mould, both mould and powder are undergone sintering process. This process consists of two steps. The first step is pyrolysis process which burn SU-8 mould out at the temperature below 480°C followed by the second step which is sintering of the remained powder. As the result of this process, the SU-8 mould was not only completely burned out, also caused great distortion, deformation, and cracks on green structures. More details regarding SU-8 pyrolysis process can be found in references.[12] The method E method resulted obviously better mould fillings than the other four moulding methods. In isostatic pressure method, the powder and soft PDMS mould are pressed isostatically at a pressure of 60 MPa for 10 min. Then the PDMS mould was carefully removed to achieve the powder components with a good shape retention and low porosity level. Since the fabricated green structure retains its patterned shape without any assistant of binder, less shrinkage ratio compare with others which using binder system is expected after sintering.

D. Demoulding and Sintering

Once the powder and mould are placed into the rubber tube, the fine powder was then aligned by pulsing two times in a 4.5T magnetic field and compacted. The green component was achieved by peeling off the soft PDMS mould. Next, the patterned component was placed inside a furnace under vacuum and heated to 1080°C, 10°C/min. The sample was held for 1 hour at designated temperature and taken out after the furnace was cooled down to room temperature.

III. Results and Characterisations

Figure 3 shows a sintered magnetic microgear. It can be seen that a relatively good shape retention of the microgear has been achieved after sintering.

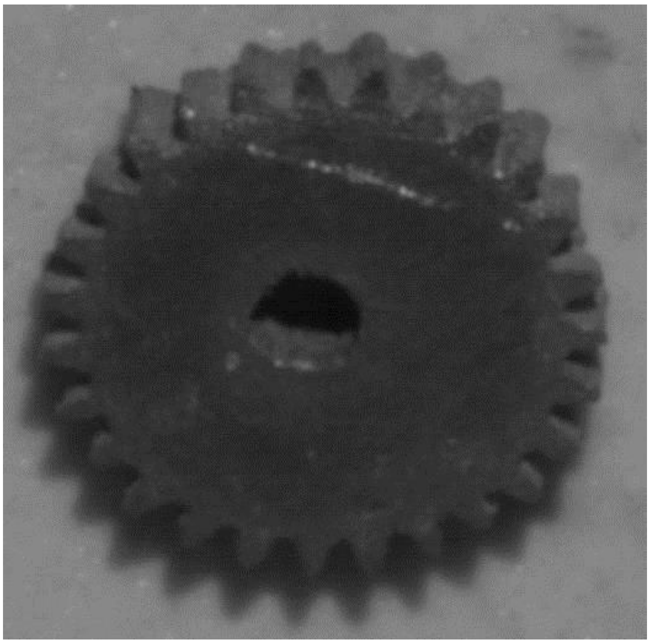

Fig. 3. Sintered microgear

The magnetic characterisation of the HD sintered microcomponents was carried out using vibration sample magnetometer (VSM) which is shown in figure 4.

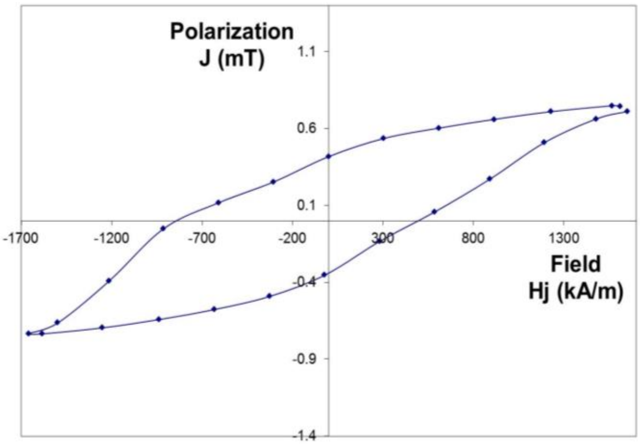

Fig 4. Typical hysteresis loop of sintered microcomponents, determined by VSM.

The second quadrant curve used to describe the quality of the magnetic sintered microcomponents have a few important parameters: remanence Br (T), intrinsic coercivity iHc (kA/m), inductive coercivity bHc (kA/m), and maximum energy product BHmax (kJ/m3). Squareness factor (SF) was use to determinate the stability of the magnets while undergoing demagentisation and defined as the applied field at 90% of the remanence Hs divided by the intrinsic coercivity. Measurements were performed after saturation in a pulsed field of 4.5T. A summery of magnetic properties is given in table 1. The density of the sample was calculated using the Archimede's principle where by a body submerged in a fluid experiences a buoyant force equal to the weight of fluid displaced.

IV. Conclusions

A new fabrication process is presented for producing magnetic microcomponents from Nd-Fe-B powder via sintering. The technology is based on □PIM but extended with the help of MEMS technology. The process involves (1) fabrication of micro master moulds in SU-8 using UV lithographical process and PDMS negative moulds are made from the SU-8 masters; (2) filling the moulds with the magnetic powder along with compaction using isostatic pressure and peeling off the PDMS moulds when the patterns are consolidated; (3) sintering patterned components under vacuum. Nd-Fe-B microcomponents have been successfully produced following this process.

The use of soft PDMS moulds makes the demoulding easier in avoiding the damage of the green patterns by the moulds. The investigation has also achieved progress in identifying the best mould filling method. After repeated experiments, using isostatic pressure method stands out. It basically avoids the forming of air bubbles and increases the density and mechanical strength of the components. The most advantage of using isostatic pressure for shape patterning is by-passing using of binder system. Applying isostatic pressure for powder compaction is eliminating the worries regarding chemical reaction between powder particles and binder during sintering and requiring no additional debinding process. Also, it decreases shrinkage ratio during sintering process. Although isostatic pressure method was most secure method among introduced mould filling methods, it caused a great shrinkage to both elastic moulds and green patterns during powder compaction. This shrinkage ratio will be calculated and enlarged moulds will be designed to fabricate net-shape magnetic microcomponents.

The HD powder was employed successfully produce fully dense, sintered magnetic micro components and the magnetic properties were determined by means of a vibrating sample magnetometer. Square loop was obtained and it was found that the remanence value was significantly reduced to those of the original magnets, probably due to poor alignment of the powder. The powder flowability is an important factor affecting the alignment degree, remanence and energy product. The main factor affecting powder flowability was mould filling and magnetic aggregation. The iHc value was significantly smaller than that of the expected NdFeB magnet (~ 1272kA/m) and this can be associated with the oxide surface of the micro components. These results have been discussed in terms of a possible method of producing magnetic microcomponents using an HDtype process through the use of MEMS technologies. The proposed process shows a new way of fabricating three-dimensional magnetic microcomponents.

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