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Precision Formed Micro Magnets: LDRD Project Summary Report

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Prepared by
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Precision Formed Micro Magnets: LDRD Project Summary Report

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Abstract

A microfabrication process is described that provides for the batch realization of miniature rare earth based permanent magnets. Prismatic geometry with features as small as 5 microns, thicknesses up through several hundred microns and with submicron tolerances may be accommodated. The processing is based on a molding technique using deep x-ray lithography as a means to generate high aspect-ratio precision molds from PMMA (poly methyl methacrylate) used as an x-ray photoresist. Subsequent molding of rare-earth permanent magnet (REPM) powder combined with a thermosetting plastic binder may take place directly in the PMMA mold. Further approaches generate an alumina form replicated from the PMMA mold that becomes an intermediate mold for pressing higher density REPM material and allows for higher process temperatures. Maximum energy products of 3-8 MGOe (Mega Gauss Oersted, $1 \text{ MGOe} = 100/4\pi \text{ kJ/m}^3$) are obtained for bonded isotropic forms of REPM with dimensions on the scale of 100 microns and up to 23 MGOe for more dense anisotropic REPM material using higher temperature processing. The utility of miniature precision REPMs is revealed by the demonstration of a miniature multipole brushless DC motor that possesses a pole-anisotropic rotor with dimensions that would otherwise prohibit multipole magnetization using a multipole magnetizing fixture at this scale. Subsequent multipole assembly also leads to miniaturized "Halbach arrays", efficient magnetic microactuators, and mechanical spring-like elements which can offset miniaturized mechanical scaling behavior.

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INTRODUCTION

A fundamental area in Micro Electro Mechanical Systems (MEMS) research pertains to the expansion of available materials for integration into planar batch processing sequences. Microsystems incorporating magnetic devices have been largely hampered by these material base constraints and would particularly profit from the availability of a permanent magnet material. The feature of permanent magnets, and in particular rare-earth based permanent magnets, which makes their use compelling in the micro-domain is that their magnetization remains independent of scale. This situation is in contrast to electromagnetic coils where constraints due to current density limits do not allow one to maintain a given magnetic field with a particular scaled coil. Even superconductors suffer the same limitation that follows from critical current density limits. Many uses of permanent magnets on the micro-scale, however, require that they be fabricated to small dimensions and corresponding scaled tolerances. Fabricating permanent magnet materials conventionally in a serial mode is not only difficult and expensive, but in situations where micro-size multipole magnets are needed, may not even be practical for the same reasons just mentioned: establishing multi-oriented high magnetic fields in a small volume is prohibited by coil scaling which is the means used to magnetize permanent magnet material. This is particularly true for rare-earth permanent magnets (REPM) that possess the largest maximum energy products (MEP) of any magnet material and require magnetizing fields greater than 35,000 Oe at room temperature. Microactuators and micromotors acquire substantial performance gains from these high MEP materials which has been indirectly revealed by the miniaturization of magnetic recording devices and the increase in use of brushless DC machines for small sized applications. Another means of exploiting permanent magnet properties at small scale is to use them to offset mechanical spring scaling limitations. In this sense, a permanent magnet may be used as a passive element to provide a holding force or alter the spring constant of a mechanical flexure for example.

PROCESS

Batch microfabrication approaches may be fundamentally categorized as either subtractive or additive processes. Patterning involves the transfer of a 2-dimensional geometry definition into a film of some thickness or a 2+ dimensional extrusion into a substantially prismatic geometry. For many devices including especially mechanical components the depth of patterning becomes a concern and a resulting vertical aspect ratio may be defined as the unavoidable amount of lateral run-out per structure thickness.

The result is a limit on the precision of a prismatic component. For a subtractive process wherein the material desired to be structured is selectively removed, the resulting aspect ratio is a function of the etch directionality or anisotropy for which a variety of wet and dry (plasma) chemistries have been developed for certain materials. Metals, and in particular magnetic metals, remain difficult to accurately pattern with this technique, and thus an additive approach is commonly used.

Additive microfabrication exploits molding on the microscale most commonly with a polymer photoresist as the mold material. A further description of this mode of processing is provided in the paper of Appendix A. The photoresist may be patterned by numerous techniques such as UV lithography common to semiconductor planar processing. Approaches to increase the depth of exposure require commensurate increases in photoresist absorption lengths. For structures several hundred microns or more thick a particularly exceptional light source is synchrotron radiation produced from a charged particle storage ring. This radiation extends from UV to over 20keV x-rays and has the additional qualities of high intensity ($\geq W/cm^2$) and high degree of collimation ($<1mrad$). This light source, available as a foundry-like service, when used in conjunction with a suitable x-ray mask provides for thick replication of sub-micron planar features. A suitable x-ray photoresist for this type of deep x-ray lithography (DXRL) is poly methyl methacrylate (PMMA or plexiglas) with corresponding absorption lengths for 5 keV x-rays of 100s of microns. The addition to this exposure step of a solvent or developer that dissolves exposed PMMA regions with high selectivity allows the practically perfect projection of an essentially 2-dimensional geometry into prismatic (2+-dimensional) thick geometry. The resulting PMMA mold may be used directly for plastic components or as is most common for an electroforming mold. In this case, a metal is electrodeposited on a seed layer residing underneath the PMMA mold to accurately replicate the complement of the PMMA geometry. This processing sequence with the addition of an injection molding step that additionally replicates plastic molds from an electroformed master was coined the acronym "LIGA" by researchers at the Nuclear Research Center in Karlsruhe, Germany. To take further advantage of the precision mold that is obtained, other types of mold filling have been examined in this work that allow for batch precision miniature permanent magnet formation. Two fundamental techniques have been identified and may be categorized as low and high temperature processes.

A straightforward approach is possible with REPM material in bonded form. This is an extension of the common procedure of using REPM powder with a binder plastic material to mold composite permanent magnets resulting in net-shaped geometry without direct machining but with a volume fraction reduction in MEP. An additional trade-off exists between mechanical strength and MEP as a function of volume fraction of binder. Practically, MEPs as large as 10 MGOe are possible with 10%/vol binder material. Additionally, REPM powder is available in anisotropic as well as isotropic form that if aligned during processing may lead to MEPs for bonded REPMs as large as 19 MGOe.

The basic process involves first grinding REPM powder to a suitable size in a ball mill while prohibiting oxidation. Since energy product and packing density degrade with

particle size, the optimum particle size is as large as possible commensurate with the narrowest mold feature. Subsequent process details are specifically outlined in the patent disclosure listed in Appendix B and thus an overview is provided here. A mold form is generated using DXRL and a PMMA film that is adherent to a flat substrate material such as glass, silicon or alumina, for example. The substrate material is also provided with a sacrificial layer that may be etched selectively with respect to the permanent magnet material and thereby eventually release the formed permanent magnets.

The resulting REPM powder is then mixed with 10-30%/vol binder which is typically an epoxy. The powder may be magnetized prior to mixing or left in an unmagnetized state. Mold filling with a mixture in the magnetized state occurs in a magnetic field of roughly 2000 Oe by properly orienting the mold in the desired direction of magnetization. This strength of field was determined empirically as it was found that a magnetic field of the order of 1000 Oe is just sufficient to re-align the magnetic particles while mixed in the un-cured epoxy. After casting a uniform layer of the mixture over the mold, the mixture and substrate combination are uniaxially pressed at pressures between 10 to 20 ksi. The epoxy is cured typically at room temperature and the resultant composite is planarized back parallel to the substrate surface with lapping. At this point an unmagnetized composite is magnetized in a uniform pulse field of at least 35,000 Oe. The magnets are released by dissolving the PMMA mold with a solvent and then chemically etching the underlying sacrificial layer.

Sintered bulk REPMs are available with MEPs over 40 MGOe. To obtain these results in batch microfabricated form, extensions to the previous bonded process were made to chiefly allow for higher temperature processing. Experiments to fabricate REPMs by directly sintering milled powder in PMMA molds were largely unsuccessful. This approach was, however, found to be suitable for ferrite processing and MnZn ferrite geometry with sub-100 μ m features were successfully molded directly.

Hot pressing techniques have proven more fruitful and properly done yield shrinkage only in the press direction which in this case is dimensionally controlled with a lapping operation. This process requires, however, a material that can accurately replicate a DXRL defined PMMA mold, subsequently withstand hot press pressures and temperatures (up to 15 ksi at up to 1000°C), and be compatible with release etch procedures. Electroformed metals that are commonly electroformed from PMMA molds such as nickel and copper will undergo significant plastic deformation at these conditions. Nevertheless, copper was found to be useful to improve the mechanical integrity over PMMA for use at moderate temperatures and for a stronger bonded REPM mold material. Copper etchants based on ammonium hydroxide also have negligible effect on REPM materials.

To accomplish yet higher temperature processing, hot pressing molds constructed from ceramic and glass-ceramic composites were pursued (see Appendix F). The prime issue is the avoidance of shrinkage of the ceramic from an initial complementary mold. A series of techniques were examined beginning with pressing alumina powder into PMMA molds, burning off the PMMA and bisque firing to strengthen the alumina. A

release layer is provided via the evaporation of a thin film of copper onto the alumina molds. The two problems that occurred with this approach are cracking and excessive shrinkage of the fired alumina form as well as difficulty in uniformly covering the alumina sidewalls with a sputtered or evaporated metal sacrificial layer. An alternative, therefore, was investigated involving a colloiddally bonded alumina material. Colloidal silica particles were used that deposit at the contact regions between the alumina particles where they densify upon heating to strengthen the alumina particle matrix while substantially prohibiting net shrinkage. This composite mold material is stable to 1000°C and has the additional quality that it may be etched fairly rapidly in hydrofluoric acid thereby alleviating the need for an additional sacrificial material.

To additionally avoid REPM powder processing these high temperature glass-ceramic molds were found to be suitable for hot-forging bulk REPM material. In this case a previously formed anisotropic unmagnetized bulk magnet is placed over the alumina mold in the desired orientation with respect to the mold geometry. Creep rate measurements for a variety of REPM materials revealed reasonable rates at 650°C to 750°C under pressures of 100MPa. The procedure is then to place a disc of material with thickness near a few millimeters on the glass-ceramic mold in a high vacuum system and hot press for a time of the order of one hour. The excess magnetic material is removed by lapping to expose the glass-ceramic mold which is then susceptible to dissolution with hydrofluoric acid.

EXPERIMENTAL AND RESULTS

Bonded REPM

For isotropic REPM material used in bonded magnets MEP scales as the square of the material density and therefore compaction behavior is a major concern. Compaction curves for a number of powders with and without epoxy were measured by in-situ monitoring of the sample density as a function of applied pressure. Results from these studies indicate that powders with 15μm particles press to about 7% higher density at a given pressure than those with micron sized particles, that the presence of 10%/vol of a low viscosity epoxy has little effect on packing density, and that densities above 80% of theoretical may be achieved only at high pressure (60ksi) for coarser powders, whereas only 70% may be achieved at lower pressure (20ksi). The high pressures necessitate the transition of a mold material from PMMA to metals such as copper.

To explore geometric variation influence on magnet properties, a test mask detailed in Fig. 1 was prepared consisting of rectangular, cylindrical, ring, Halbach array, and multipole rotor geometry. This layout also tests the ability to batch magnetize various permanent magnet shapes fabricated in multitude over a large substrate area. Features with low demagnetization factor, long bars, are included to allow reasonably accurate open magnetic measurements with a SQUID magnetometer to identify maximum energy product variation with binder content. The results are indicated in Fig. 2 for an isotropic Nd₂Fe₁₄B powder. After examining the results from many runs a maximum MEP of near 10 MGOe was found to be the limit for the isotropic powder.

Although claims of achieving up to 19 MGOe with bonded REPMs using anisotropic $\text{Nd}_2\text{Fe}_{14}\text{B}$ powder have been reported, attempts to achieve this by molding in a magnetic field were not successful. Bonded REPM results from the test mask in Fig.1 are depicted in Fig.3. A typical magnetization curve is shown in Fig. 4 for these bonded magnets.

Fig. 1 REPM test mask pattern with total area of 2.4 x 4.6 cm. Features as small as $10\mu\text{m}$ are present as well as geometry for multipole structures.

Fig. 2 Bonded REPM maximum energy product as a function of binder volume concentration for isotropic $\text{Nd}_2\text{Fe}_{14}\text{B}$ powder.

Fig. 3 Bonded $\text{Nd}_2\text{Fe}_{14}\text{B}$ micromagnets fabricated using DXRL molding at 200 micron thickness. a) and b) show quadrant shaped geometry which are intended for assembly into a 2 pole-pair permanent magnet rotor. The magnets are magnetized in the direction indicated by the arrow in a). Distortion is present as an artifact of the magnetic field interaction with the SEM imaging and is evident by comparing a) and b) which were taken with different accelerating potentials. c) shows magnet shapes for a 4 pole-pair permanent magnet rotor along with trapezoidal shapes to be used in a magic ring as described in Fig. 8. d) indicates that bar shaped magnets may be processed. Permanent magnet features as small as 5 microns have been replicated at 200 micron structure height.

Fig. 4 Full hysteresis loop of bonded isotropic $\text{Nd}_2\text{Fe}_{14}\text{B}$ powder in 20%/vol epoxy with a density of 6.3 g/cm^3 . The remanent magnetization is 0.62 T and the coercivity is 1.35 T.

High Temperature Processing

To achieve higher MEP magnets than achievable with bonded REPMs, higher density anisotropic REPM material is required. This leads immediately, however, to higher temperature processing. The best results were obtained by using an intermediate alumina mold form replicated from a DXRL defined PMMA mold and then hot forging pre-aligned fully dense REPM material.

Precision alumina micro-mold results are shown in Fig. 5. These forms were achieved by repetitive slurry casting layers of a alumina/silica mixture into DXRL defined PMMA mold forms with intermediate slurry drying steps at 100°C . After removing the excess alumina/silica by lapping, the PMMA is burned out and the ceramic composite is heated to 950°C in air for 1 hour. In a similar manner, NiZn ferrite geometry was also achieved as shown in Fig. 6 and was found to have favorable high frequency ferromagnetic response to over 1MHz. Further details are described in appendices D, F, and G.

(a)

(b)

(c)

Fig. 5 (a) and (b) Alumina mold forms created from DXRL defined PMMA. (c) alumina/silica composite mold with 80 μ m wide apertures 250 μ m deep. Apertures as small as 10 μ m were realized.

Fig. 6 NiZn ferrite structures fabricated directly from DXRL defined PMMA molds.

Experiments were conducted to identify strain rates of die-upset aligned $\text{Nd}_2\text{Fe}_{14}\text{B}$ material (Magnequench MQ3-f). The results shown in Fig. 7 for a 650°C test reveal substantial strain rates that were thought sufficient to allow deformation into micro-patterned molds. This was determined to be the case as the optical micrographs of hot-forged $\text{Nd}_2\text{Fe}_{14}\text{B}$ in Fig. 8 reveal. The process conditions include a 10°C/min. ramp rate, 1 hour hold at the maximum temperature, high vacuum ambient with a maximum press pressure of 10ksi ramped linearly over the first 20 min. of the maximum temperature plateau, and maximum temperatures of 650, 700, and 750°C. The best MEP result is shown in Fig. 9.

Fig. 7 Strain rate (a) and resulting strain (b) measured for a sample of Magnequench MQ3-F die-upset $\text{Nd}_2\text{Fe}_{14}\text{B}$ material at a temperature of 650°C and 25 MPa (4ksi) applied pressure.

Fig. 8 Optical micrograph of hot-forged die-upset $\text{Nd}_2\text{Fe}_{14}\text{B}$ bars 200μm deep extracted from an intermediate alumina/silica mold.

(a)

(b)

Fig. 9 M-H loop (a) and MEP curve (b) extracted from the second quadrant of the M-H loop in (a) for a hot-forged $Nd_2Fe_{14}B$ permanent micro-magnet as shown in Fig. 8. Fig (a) reveals an intrinsic coercivity of 12,500 Oe with a remanent magnetization of 1.2 T while plot (b) reveals an MEP of 23.1 MGOe.

APPLICATIONS (Brushless DC Micromotor)

To demonstrate the utility of batch fabricated precision miniature permanent magnets a multipole rotor for a brushless DC machine was constructed using DXRL defined REPMs. Due to the precision that is possible at small dimensions with this REPM forming process, a radially anisotropic PM rotor configuration is possible with sub-millimeter dimensions. This type of permanent magnet arrangement leads to additional multipole structures such as Halbach arrays or “magic rings” and sub-millimeter scale ion optic arrangements. Their use in large throw micro to milli scale actuation is also evident.

A low profile rotary motor design was implemented similar to the aspect ratios found in portable magnetic hard disk drive motors. The configuration uses a 9-slot stator 4-pole inner rotor geometry with a slotless stator to minimize cogging torque. Fabrication makes use of precision DXRL formed parts as well as precision gauge pins. A PMMA base plate fabricated with DXRL provides a “clock-plate” function by accommodating the press fit of alignment gauge pins and a miniature ball bearing (RMB Miniature Bearings, Inc.) as shown in Fig. 10. A 320 μ m diameter shaft press-fit into the

Fig. 10 (a) cross-section and plan views of miniature planar brushless DC motor. (b) coil winding form fabricated from PMMA and used to wind multiple coil windings in an automatic coil winder. (c) coil form fabricated from PMMA and used to support 9-pole slotless stator.

ball bearing supports the rotor about a 1.6 mm diameter 80 Permalloy collar. Stator coils of 100 turns of 47 AWG wire are wound on a PMMA pre-form, potted and then freed by dissolving the PMMA. A second circular PMMA coil form shaped in a 9-slot configuration is used for a support to insert the 9 coils after which the entire coil arrangement is press-fit inside an outer magnetic return path ring held by 4 press fit pins into the PMMA clock plate. A small PC board with conductor patterns that allow for either a "Y" or "Δ" connected 3-phase motor is used to bond out wires from the stator. The assembly is depicted in Fig. 11 with a 4-pole REPM rotor. An 8-pole rotor may also be accommodated in this design.

Testing of this type of millimotor shows the benefits of incorporating permanent magnets in sub-miniature magnetic devices: excitations as small as $400\mu\text{A}$ are found to be sufficient to induce rotor rotation which results in a total power dissipation of a few micro-Watts. Driven at 200 mW this motor generates approximately $40\mu\text{N}\cdot\text{m}$ of maximum torque with ultimate speeds of 25,000 rpm. Expected efficiencies are near 25%. A suitable dynamometer, however, is needed to conduct the appropriate tests.

Fig. 12 9-slot, 4-pole slotless, brushless low profile DC motor with 5 mm diameter permanent magnet rotor fabricated with DXRL defined bonded REPM.

LIST OF PUBLICATIONS

- (1) T.R. Christenson, "Advances in LIGA-based post-mold fabrication," Invited Paper in Proceedings of SPIE Conference on Micromachining and Microfabrication Process Technology IV, Santa Clara, CA, Sept. 1998, pp. 192-203.
- (2) T.R. Christenson, T.J. Garino, E.L. Venturini, "Deep X-Ray Lithography Based Fabrication of Rare-Earth Based Permanent Magnets and their Applications to Microactuators," Invited Paper in Electrochemical Society Proceedings, Vol. 98-20, pp.312-323.
- (3) T.J. Garino, T.R. Christenson, E.L. Venturini, "Fabrication of MEMS Devices by Powder-Filling into DXRL-Formed Molds," Mat. Res. Soc. Symp. Proc. Vol. 546, 1999, pp. 195-200.
- (4) T.J. Garino, T.R. Christenson, E.L. Venturini, "The Development of a Ceramic Mold for Hot Forging Micromagnets," Proc. of Ceramics Society, Indianapolis, IN, April 26-28, 1999.
- (5) T.R. Christenson, T.J. Garino, E.L. Venturini, D.M. Berry, "Application of Deep X-Ray Lithography Fabricated Rare-Earth Permanent Magnets to Multipole Magnetic Microactuators," Proc. Transducers '99, Sendai, Japan, June 7-10, 1999, pp. 98-101.
- (6) T.R. Christenson, T.J. Garino, E.L. Venturini, "Microfabrication of Fully-Dense Rare Earth Permanent Magnets via Deep X-Ray Lithography and Hot Forging, Book of Abstracts, HARMST'99, Kisarazu, Japan, June 13-15, 1999.
- (7) T.R. Christenson, T.J. Garino, E.L. Venturini, "Batch Fabrication of Precision Miniature Permanent Magnets, U.S. Patent Application, Filed 11/29/99.

SUMMARY AND IMPACT

A process to fabricate rare-earth based permanent magnets with micro-scale dimensions and sub-micron tolerance has been realized with a technique based on deep x-ray lithography. Maximum energy products between 6 and 10 MGOe are achievable in bonded form and as great as 23 MGOe using a hot forging scheme with an intermediate glass-ceramic composite mold. Integration of these magnets into MEMS requires individual assembly and this has been demonstrated with sub-millimeter geometry. Approaches involving wafer-scale assembly have been conceived and are appropriate but have not been carried out and clearly define area for future work.

The benefit of permanent magnet integration to microactuators has been demonstrated and it has been further realized that the ability to precisely define REPMs on the microscale enables miniaturizing multi-pole REPM structures with exceptional qualities. The possibilities include true radial anisotropy for multi-pole rotors and repeated isolated magnetization definition in regions smaller than 10 microns. Highly uniform and amplified fields generated with Halbach array forms may be realized on the microscale. An additional effect concerns mechanical scaling issues in microdevices. Realizing compliant springs, for example, becomes more of a challenge due to mechanical stiffness increases with direct scaling. This result places an even greater burden on lithography as even finer features become desired. Suitable arrangement of permanent magnet structures will perform the equivalent of a mechanical spring function without scaling difficulty and therefore can potentially offset mechanical scaling behavior. In addition batch fabrication of micro-scaled power generation devices requiring electromagnetic generators may be optimally supported.

A summary of other LDRD work is summarized in the following:

Number of Technical Advance Disclosures:	2
Number of U.S. Patent Applications:	1
Number of Proceedings Publications:	6 (2 Invited)
Number of Students Supported:	3

Advances in LIGA-based post-mold fabrication

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ABSTRACT

The establishment of a process to allow planarization of deep x-ray lithography based microfabricated metal components via diamond lapping has enabled examination of three additional microfabrication issues. The areas of improvement that are discussed include materials, microassembly and packaging, and multilevel fabrication. New materials work has centered on magnetic materials including precision micromagnets and surface treatments of electrodeposited materials. Assembly and packaging has been aided by deep silicon etch processing and the use of conventional precision milling equipment combined with press-fit assembly. Diffusion bonding is shown to be a particularly important approach to achieving multilevel metal mechanisms and furthermore shows promise for achieving batch assembled and packaged high aspect-ratio metal micromechanics.

Keywords: LIGA, MEMS, magnetic materials, permanent magnets, assembly, packaging, diffusion bonding

1. INTRODUCTION

LIGA-based processing or deep x-ray lithography based mold fabrication and subsequent additive processing has demonstrated the ability to extend precision machining limits to the millimeter part regime while maintaining tolerances of 1 part in 10^4 . The unique processing feature that enables this result is the high precision mold obtained by exposing a photoresist material with highly collimated and long absorption length x-rays combined with accurate masking, development and subsequent mold filling. The prismatic geometry that results is further defined by precision lapping to control thickness¹.

Growing interest and use of deep x-ray lithography² (DXRL) has led to improved storage-ring access to the point where it may be acquired as a foundry-like service. Exploitation of high-energy x-ray flux has further shown sufficient cost efficiency to support direct deep x-ray lithography and mold filling processing without mold replication³. Additionally, improved stability of the x-ray photoresist, namely PMMA, has resolved many issues with respect to maximum mold aspect ratio, ultimate structure height, and minimum feature size⁴. The resulting high molecular weight PMMA mold material has proven to have sufficient mechanical integrity to withstand single level processing and is able to be readily dissolved subsequent to mold filling and planarization with organic solvent. The mold filling materials have most commonly been readily electroformable metals such as nickel and alloys of nickel including nickel-iron, nickel-cobalt, and nickel phosphorous, and copper, silver, and gold. This list of electroplatable metals which is expanding and also includes electroplated composites⁵ is sufficient for many applications. More material choices are still needed in particular for magnetic devices and high temperature devices. Work in powder metallurgy and plastic injection molding has given an indication of what additional materials are possible^{6,7}.

To further advance deep x-ray lithography based additive processing, three areas of processing are receiving attention: the areas include material base expansion, assembly and packaging techniques, and multi-layer processing.

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2. MATERIALS

Several means to incorporate new materials into deep x-ray lithography based molding while maintaining the same mold precision have been investigated. A large effort is directed at improved magnetic materials including soft (high permeability) and hard (permanent magnet) materials. Soft magnetic material may be achieved with an electroplated 78/22 nickel/iron or 78 Permalloy alloy with initial permeability of 2000 and a saturation magnetic flux density of 1.0 Tesla. More recently a higher percentage of iron has been implemented at a 45/55 nickel/iron composition with a sulfamate base electrolyte. The advantage of this composition is its similar high permeability with an increase in saturation magnetic flux density to 1.6 Tesla. The higher saturation induction yields an increase in the magnetic pressure that may be achieved in a magnetic actuator by a factor of 2.5.

The incorporation of a permanent magnet material in MEMS has received comparatively little attention^{8,9}. This is the situation despite the attractive MEMS feature of a permanent magnet which is that its magnetization remains independent of scale. Electromagnetic coils, on the other hand, are constrained during scaling due to current density limits. Superconductors also suffer this limit, which results in a reduction of magnetic field that may be produced from a coil of decreasing dimensions due to a scaling dependence on length.

The best magnets available today in terms of maximum energy product, which is the figure of merit important for actuator performance and volume efficiency, are rare-earth based permanent magnets with available energy products over 40 MGOe (Mega-Gauss-Oersted, 1 MGOe = $100/4\pi$ kJ/cm³). Implementing fully dense rare-earth based permanent magnets requires high sintering temperatures that are not compatible with precision molding due to volume shrink. It is possible, however, to incorporate rare-earth magnet powder with a binder to make bonded or composite magnets. A reduction in magnet material density results in a reduced remanence and energy product that still far exceeds that possible in non rare-earth based magnets. It is this bonded form of rare-earth permanent magnet which has been used for molding into DXRL defined PMMA. The Nd₂Fe₁₄B intermetallic has been used primarily with various powder particle sizes less than 20 micron and an epoxy binder. A preferred process has been the incorporation of the unmagnetized powder/binder matrix in a low viscosity state into a PMMA mold via calendering and pressing. The resulting substrate containing a multitude of permanent magnet geometry is batch magnetized in a preferred orientation with a field of at least 35,000 Oersted. An epoxy composition of 20% by volume has been found to yield a micro-magnet with sufficient mechanical strength to be handled and a maximum energy product of 10 MGOe. The PMMA mold material may be dissolved without affecting the binder material and the magnets may be released by undercutting a suitable sacrificial material. An M-H loop of a magnet 900 μ m x 900 μ m x 120 μ m is shown in Fig. 1. Example micro-magnet geometry is depicted in Fig. 2 along with close-up views of sidewall reproduction.

Fig. 1 M-H loop for bonded micro-formed permanent magnet with Nd₂Fe₁₄B powder and 20% epoxy binder measured with the use of a SQUID magnetometer.

In order to increase the maximum energy product, incorporation of anisotropic $\text{Nd}_2\text{Fe}_{14}\text{B}$ powder is under investigation which has been reportedly used to achieve energy products of 19 MGOe. This material requires processing in a magnetic field to orient the powder particles. In the pursuit of fully dense rare-earth magnets, MnZn and NiZn ferrite materials have successfully been formed using DXRL defined molds followed by their release and sintering. This approach, however, is only appropriate if volume reduction can be tolerated. Hot pressing methods that may alleviate this dimensional shift for prismatic geometry are also being pursued.

a)

b)

c)

d)

Fig. 2 SEM views of bonded and molded micro-magnets with $\text{Nd}_2\text{Fe}_{14}\text{B}$ powder particle size less than 10 microns. The partial arrow-shaped tabs on the structures in a) are patterned to indicate easy identification of magnetization direction after release. The thickness of these particular magnets is 30 microns.

Another means to fill DXRL molds with different materials has been with thermal spray techniques. In this approach, molten metal droplets are sprayed against a substrate where they coalesce and solidify. Substrates covered with DXRL defined PMMA molds have been tested with the use of a subsequent lapping step to remove the overspray. Initial results are shown in Fig. 3 with the material Kirksite (3.5%Cu, 4% Al, 0.04% Mg, bal. Zn). No observable perturbation of the PMMA mold occurred and vertical sidewall replication is apparent. The variation in density as a function of aperture size is also

readily apparent and suggests a classic wetting phenomenon. Additional study is being carried out which if successful would yield a deposition speed far surpassing electroplating rates with the incorporation of an extremely broad material base.

Fig. 3 SEM photographs of thermal spray result with Kirksite in a screen geometry. The PMMA mold material has been removed.

Methods to improve the mechanical properties of electroformed metals are also receiving attention. Since nickel and copper are easily electrodeposited with low stress with reasonably fast rates they are preferred materials from the standpoint of processing. Their mechanical use could be expanded if a suitable batch treatment was available to improve their surface properties. One such approach has been demonstrated with electroformed nickel by using implantation of titanium and carbon to increase its yield strength and hardness¹⁰. The result is an amorphous near surface layer extending 100 nm into the nickel surface. Characterization by finite-element modeled nano-indentation reveals a yield strength of 6 GPa, hardness of 16 GPa, and an elastic modulus of 400 GPa in the amorphous region, all of which far exceed bulk or as-electrodeposited material values. The result is a substantial reduction in wear and a 50% decrease in the friction coefficient¹¹. An additional attribute of this surface is its anti-corrosion character and reduced surface chemical activity that has significant implications for long term static friction or "stiction" concerns.

Because implantation treatments are "line of sight" processes, the ability to treat all geometry including narrow apertures may present accurate implementation issues. A conformal technique removes this difficulty and has been demonstrated with PECVD (Plasma Enhanced Chemical Vapor Deposition) silicon carbide films deposited directly on a substrate decorated with LIGA nickel structures. A silicon carbide film 900 angstroms thick on electroplated nickel has demonstrated hardnesses of nearly that of the implant treatments and has an added potential benefit of forming an insulating layer.

3. ASSEMBLY

To date, the majority of MEMS implementations of LIGA based fabrication have been accomplished by assembly of individual LIGA parts. The assembly substrate is commonly decorated with LIGA fabricated assembly geometry such as locating and assembly pegs or shafts, guide posts and other fixed geometry. This substrate is then used to assemble components via pick and place assembly which are held captive by retaining clips and springs. This approach is particularly sensitive to two processing concerns of adhesion and internal strain. The example of an assembled gear on a shaft to implement a journal bearing reveals the problems. Electroplating a shaft on a substrate typically involves at least two interfaces: one interface exists between the plating base and the substrate and the other exists between the plating base and the electrodeposited material. At each of these interfaces, a concern for adhesion exists. The necessary minimum adhesion is complicated by several factors. Lapping and assembly operations create shear stresses acting on the top surface of the electrodeposited structures. For high aspect-ratio geometry, this stress is magnified by a resulting lever arm thereby creating

large tensile stresses at the substrate interfaces. In addition, any residual strain in the electrodeposit adds to the burden on the interfaces.

Two approaches are being pursued to eliminate the interface adhesion problem. One approach involves the use of press-fit pins which are sunk into either a LIGA fabricated peg-board or a pre-drilled substrate. This transfers the adhesion problem into a material strength problem. The trade-off is an increased reliance on assembly. The assembly in Fig. 4 demonstrates this capability. The aluminum substrate was drilled with locating holes using a precision mill capable of 0.000020" positional accuracy. Pins were made from tool steel gauge pin stock that in this case were 0.0059" in diameter and cut to 0.033" lengths which after being press fit into the 0.020" thick substrate provided a 330 μ m high pin. The gear train was assembled by first fitting a collar with a key on each shaft and then two gear thicknesses were used to assemble a gear train with a 450:1 transmission ratio coupled into a rack (see Fig. 4.a). The large pin located in the front of the gear train pictured in Fig. 4.c) is

a)

b)

c)

Fig. 4 SEM photographs of a two-level gear train assembled on a press-fit pin array. The substrate material is aluminum, the pins are tool steel, and the gears are LIGA fabricated from nickel.

used to align an output gear from a miniature motor to couple into and drive the mechanism. A flat plate that covers all assembly pins is placed over the mechanism to keep it captive.

Another means that has been pursued to eliminate interface adhesion limitations and expand packaging and system assembly possibilities is to create an assembly substrate monolithically. By taking advantage of precision high aspect-ratio silicon etching multi-level silicon alignment structures may be fabricated by etching into a standard silicon substrate that may be used to assemble LIGA fabricated components. The results are shown in Fig. 5 for the same gear train mechanism

a)

b)

Fig. 5 Two-level nickel gear train assembled into high aspect-ratio silicon etched substrate. The silicon pins are 150µm in diameter and 330µm tall.

described previously. A glass plate was bonded on top of the etched silicon die to provide for packaging during testing. The mechanism was run for several days indicating that silicon is a reasonable material to be used with metal LIGA components.

This is the first step in demonstrating the use of silicon as a packaging material for LIGA components which allows the pursuit of further system integration. Integrated silicon microelectronics may be resident on the same assembly substrate and connected to the electromechanical assembly with electrical vias. Also, a complementary etched silicon substrate may be used to bond with the assembly substrate to serve as a cover and enable batch packaging.

Furthermore, the array of silicon assembly die provides a basis for batch assembly of LIGA components via wafer-to-wafer transfer and batch release. In this assembly sequence a silicon wafer is deep RIE etched to form assembly geometry and alignment pegs. A complementary substrate with suitable sacrificial layer is fabricated with first level LIGA components, aligned to the silicon assembly substrate with which it engages, and finally a sacrificial etching step releases the components onto the assembly substrate. This batch assembly process has been successfully tested for one level and is expected to provide a technique suitable for multi-level LIGA batch assembly.

4. MULTILEVEL FABRICATION (DIFFUSION BONDING)

The ability to construct 3-dimensional micromechanisms with planar-based processing continues to be a challenge with ever increasing complexity. In the area of high aspect-ratio processing considerable advancement has occurred via deep silicon etching, novel polysilicon processing, and LIGA-like fabrication. Process integration issues for high aspect-ratio structures, however, lead to many complications for extending this type of processing to multiple levels in batch form. One example of the problem is evident with a simple journal bearing several hundred microns in length. In order to maintain a reasonable bearing tolerance ($<1\mu\text{m}$) subtractive fabrication by assembly of two closely dimensioned parts has proven to be a suitable albeit cumbersome technique. The relatively recent capability of accurate thickness control of electroformed structures in LIGA processing with precision diamond lapping and polishing has brought a significant improvement and expansion to LIGA-based device implementations. Additional obstacles remain in establishing a multi-level process with batch assembly and packaging.

Previous work to incorporate multilayer LIGA structures has used successive electroplating upon electroplating sequences¹². The main concern in this approach is photoresist stability that in the case of the DXRL photoresist of choice, PMMA, is prone to crazing and adhesion loss when subjected to multiple liquid exposures and temperature cycles. Plating base complications also constrain geometry and lead to consideration of electroless deposition⁷ which has further complications due to deposit stress and limited thickness.

The multilevel technique to be discussed takes advantage of the unique qualities of DXRL-based fabrication including the attributes of additive processing to develop a new concept in metal microfabrication of MEMS. Use is made of diffusion bonding (or diffusion welding, or solid-state bonding) to develop a process for the batch fabrication of multilevel LIGA components. The ability to planarize LIGA structures is a critical enabler of such a process since it allows two substrates decorated with LIGA components to be placed face-to-face in close contact across an entire substrate. Diffusion bonding possesses many attributes ideally desired for a multilevel precision micromachining process. Bonding takes place by definition without any material melting thereby retaining the precision of the microcomponent definition. The time-temperature-pressure trade-off may be used to advantage by conceding time for temperature and pressure. This is reasonable for batch part microfabrication where several hour bond times are allowable and additionally many wafers may be processed simultaneously as opposed to more conventional single part diffusion bonding where bond times are excessive if greater than tens of minutes. In addition, it is possible to obtain a diffusion bonded component using only a single material.

Initial tests for diffusion bonding electroformed nickel to itself have shown successful bonding at temperatures surprisingly as low as 450 degrees C at a pressure of 7 ksi (~50 MPa). This is well less than half (590 deg. C) the absolute melting temperature of nickel. A possible reason for this low temperature result may include the very fine grain size associated with as-electroplated nickel. Electroplated metals in general have this fine-grain character. During the diffusion bonding temperature cycle grain growth encourages coalescence of grains at the bond interface. Also, careful surface preparation included chemical removal of the nickel oxide and highly flat, parallel and polished components resulting from diamond lapping. Bond strength testing was performed with a Sebastian adhesion tester and a sample geometry outline in

Fig. 6. A nail head pull test peg was bonded to the individual diffusion bonded discs which could be gripped and pulled in the adhesion tester. Temperatures between 450 deg. C and 600 deg. C and pressures between 7 ksi and 10 ksi were used with bond times of 6 hours. All bonds exceeded the capacity of the pull tester which for the smallest geometry resulted in a maximum load of ~ 15 ksi. The indication is that the temperature and pressure may be further reduced. Photographs revealing the diffusion bond interface metallography are depicted in Fig. 7. The bond line is difficult to see until a grain boundary etch is carried out.

Fig. 6 Preparation of diffusion bond test specimens.

a)

b)

Fig. 7 SEM photographs of nickel-nickel diffusion bond interface. Fig. a) shows the as polished bond line indicated by the arrows and Fig. b) shows the interface after a grain boundary etch.

One result is shown in Fig. 8 for a nickel multiple layer stacked component that was diffusion bonded in one step at 500 deg. C and 10 ksi for 6 hours. No observable deformation occurred near interface edges which is a sign of insignificant creep. The proposed batch diffusion bonding sequence is shown in Fig. 9 which incorporates a suitable sacrificial layer that is expected to be copper and a precision alignment technique utilizing similar gauge pins to those used in the assembly shown in Fig. 4.

Fig. 8 SEM photographs of 7 layer diffusion bonded all-nickel component. Alignment of the stack was achieved with two copper wires press fit through all seven layers. This particular component may be diffusion bonded in one step since all interfaces have the same overlap area. Such a situation will not be true in general for a batch wafer scale bonding process.

Fig. 9 Batch diffusion bonding procedure.

In order to more accurately assess the diffusion bond strength a method to allow measurement of local shear strength across a substrate has been devised using a milli-scale torsion tester. The specimen geometry is depicted in Fig. 10 and includes a scalloped ring geometry which accepts coupling to a circular array of six pins that extend from the end of a torsion arm. The pins are located with a LIGA fabricated part fit into the pin housing part of the instrument depicted in Fig. 11.

Fig. 10 SEM photograph of distributed array of 3mm diameter torsion shear test rings. The measurement bar indicates 5 mm and there is some distortion occurring due to the low magnification SEM image.

The milli-torsion testing has just been initiated and it is evident that the ability to test other types of LIGA adhesion concerns in this fashion is also accommodated.

Further extensions of microfabrication by diffusion bonding include the use of a patterned sacrificial layer for eventual partial part release and fully integrated metal micromechanisms. Since diffusion bonding takes place in a high vacuum, batch vacuum packaging and packaging in an inert ambient are also possible extensions.

a)

b)

Fig. 11 Photographs of torsion shear test instrument showing x-y-z substrate mounting stage, lever-arm mounted to 6-pin fixture and in background: motorized micrometer, load cell and LVDT. Fig. b) shows a close-up of partial pin engagement with the scalloped ring specimen.

5. CONCLUSIONS

Obvious opportunities exist for expanding the material base in deep x-ray lithography based micromolding with the pay-off being more efficient microactuators and expanded design flexibility. Approaches to micro assembly of high aspect-ratio metal mechanisms benefit from conventional and silicon machining technology. The application of diffusion bonding to high aspect-ratio metal microfabrication appears to be a significant tool for addressing multilevel, batch assembly, and packaging needs.

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Batch Fabrication of Precision Miniature Permanent
Magnets

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Batch Fabrication of Miniature Permanent Magnets

This invention was made with Government support under Contract DE-AC04-94DP85000 awarded by the U.S. Department of Energy. The Government has certain rights in the invention.

Background

The present invention relates generally to permanent magnets, and more specifically to a new process for making miniature (typically 0.1 to 10 millimeter) rare earth permanent magnets with dimensional tolerances as small as one micron.

SUMMARY OF THE INVENTION

The present invention relates to new processes for fabrication of precision miniature rare earth permanent magnets, and to the magnets so fabricated. Typical magnet dimensions for the processes disclosed range from about 0.1 to 10 millimeters, while dimensional tolerances as small as one micron can be attained.

Brief Description of the Figures

Figure 1 schematically illustrates an implementation of the class of processes comprising the instant invention.

Figure 2 schematically illustrates an implementation of the process of making polymer molds using precision high aspect ratio lithography.

Figure 3 schematically shows the process of making metal molds.

Figure 4 schematically shows the process of making ceramic molds.

Figure 5 schematically shows various techniques which can be used to fill a mold with a flowable molding substance.

Figure 5a shows a calendering process, figure 5b shows a doctor blading process, figure 5c shows a pressing process, figure 5d shows an injection molding process, and figure 5e shows a dipping process.

Definitions

Principal plane - a plane parallel to that of the substrate on which a component is fabricated.

Miniature component - a component which has dimensions in the principal plane roughly in the range 0.1 to 10 millimeters, and dimensions parallel to the principal plane roughly in the range 10 to 1000 microns.

Precision component - a component which has dimensional tolerances roughly in the range 1 to 50 microns in the principal plane, and in the range 0.1 to 10 microns parallel to the principal plane.

2.5 dimensional object - an object which may have a complex outline in the principal plane, but is of substantially constant thickness perpendicular to the principal plane. An object which is stepped to show different outlines on several planes, all of which are parallel to the principal plane, is also a 2.5 dimensional object.

Detailed Description

The present invention is of a process for fabricating precision miniature magnetic components comprising rare earth magnetic materials, and the components made thereby.

Such magnets can exhibit very large magnetic energy products and micron-scale dimensional tolerances. Various implementations of the basic process are described below. However, the details of the specific implementations chosen are not intended to limit the scope of the invention.

A schematic illustration of the present process is shown in Figure 1. Figure 1a shows a mold 1, comprising a structured polymer layer 12 attached by a sacrificial layer 11 to a substrate 10. A layer 13 of flowable magnetic material comprising rare-earth magnetic particles is applied to the opposite surface of structured polymer layer 12, and the whole is positioned within a pressing plate 14. The structured polymer layer 12 is often made of polymethacrylate (PMMA), and can be structured using various lithographic techniques.

At this point, the pressing plate 14 is lowered, forcing the flowable magnetic material 13 into mold 1. In some implementations, the flowable magnetic material 13 is now caused to solidify, for example by curing an epoxy constituent thereof. After the material is solidified, the voids of mold 1 are accurately filled with the solidified magnetic material, but an uncontrolled amount of excess solidified magnetic material remains on top of mold 1. This leads to the step shown in Figure 1b, where the excess material is lapped off, leading to magnetic components 15, still positioned within mold 1, with precision mechanical tolerances.

It is now necessary to magnetize, or at least align, the rare earth particles so that the magnetic components 15 have a macroscopic magnetic moment, and thereby generate a large macroscopic magnetic field. (Note that the field direction can vary throughout a given component if the magnetization procedure is appropriately chosen.) This step is shown in Figure 1c, where a large magnetic field 16 is applied to the magnetic components 15 by an external magnet 17. External magnet 17 may function in a pulsed mode to allow sufficiently large magnetic fields to be attained.

If the magnetic components 15 are not intended to be used in place, they are then released from mold 1 (Figure 1d) by dissolving the sacrificial layer 11 and the structured polymer layer 12. The resulting components can exhibit magnetic energy densities as large as 20 megagauss-Oersteds, roughly half that of a fully dense rare earth magnet.

There is a wide class of processes along the lines of that outlined briefly above that are within the scope of the present invention. At nearly every step mentioned above, there can be a fork leading to one or several alternate process routes. Many of these alternate paths will be described below.

The fragility and brittleness of rare earth magnetic materials makes conventional fabrication of precision miniature rare earth magnets impractical. The present invention avoids such limitations, partially by new

precision mold fabrication techniques, and partially by new combinations of process steps.

If precision miniature magnetic components are to be fabricated as shown in Figure 1, the first challenge is to create suitable molds. This is carried out lithographically in the present invention, conventional machining techniques being either inadequate or too expensive for fabrication to the dimensional tolerances required.

A variety of lithographic techniques can be applied to the problem of defining the molds. A class of examples are provided by high aspect ratio lithographic techniques, one example of which is the basis for the LIGA technique. High aspect ratio lithography is illustrated in Figure 2. Figure 2a shows a substrate 20 with a layer of a polymeric lithography resist 21. In the techniques to be described this layer can be as thick as several millimeters, compared to the ~10 micron layers more often used in integrated circuit fabrication. A mask 22 is placed on or above the resist layer 21. Mask 22 is made of a high atomic weight material (often gold), so that it can effectively stop radiation.

At this point, the masked resist layer is exposed to a suitable source of energetic radiation. In the LIGA process this radiation is usually soft x-rays generated by a synchrotron, but other sources and wavelengths of photons can be used, as can certain types of particle beams.

Following the exposure, the exposed resist is dissolved away (if the resist is positive - otherwise the unexposed resist is dissolved away), and the mask 22 is removed. The result, as shown in Figure 2b, is a structured polymer layer 23 resting on a substrate 20. Traditionally PMMA resists are used, but a wide variety of polymeric resists are compatible with this technique.

The precision of the technique depends on being able to accurately replicate the open areas of the mask 22 with exposed resist throughout the thickness of the resist layer. This requires that the radiation source produces a substantially parallel beam, and that neither diffraction from the mask nor diffusion within the resist layer significantly disturbs that beam. The result is tall features with nearly vertical sidewalls, quite suitable for molding 2.5 dimensional objects.

The process outlined in figure 2 produces precision miniature structured polymer molds which can exhibit high aspect ratios. However, a polymer-based mold is not suited to fabrication of certain classes of magnetic components. It is therefore useful to provide for a variety of types of molds.

Figure 3 outlines the formation of precision miniature metal molds. In figure 3a one begins with a negative mold 3, comprising a substrate 30 covered by a structured polymer layer 31. The top surfaces of the negative mold 3 are then coated with a release layer 32, which can be dissolved or

otherwise removed without damaging the final metal mold. If it is possible to so dissolve the negative mold 3, forming the release layer 32 can be left out of the process.

Figure 3b shows the structure attained after a thick layer 33 of metal has been deposited over the release layer 32. There are several possible routes toward depositing metal layer 33. These include electrodeposition onto substrate 30 (requiring that substrate 30 be conducting and that no release layer 32 be deposited), electrodeposition onto release layer 32 (requiring that sacrificial layer 32 be conducting). The above process steps can also be carried out using electroless deposition instead of electrodeposition. Deposition of metal layer 33, or deposition of a conducting release layer 32, or both, can also be carried out using physical deposition of various types, including thermal evaporation and sputtering.

In figure 3c the negative mold 3 and the release layer 32 have been removed, leaving the final metal mold 33 ready for use. Practical considerations (strength, durability, etc.) can make it appropriate to perform additional tasks, such as lapping flat the back side of metal layer 33, or binding the metal layer to a supporting block (not shown), prior to removing negative mold 3 and release layer 32.

There are also circumstances under which high temperatures will be combined with high pressures. Under such conditions even a metal mold may not prove suited to the task of fabricating precision miniature magnetic

components. Ceramic-based molds can often be used to address such problems.

Figure 4 outlines how a ceramic-based mold can be formed. Such a mold is built upon a negative mold 4 comprising a structured layer 41 on top of a substrate 40. A ceramic precursor layer 42 is applied on top of negative mold 4 so that it thoroughly fills in the voids thereof (figure 4a). The precursor layer can consist of a ceramic slurry, a pressed ceramic powder (with or without an added binding element), or other formulations known in the art. If the negative mold is not easily removable (for example, during a later firing step), a release layer (not shown) can be introduced between the negative mold and the ceramic precursor layer.

The next step in the process of fabricating a ceramic-based mold involves sintering the ceramic precursor layer. This sintering step is generally carried out under conditions which avoid densification of the ceramic, so that the requisite level of dimensional precision is copied from the negative mold. The sintering can be carried out while the ceramic precursor layer is still in contact with the negative mold, or a preliminary step of stabilizing the ceramic precursor can be carried out, followed by removing the negative mold, then followed by sintering the precursor.

It is also possible, if the negative mold will survive the temperatures and pressures required, to use uniaxial hot pressing to sinter the precursor layer in situ. When this

is done, the ceramic can be allowed to fully densify, as the uniaxial pressure will insure that the sintered ceramic fully and accurately fills the voids in the negative mold.

The negative mold can be removed by using a release layer between the ceramic precursor layer and the negative mold, by burning out an organic negative mold, or by other methods known in the art. The result is the ceramic mold 43, shown here with an optional metallic coating 44. Metallic coating 44 can be used as a release layer, or to provide a smoother surface for the subsequent molding operations.

A suitable mold must be filled accurately with a molding substance to produce precision miniature magnetic components. Such a molding compound will comprise rare earth magnetic particles. These particles can individually be magnetized prior to filling the mold, although this is not required to practice the present invention.

A molding compound can consist essentially of such magnetic particles, but can also comprise a dispersion of such particles in a carrier material, or in a binding material, or both. (A carrier material is one which aids the flowability of the magnetic particles, whereas a binding materials is one which enhances the tendency of the magnetic particles to remain in place, either during or after fabrication.) Examples of carrier materials would include entraining gases and fluids.

Several classes of binder materials exist. A binding material can be one that is solid at the intended operational temperatures, but is molten at the time when the mold is being filled. Cooling the mold then solidifies the binding material, fixing the magnetic particles in place.

A binding material can comprise a polymer or other fixing agent dissolved in a solvent. During the process of filling the mold, the magnetic particles are suspended in this solution. Afterwards, the flowable molding substance solidifies when the excess solvent is removed from said substance.

A binding material can comprise a low-order polymer or monomer which is liquid during the step of filling the mold, but in which further polymerization is later initiated, thereby solidifying the substance. Initiation of polymerization can take place through thermal, chemical, or radiation means. In a specific example, the binding material can consist essentially of an uncured epoxy resin.

A wide variety of techniques for filling a mold with a flowable molding substance exist, some of which are illustrated schematically in figure 5. In all cases the arrow indicates the direction of motion of the mold.

Figure 5a shows a calendaring process, in which the flowable material 53 is forced into the voids 51 of the mold 50 through the action of pressing roller 52. Figure 5b shows a doctor blading process, in which flowable material

53 is forced into the voids 51 of the mold 50 through the action of doctor blade 54.

Figure 5c shows a pressing process, in which the flowable material 53 is forced into the voids 51 of the mold 50 through the action of pressing plate 55, which moves toward the upper surface of the mold. The mold and/or the plate can be heated. Such hot pressing often results in stronger final components.

Figure 5d shows an injection molding process, in which the flowable material 53 is forced into the voids 51 of the mold 50 through the action of injection of flowable material through injection port 58. The injected material is contained by the combined action of mold 50 and elements 56 and 57. Means to allow trapped gases to escape are usually included in the mold design in a variety of ways known in the art (not shown).

Figure 5e shows a dipping process, wherein the flowable material 53 is forced into the voids 51 of the mold 50 by surface tension and capillary action as the mold is withdrawn from a bath 59 of said flowable material. This technique is usually reserved for thin layers of low-viscosity flowable materials.

Once the mold has been filled and solidified (as described above), excellent dimensional tolerances perpendicular to the principal plane can be obtained by lapping the top surface of the mold. Other means of surface

finishing can also be used, such as milling, sanding, planing, and the like.

There are several ways of inducing a macroscopic magnetization in a magnetic component. If the individual magnetic particles are magnetized prior to filling the mold, they can be physically rotated toward a common magnetic orientation by applying an external magnetic field to the mold prior to solidification of the molding substance. It is also possible to reorient the magnetization vector of the individual particles after solidification using an external magnetic field. The magnetic particles can also be given their magnetization using an external magnetic field, even if the magnetic particles are not previously magnetized. It is also possible to provide different portions of a given magnetic component according to this invention with different magnetic orientations through the application of strong local fields.

The examples and implementations described above are intended to illustrate various aspects of the present invention, not to limit the scope thereof. The scope of the invention is set by the claims interpreted in view of the specification.

Claims

1. A process to fabricate precision magnetic components comprising rare earth magnetic particles, comprising the steps of:
 - a) constructing a mold suitable for fabrication of the desired components;
 - b) filling said mold with a flowable molding substance comprising rare earth magnetic particles;
 - c) inducing a desired magnetization in said molding substance by applying an external magnetic field; and,
 - d) solidifying said molding substance.
2. The process of claim 1, wherein the step of constructing a mold comprises precision high aspect ratio lithography.
3. The process of claim 1, wherein the step of constructing the mold comprises coating a substrate with a polymer layer, and patterning said layer using precision high aspect ratio lithography.
4. The process of claim 3, wherein said polymer layer consists essentially of polymethylmethacrylate.
5. The process of claim 3, wherein the step of constructing the mold further comprises depositing a

metal layer over the substrate and patterned polymer layer.

6. The process of claim 5, wherein said metal layer acts as a sacrificial layer, and said process further comprises releasing the components by removing the metal layer.
7. The process of claim 5, wherein said mold comprises said metal layer, and said process further comprises removing said metal layer from the substrate and patterned polymer layer.
8. The process of claim 7, wherein the substrate is electrically conducting, and said metal layer is electrodeposited on the substrate through openings in the patterned polymer layer.
9. The process of claim 3, wherein the step of constructing the mold further comprises forming a layer of ceramic powder over the substrate and patterned polymer layer.
10. The process of claim 9, wherein the step of constructing the mold further comprises sintering said layer of ceramic powder.
11. The process of claim 10, wherein the step of sintering said layer of ceramic powder is carried out without significant densification.
12. The process of claim 10, wherein the step of constructing the mold further comprises removing said

layer of ceramic powder from the substrate and patterned polymer layer.

13. The process of claim 9, wherein said layer of ceramic powder comprises ceramic powder and a binder.
14. The process of claim 12, further comprising the step of covering said sintered layer of ceramic powder with a metal film.
15. The process of claim 1, wherein said molding substance consists essentially of rare earth magnetic particles.
16. The process of claim 1, wherein said rare earth magnetic particles are magnetized prior to filling said mold with said molding substance.
17. The process of claim 1, wherein said molding substance further comprises a carrier material.
18. The process of claim 1, wherein said molding substance further comprises a binding material.
19. The process of claim 18, wherein said molding substance further comprises a binding material which is liquid when the mold is being filled, and can be solidified after the mold is filled.
20. The process of claim 19, wherein said binding material is melted during the step of filling the mold.
21. The process of claim 19, wherein said binding material comprises a polymer which is liquid during the step of filling the mold.
22. The process of claim 19, wherein said binding material comprises a polymer dissolved in a solvent.

23. The process of claim 19, wherein said binding material comprises an uncured epoxy resin.
24. The process of claim 19, wherein said binding material comprises a monomer which is liquid during the step of filling the mold.
25. The process of claim 1, wherein the step of filling the mold comprises a technique chosen from the group consisting of calendering, doctor blading, pressing, hot pressing, dipping, and injection molding.
26. The process of claim 15, wherein the step of solidifying said molding substance comprises sintering said rare earth magnetic particles.
27. The process of claim 15, wherein the step of solidifying said molding substance comprises hot pressing said rare earth magnetic particles.
28. The process of claim 15, wherein the step of solidifying said molding substance comprises electroplating a metal on said mold, thereby entrapping said rare earth magnetic particles therein.
29. The process of claim 20, wherein the step of solidifying said molding substance comprises freezing said binding material.
30. The process of claim 21, wherein the step of solidifying said molding substance comprises inducing polymerization in said binding material.

31. The process of claim 22, wherein the step of solidifying said molding substance comprises removing said solvent.
32. The process of claim 23, wherein the step of solidifying said molding substance comprises curing said epoxy resin.
33. The process of claim 24, wherein the step of solidifying said molding substance comprises polymerizing said monomer.
34. The process of claim 1, further comprising reducing the thickness of said molding substance to a desired value by lapping the exposed surface of said substance.
35. The process of claim 1, wherein the rare earth magnetic particles are each magnetized before filling the mold.
36. The process of claim 35, wherein the step of inducing a desired magnetization comprises rotating said particles toward a common magnetic orientation prior to solidification of said molding substance through the action of an external magnetic field.
37. The process of claim 35, wherein the step of inducing a desired magnetization comprises reorienting the magnetization vector of said particles toward a common orientation through the action of an external magnetic field.
38. The process of claim 1, wherein the step of inducing a desired magnetization in the molding substance comprises application of an external magnetic field

after the mold has been filled with the flowable substance.

39. The process of claim 1, wherein the step of inducing a desired magnetization in the molding substance comprises application of an external magnetic field after the molding substance has been solidified.
40. The process of claim 1, wherein said desired magnetization is not uniform throughout the precision magnetic component.

Abstract

A new class of processes for fabrication of precision miniature rare earth permanent magnets is disclosed. Such magnets typically have sizes in the range 0.1 to 10 millimeters, and dimensional tolerances as small as one micron. Very large magnetic fields can be produced by such magnets, lending to their potential application in MEMS and related electromechanical applications, and in miniature millimeter-wave vacuum tubes. This abstract contains simplifications, and is supplied only for purposes of searching, not to limit or alter the scope or meaning of any claims herein.

Figure 1

2a)

2b)

Figure 2

Figure 3

Figure 4

Figure 5

DEEP X-RAY LITHOGRAPHY BASED FABRICATION OF RARE-EARTH BASED PERMANENT MAGNETS AND THEIR APPLICATIONS TO MICROACTUATORS

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Abstract

Precision high aspect-ratio micro molds constructed by deep x-ray lithography have been used to batch fabricate accurately shaped bonded rare-earth based permanent magnets with features as small as 5 microns and thicknesses up to 500 microns. Maximum energy products of up to 8 MGOe have been achieved with a 20%/vol. epoxy bonded melt-spun isotropic $\text{Nd}_2\text{Fe}_{14}\text{B}$ powder composite. Using individually processed sub-millimeter permanent sections multipole rotors have been assembled. Despite the fact that these permanent magnet structures are small, their magnetic field producing capability remains the same as at any scale. Combining permanent magnet structures with soft magnetic materials and micro-coils makes possible new and more efficient magnetic microdevices.

INTRODUCTION

Permanent magnets, and in particular rare-earth based permanent magnets, have fundamental properties that make their use on the microscale highly attractive. The most interesting of these properties is that the magnetization of a permanent magnet is independent of scale. Stated another way, the field strength of a permanent magnet does not depend on the size of the magnet. The advantage of a permanent magnet relative to an electromagnet has been pointed out with particular emphasis by Halbach who stated the point in the following way: "... when it is necessary that a magnetically significant dimension of a magnet is very small, a permanent magnet will always produce higher fields than an electromagnet ... with permanent magnets one can reach regions of parameter space that are not accessible with any other technology."(1) This situation occurs due to an inverse linear scaling dependence of an electromagnetic coil brought about by current density constraints due to thermal and electromigration limitations. Coils utilizing superconductors share the same scaling relationship which arises due to a similar critical current density limit.

To quantify at what reduced dimension a permanent magnet begins to possess significantly improved performance compared to a coil is in general not possible, as this is a function of many parameters of a particular application. An estimate for a typical

microactuator application involving a "horseshoe" geometry is presented here. By comparing the flux generating capabilities of an equivalent volume of permanent magnet and coil an expression for the ratio, γ_H , of the two is found to have a dependence as given by equation [1]. In this equation γ_H is the ratio of magnetic field produced by the

$$\gamma_H = \frac{B_r / \mu_m}{2\alpha\mu_0 t_c J_{\max}} \quad [1]$$

permanent magnet to that produced by the coil where B_r is the remanent flux density of the magnet material, μ_m is its recoil permeability, α is the coil winding efficiency, μ_0 is the permeability of free space, t_c is the coil thickness (or describing dimension normal to the flux path) and $J_{\max}$ is the maximum current density of the coil wire. This relationship assumes the use of a magnetic material with a linear demagnetization curve which is possessed by rare-earth based permanent magnets. The linear dimensional dependence of the coil is readily seen. Equation [1] is plotted in Fig. 1 with various values of $J_{\max}$ and shows that large benefits occur with permanent magnet flux generation in this case below about several millimeters.

The permanent magnet advantages are most fully exploited if in particular rare-earth based permanent magnets (REPM) are considered. These types of magnets of the Sm-Co and Nd-Fe-B family possess very large magnetization per unit volume with high intrinsic coercivities. The large remanent magnetization yields high flux densities which is important for magnetic microactuators where the pressure developed is proportional to the square of the magnetic flux density. The large coercivity makes the REPM highly resistant to demagnetization by external or internal demagnetizing fields. This property reveals itself in the linearity of the B vs. $\mu_0 H$ second quadrant behavior which allows the magnet to be immersed in fields greater than remanence. The result is a nearly constant magnetization in opposing fields as high as 15,000 Oersted thereby establishing a nearly ideal mmf (magnetomotive force) source much like a "magnetic battery." (2) This property of REPM allows for relatively straightforward design of novel magnetic structures which can generate very finely tailored magnetic fields (3,4). The last advantage to be mentioned for permanent magnets is of particular value to static magnetic devices which is that there are no ohmic coil losses or currents required to sustain the magnetic field.

Implementing multipole REPM based structures on the microscale, however, immediately raises the problem of generating the necessary magnetizing field. For typical REPM materials this required field is in excess of 35,000 Oe. As a multipole structure becomes smaller, generating this required alternating high intensity magnetizing field with a magnetizing fixture becomes impractical and eventually not possible. For this reason an assembly technique based on individual precision microfabricated REPM sections is proposed. The fabrication approach to be explained is based on deep x-ray lithography (DXRL) techniques which can facilitate the necessary precision. Microscale assembly techniques that have been further implemented to generate three dimensional microstructures (5) provide the basis for the required assembly.

The resulting permanent magnet structures when augmented with appropriately constructed soft (highly permeable) magnetic materials as well as suitable micro coils form the basis for a more efficient magnetic microactuator technology.

This situation has been appreciated by several previous reports (6,7) which used conventionally machined rare-earth permanent magnets in combination with silicon micromachining. Methods to electroform (8) and pattern ferrite embedded photo polymers (9) have also been reported. What is evident from these studies is the complex implementation problems inherent in batch integration of high performance permanent magnets which must be addressed for MEMS (Micro Electro Mechanical Systems) applications.

DXRL BASED FABRICATION OF REPM

Deep x-ray lithography provides a superb tool for the fabrication of high aspect-ratio microstructures. (10,11) The process creates a mold form in PMMA (poly methyl methacrylate), the preferred deep x-ray photoresist, with nearly perfect prismatic features that possess dimensions down to a few microns, thicknesses up to several millimeters, and tolerances below one micron. This mold form is typically transferred to a complementary metal structure via electroforming. The mold may, however, be used to press other materials as has been demonstrated with ceramics (12,13) which forms the basis for filling DXRL defined molds using powder metallurgy and expands the available materials over electroformable materials considerably. This type of micromold filling process is what is proposed as a basis for precision micro magnet fabrication to reach the ultimate goal of perfectly replicated REPM structures.

A common figure of merit for permanent magnets is their maximum energy product which directly pertains to actuator performance and efficiency. Rare-earth based permanent magnets possess the highest available energy products of over 40 MGOe (Mega-Gauss-Oersted, $1 \text{ MGOe} = 100/4\pi \text{ kJ/m}^3$). These energy products refer to fully dense sintered forms that present a problem for direct replication from PMMA molds due to an unacceptable volume shrink occurring at the required high sintering temperatures. A less involved approach, however, is possible by incorporating a rare-earth magnet powder with a binder such as an epoxy to produce bonded or composite magnets. This type of magnet is experiencing extremely rapid commercial growth due to its ability to be molded and thus inexpensively form precise shapes much more easily than its brittle fully dense REPM counterpart. The concession is a reduction in remanence and thus energy product due to the reduced density. Nevertheless, energy products of bonded REPM of 10 MGOe are common for isotropic powder which is still well in excess of that of ferrites or ceramic magnets. A summary of maximum energy densities possessed by various magnet materials is listed in Table I.

A variety of techniques have been investigated to form bonded REPM directly from a DXRL defined PMMA mold. A basic preferred sequence is outlined in the

Table I. Permanent Magnet Maximum Energy Product Comparison

	<i>Material</i>	<i>Energy Product - MGOe (typ.)</i>	<i>kJ/m³ (typ.)</i>
	Carbon steel	0.25	2
	Alnico	5 - 9 (5.5)	44
	Ferrite (Ceramic)	1 - 4.5 (3.5)	28
	Bonded Ferrite	0.3 - 2 (0.6)	5
	PtCo	7-9	64
	Sm-Co	6 - 31 (26)	208
Rare Earth Based	Nd-Fe-B	22 - 48 (40)	320
	Bonded Nd-Fe-B	6-19 (10)	80

process cross sections of Fig. 2. A DXRL defined PMMA mold is provided on a substrate coated with a suitable sacrificial material such as copper. An unmagnetized powder/epoxy mixture is then applied to the substrate while in a low viscosity state by calendering and pressing with additional centrifugal force being sometimes rendered. Powder particle sizes of less than 20 microns are commonly used with meltspun isotropic Nd₂Fe₁₄B powder being used predominantly.(14) After curing in a press with a typical pressure of 10 ksi, the substrate with pressed composite is lapped to thickness. The entire substrate of permanent magnets is then subjected to a magnetizing field of at least 35 kOe in the desired orientation thereby batch magnetizing all magnets. The PMMA is removed with a solvent to which the epoxy binder is resistant and the permanent magnets are released by undercutting the underlying sacrificial layer.

RESULTS

Bonded REPM with dimensions as small as 5 microns have been made using the DXRL based molds. Results are shown in Fig. 3 for a variety of geometry at a magnet thickness of 200 microns. Most notable is the accurate reproduction of the PMMA sidewall. Distortion is present in the SEM photographs since the magnets are magnetized. This artifact is made evident by using a different accelerating voltage as shown in a) and b) where the 5kV image shows more distortion.

Figure 4 shows the full measured hysteresis loop for isotropic Nd₂Fe₁₄B powder with 20% by volume epoxy that was measured using a squid magnetometer. The remanent magnetization is 0.63 T and the coercivity is 1.4 T with a maximum energy product of near 8 MGOe. The dependence of maximum energy product on epoxy fraction is graphed in Fig. 5. A 30% epoxy volume is used most frequently as this yields magnets with sufficient mechanical strength to be handled during assembly. Greater epoxy fractions increase mechanical strength while further reducing energy product. The maximum energy product for this powder alone without epoxy is 10 MGOe. The results for the smallest volume magnet that was measured are given in Fig. 6. Although still a

strong magnet, it is suspected that a uniform composite density was not achieved as is suggested by the sidewall of the magnets shown in Fig. 3 d). Further improvement of the energy product can be accomplished by utilizing anisotropic powder which in bonded form can attain energy products up to 19 MGOe. Work is being conducted to incorporate these powders which requires pressing in an orienting magnetic field of about 1 T.

APPLICATIONS

Two broad areas of application are of interest for the DXRL defined permanent magnets. The first involves microactuators and is demonstrated by the magnet geometry depicted in Fig. 3. The quadrant shapes are patterned in a way to identify the direction of magnetization resulting from the batch magnetizing step after they are released. Subsequent to release they are assembled together in a cylinder which comprises a multipole rotor for a brushless DC motor. This assembly is shown in Fig. 7 for a 4-pole rotor intended for use with a 9-pole stator which surrounds the permanent magnet rotor. Since the torque developed by such a motor is directly proportional to the number of permanent magnet poles (as long as adjacent pole – pole fringing remains low) further performance improvements may be made by accommodating an 8 pole rotor which has been implemented by using the octant shaped segments shown in Fig. 3c). More complex geometry may profit from assembly approaches involving successive batch wafer transfer. With the use of permanent magnets magnetic microactuators exhibit improved efficiency as has been demonstrated by conventionally machined miniature brushless DC motors.(15) A major class of devices including micro generators and servo systems is also realizable.

The second area of application involves static magnetic structures such as miniature Halbach arrays (or magic rings) and field reinforcing structures that utilize intricate multipole elements. This category also includes charged particle focussing arrangements. Fig. 3c) shows some trapezoidal shapes which when assembled in an array fashioned in a manner depicted in Fig. 8 constitute a Halbach array. Partial arrow shapes on the trapezoidal shapes in Fig. 3c) indicate magnetization direction. Two factors result from this arrangement. The first is that the field generated in the inside region is uniform, and the second is that this inside field is magnified by an amount indicated in equation [2] where B_r is the remanent magnetization, and r_0 and r_i are the

$$B_{\text{inner}} = B_r \ln \left(\frac{r_0}{r_i} \right) \quad [2]$$

respective outer and inner array radii. This practically allows for an increase in magnetic flux density of nearly 2 which increases the magnetic pressure fourfold and makes possible pressures in excess of 300 psi. Such scale independent forces become extremely compelling for use in microactuators. The use of cylindrical Halbach arrays has also been recognized for conventional scale permanent magnet motors. Such an array also has application to microsensors which need a static magnetic bias field such as for magnetically excited flexural plate wave resonators.(16)

Another miniature permanent magnet application is in compensating for a MEMS scaling problem involving springs. Mechanical spring constants scale inversely proportional to length and thus spring geometry can become relatively large on the microscale to yield sufficiently soft springs. The very nearly constant second quadrant magnetization of REPMs leads to interacting behavior very much like mechanical springs with linear spring constants. If permanent magnets are integrated into a spring geometry to produce a repulsion force, this force may be used to subtract from the mechanical spring force resulting in a decreased effective spring constant as described by equation

$$F_s = (k_s - k_{PM})x \quad [3]$$

[3] where k_s is the mechanical spring constant and k_{PM} is the "spring constant" due to the permanent magnet arrangement. One possible arrangement is drawn in Fig. 9. A technique is thereby demonstrated that uses permanent magnets to negate unwanted scaling behavior.

CONCLUSIONS

The use of permanent magnets on the microscale is compelling due to their scaling properties. This is particularly true for rare-earth based permanent magnets which due to their high demagnetization resistance are amenable to flat and nearly arbitrary aspect-ratio. Deep x-ray lithography based processing provides a convenient means to fabricate a multitude of miniature REPMs in a simultaneous batch manner which further facilitate efficient microactuators and other novel microdevices. The nature of multipole REPM structures necessitates an assembly approach which would benefit from batch assembly techniques.

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FIGURES

Fig. 1 Equation [1] plotted for various values of $J_{\max}$ with $B_r=1.2$ T, $\mu_m=1.05$, $\alpha=0.75$, and $\mu_0=4\pi(10)^{-7}$ Hy/m. This plot is intended only as a rough gauge of the trade-off involved between a permanent magnet and a coil in generating a static magnetic field.

Fig. 2 DXRL based bonded REPM process.

b)

b)

d)

d)

Fig. 3 Bonded $\text{Nd}_2\text{Fe}_{14}\text{B}$ micromagnets fabricated using DXRL molding at 200 micron thickness. a) and b) show quadrant shaped geometry which are intended for assembly into a 2 pole-pair permanent magnet rotor. The magnets are magnetized in the direction indicated by the arrow in a). Distortion is present as an artifact of the magnetic field interaction with the SEM imaging and is evident by comparing a) and b) which were taken with different accelerating potentials. c) shows magnet shapes for a 4 pole-pair permanent magnet rotor along with trapezoidal shapes to be used in a magic ring as described in Fig. 8. d) indicates that bar shaped magnets may be processed. Permanent magnet features as small as 5 microns have been replicated at 200 micron structure height.

Fig. 4 Full hysteresis loop of bonded isotropic $\text{Nd}_2\text{Fe}_{14}\text{B}$ powder in 20%/vol epoxy with a density of 6.3 g/cm^3 . The remanent magnetization is 0.64 T and the coercivity is 1.4 T.

Fig. 5 Maximum energy product as a function of epoxy fraction. An increased epoxy fraction improves the mechanical strength of the permanent magnet.

30 volume
% epoxy

$$H_{\text{coer}} = 1.4 \text{ T}$$

$$(B \cdot H)_{\text{max}} = 3.7 \text{ MG} \cdot \text{Oe}$$

Fig. 6 Data from the smallest permanent magnet which was measured

a)

b)

Fig. 7 Assembled multipole rotors for brushless DC motor.

Fig. 8 Magic ring approximation by discrete permanent magnet elements.

Fig. 9 Schematic diagram demonstrating possible integration of permanent magnets into a “crab-leg” flexure to provide an effectively reduced spring constant.

FABRICATION OF MEMS DEVICES BY POWDER-FILLING INTO DXRL-FORMED MOLDS

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ABSTRACT

We have developed a variety of processes for fabricating components for micro devices based on deep x-ray lithography (DXRL). Although the techniques are applicable to many materials, we have demonstrated them using hard ($\text{Nd}_2\text{Fe}_{14}\text{B}$) and soft (Ni-Zn ferrite) magnetic materials because of the importance of these materials in magnetic micro-actuators and other devices and because of the difficulty fabricating them by other means. The simplest technique involves pressing a mixture of magnetic powder and a binder into a DXRL-formed mold. In the second technique, powder is pressed into the mold and then sintered to densify. The other two processes involve pressing at high temperature either powder or a dense bulk material into a ceramic mold that was previously made using a DXRL mold. These techniques allow arbitrary 2-dimensional shapes to be made 10 to 1000 μm thick with in-plane dimensions as small as 50 μm and dimensional tolerances in the micron range. Bonded isotropic $\text{Nd}_2\text{Fe}_{14}\text{B}$ micro-magnets made by these processes had an energy product of 7 MGOe.

INTRODUCTION

The LIGA process has been developed in recent years as a means of producing metal parts in the 10 to 1000 μm size range for micro-electromechanical (MEMS) systems.^{1,2} In this process, an x-ray mask, consisting of a pattern of an x-ray absorber such as gold on a low Z support, is used to expose regions of a PMMA layer on a substrate to synchrotron x-rays (DXRL). Since synchrotron x-rays are nearly parallel and have high energy, highly precise patterns with straight sidewalls can be formed in a PMMA layer up to 1 mm thick after exposure and development. Once the PMMA mold is formed, it is filled by plating a metal into the desired regions, utilizing the preexisting plating base. This limits the standard LIGA process to materials that can be electroplated. The excess plated metal is then removed by precision lapping, leaving parts of desired thickness that are then released from the substrate by dissolving the plating base layer.

Permanent (hard) magnet materials have numerous applications in MEMS including micro-actuators and static field devices such as Halbach arrays.³ They are particularly useful because the strength of the magnetic field that they can produce is independent of scale, as opposed to electromagnets where the field scales with length.⁴ The hard magnetic materials with the best properties are rare-earth transition metal compounds such as $\text{Nd}_2\text{Fe}_{14}\text{B}$.⁵ These materials can have energy products over 40 MGOe, over 5 times what can be achieved with other materials. However, they cannot readily be plated

because of their complex chemistry and microstructure and therefore cannot be made using the standard LIGA process. Macroscopic magnets of these materials are commercially fabricated in bonded form, which consists of a magnetic powder and an epoxy binder, or, for better properties, in a dense form made by either sintering or hot-pressing.⁶ Also, isotropic hot-pressed magnets can be subsequently hot-forged, or die-upset, to create anisotropic magnets with their preferred direction parallel to the pressing direction.⁶

Soft magnetic materials also have applications in MEMS as cores in electromagnets, for example. Since many MEMS devices operate at relatively high frequency, eddy current losses, which increase as the square of the frequency, make metallic soft magnetic materials useless in these situations. The best high frequency soft magnetic materials are ceramic ferrites that have very high electrical resistivity and therefore low eddy current losses.⁷ These materials also cannot be electroplated.

To fabricate micro-components of these magnetic materials, we have developed a variety of processes based on filling DXRL-formed PMMA molds with magnetic powders.⁸ These techniques keep the benefits of the LIGA process (arbitrary shape, small size, high precision, high aspect ratio and batch fabrication) without having the limitation of electroplating.

EXPERIMENT

The DXRL molds used in this work were fabricated on either Si wafer or alumina substrates. For bonded magnets, a copper release layer 0.5 μm thick was sputter coated onto the substrate prior to bonding on the PMMA. The PMMA layers ranged in thickness from 100 to 500 μm and were exposed under x-ray masks using a synchrotron facilities at the Center for Advanced Microstructure Devices at Louisiana St. University.

Bonded Magnets

Both isotropic and anisotropic bonded magnets were fabricated from $\text{Nd}_2\text{Fe}_{14}\text{B}$ powder and epoxy using PMMA DXRL molds on Si wafers with a Cu release layer. For isotropic magnets, Magnequench MQP-C powder was used. The fabrication process for isotropic magnets is shown in Figure 1. The powder was ground in inert atmosphere and then sieved to 20 to 25 μm typically. It was then mixed with a room temperature curing epoxy to give 30 to 40 vol% epoxy, applied to the mold by a calendering process and then uniaxially pressed at 10 ksi.. After curing, the excess material was removed by precision lapping and the PMMA was removed. The parts were then magnetized at 35 kOe using a magnetizer (LDJ Electronics). Finally, the parts were released by dissolving the release layer in an etchant.

Figure 1. The process for fabricating bonded isotropic magnets from LIGA molds: 1) LIGA mold 2) Apply powder and binder 3) Lap 4) Magnetize 5) Dissolve PMMA and 6) Dissolve release layer.

Figure 2. The process for fabricating sintered magnets from LIGA molds: 1) LIGA mold 2) Apply powder 3) Lap 4) Burnout PMMA and 5) Sinter.

A similar process was used to fabricate bonded anisotropic magnets, as shown in Figure 2. In this case, Magnequench MQT (HDDR) powder was used after grinding and magnetizing at 35 kOe. The powder was mixed with epoxy and calendered onto the mold. The mold was then placed between the poles of an electromagnet and a field of 10 kOe was applied to align the particles. While the field was on, a clamp was used to compress the aligned powder mixture and it was subsequently pressed at 10 ksi after removal from the field. The rest of the process was the same as for the isotropic case.

Sintered Magnets

The process used to make sintered micro-magnets is shown in Fig. 3. In this case, a Ni-Zn ferrite powder (Steward, #xx) was used as received. The powder was mixed with a solvent and a dispersant to form a paste that was then spread onto a PMMA LIGA mold on a Si wafer and dried. The excess powder was removed by lapping and the mold was then heated at 1°C/min in air to 500°C to burnout the PMMA. At this point, the parts released from the substrate and were transferred to an alumina plate for sintering. Sintering was done in air at 1300°C for 4 hr in a closed crucible that contained an excess of the starting powder to limit Zn loss.

Hot-Pressed and Hot-Forged Magnets

To fabricate micro-magnets by hot pressing or hot forging, a mold that can withstand the required pressure and temperature must first be made since a standard PMMA mold is obviously inadequate. Typical hot pressing conditions for $\text{Nd}_2\text{Fe}_{14}\text{B}$ materials are 750°C at 15 ksi. Since the mold must not deform if precise dimensions are to be maintained, it is desirable that the mold be made of a ceramic such alumina, as most metals will deform severely under these conditions. Therefore, a process, shown in Figure 3, was developed to fabricate alumina molds from PMMA LIGA molds. First, a PMMA LIGA mold with the PMMA in the shapes of the desired magnetic parts on an alumina substrate was fabricated. Alumina powder was applied to the mold either by uniaxial pressing or by slurry casting with or without the aid of a centrifuge. We used a 94% alumina powder

with 2 μm average particle size. The excess alumina was removed by lapping and the PMMA was then burned out. The sample was then heated to 1250°C in air for 2 hr to give the alumina sufficient strength without causing significant densification, which would distort the desired shapes. Two SEM images of a fired alumina mold with a 250 μm thick alumina layer are shown in Figure 4. As can be seen, features in the 100 μm range were reproduced with relatively straight sidewalls.

Figure 3. Process for forming a ceramic mold from a LIGA mold: 1) LIGA mold on alumina 2) Apply alumina powder 3) Lap 4) Burnout PMMA and 5) Bisque fire.

Figure 4. SEM images of a bisque-fired alumina mold made by the process in Fig. 3 for hot pressing and forging.

The process used to make $\text{Nd}_2\text{Fe}_{14}\text{B}$ micro-magnets by hot-pressing is shown in Figure 5. To form isotropic magnets, MCP-C powder was used after being ground in inert atmosphere to $\sim 20 \mu\text{m}$ particle size. A 1 μm thick release layer of copper was first sputter coated onto the alumina mold using conditions that maximize sidewall coverage. Copper was chosen because it is inert with respect to the $\text{Nd}_2\text{Fe}_{14}\text{B}$ at the pressing temperature and because it is soluble in a solvent that does not react with the $\text{Nd}_2\text{Fe}_{14}\text{B}$. A layer of the powder about 2 mm thick was pressed onto the mold at room temperature and

Figure 5. The process for fabricating micro-magnets By hot-pressing: 1) Ceramic mod 2) Coat with release Layer 3) Apply powder 4) Hot-press 5) Lap and Magnetize and 6) Dissolve release layer

Figure 6. The process for fabricating hot-forged micro-magnets: 1) Ceramic mold 2) Coat with release layer 3) Apply slice of bulk magnet 4) Hot-forge 5) Lap and magnetize and 6) Dissolve release layer.

the sample was then heated under high vacuum to 700°C at which time a compressive stress of 10 ksi was applied for 30 min. After cooling, the excess material was lapped off and the sample was magnetized. Finally, the copper layer was dissolved in ammonium hydroxide to release the parts. Anisotropic magnets were made by a similar process using powder formed by regrinding a bulk $\text{Nd}_2\text{Fe}_{14}\text{B}$ MQP-3 magnet from Magnequench to a 20 μm particle size. This is an anisotropic material that was made by hot-pressing and then die-upsetting a melt-spun powder. The powder was then magnetized and placed on the copper coated alumina mold in a 10 kOe field to align the particles. The powder was then vacuum hot-pressed, lapped, magnetized and released as described above.

The hot-forging process developed to make $\text{Nd}_2\text{Fe}_{14}\text{B}$ micro-magnets is shown in Figure 6. The process is essentially identical to the hot-pressing process except that a dense slice of Magnequench MQP-3 magnet 2 mm thick was used instead of powder. The slice was oriented with its preferred direction parallel to the plane of the substrate in the desired direction with respect to the shapes of the parts.

RESULTS

Bonded isotropic micro-magnets are shown in Figure 7. Shapes with high dimensional tolerance with features below 100 μm were formed as thick as 0.5 mm. A SQUID magnetometer was used to measure the magnetic hysteresis loop for some of the parts. The measurements indicated that the isotropic magnets had an energy product of 7 MGOe.

Figure 7. SEM micrographs of isotropic bonded micromagnets.

Two SEM images of a micro-toroid of Ni-Zn ferrite made by the sintering process are shown in Figure 8. The toroid has an OD of 800 μm and a wall thickness of 80 μm , indicating that 20% linear shrinkage occurred during sintering since the initial dimensions were 1 mm and 100 μm , respectively. The micrograph indicates that full density was achieved with an average grain size of about 15 μm , a microstructure that produces optimal properties in this materials.⁷ Also, no distortion occurred to the circular shape of the toroid during processing.

Figure 8. SEM micrographs of a Ni-Zn ferrites micro-toroid made using sintering-DXRL process.

The hot-pressing and hot-forging processes were developed as ways to produce dense parts without the in-plane shrinkage that occurs during sintering so that higher precision could be achieved. If the mold used in these processes is rigid, as we believe it was for the alumina molds, then the part tolerance is determined by the initial mold tolerance. This is in turn set by any distortion that occurs during bisque-firing of the mold. We noticed a slight sloping of the sidewalls on our alumina molds. Using hot-forging, parts 100 μm wide and 250 μm thick were fabricated using the following pressing conditions: 800°C, 15 ksi for 1 hr.

Each of the four techniques developed has their own relative advantages and disadvantages. The bonded magnet technique is the simplest and yields the most precise parts. However, it gives the worst magnetic properties due to the presence of the bonding phase. The sintering method is also relatively simple and it can produce dense parts with excellent properties. However, dimensional change due to sintering shrinkage must be compensated for in mask design and final part dimensional tolerance is decreased due to shrinkage variability. The hot-pressing and hot-forging techniques are the most difficult since they require the fabrication of a ceramic mold and the use of a hot-press. However, they yield excellent properties and have the potential for excellent dimensional tolerance if the tolerance of the ceramic mold is optimized.

CONCLUSIONS

Four processes have been developed for fabricating both hard and soft micro-magnets based on the LIGA process. They were used to make magnets with precise dimensions in the 100 to 1000 μm range with excellent magnetic properties. Finally, these processes are applicable to many other materials that may be useful in MEMS applications that cannot be made using the traditional LIGA technique.

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APPLICATION OF DEEP X-RAY LITHOGRAPHY FABRICATED RARE-EARTH PERMANENT MAGNETS TO MULTIPOLE MAGNETIC MICROACTUATORS

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ABSTRACT

Deep x-ray lithography patterned molds with submicron tolerances are demonstrated to be of direct utility in fabricating precision bonded rare-earth permanent micro-magnets. Using a melt-spun isotropic powder/epoxy composite, precision prismatic shaped magnets with maximum energy products of 8 MGOe, features as small as 5 microns, and thicknesses as great as 750 microns have been realized with direct molding techniques. The resulting precision magnet geometry facilitates subsequent assembly into multipole permanent magnet arrangements with very fine scale magnetic field control. Static device applications include magic ring structures for the production of high uniform magnetic fields in submillimeter volumes. Microactuator applications are presented in the form of a 5 mm diameter 4 and 8 pole rotor for a brushless DC motor. Design and construction of a resulting brushless DC motor is presented.

INTRODUCTION

The importance of permanent magnets to MEMS is apparent from their scaling behavior: the magnetization possessed by a permanent magnet is independent of scale. This size independence is in contrast to electromagnetic coils that have a detrimental scaling dependence due to current density limits resulting from thermal and electromigration constraints that are also shared by superconducting coils. Magnetic microactuators profit from this permanent magnet property which partially offsets the efficiency loss resulting from the adverse scaling of miniature electromagnetic coils. A direct comparison of electromagnetic coil and permanent magnet performance as a function of size is in general difficult as it depends on the particular actuator function. A rough comparison, however, may be made for the simple case of a "horseshoe" magnet geometry which is a common component for many magnetic actuators. In this case, the ratio, γ_H , of magnetic flux generating capability of an equivalent volume of permanent magnet to electromagnetic coil is derived to have the relationship in equation (1), where γ_H is the ratio of magnetic field produced

$$\gamma_H = \frac{B_r / \mu_m}{\alpha \mu_0 t_c J_{\max}} \quad (1)$$

by the permanent magnet to that produced by the coil, B_r is the remanent magnetic flux density of the magnet material, μ_m is the recoil permeability of the permanent magnet, α is the coil winding efficiency, μ_0 is the permeability of free space, t_c is the coil thickness (dimension normal to the flux path), and $J_{\max}$ is the maximum current density of the coil wire. This expression is derived for the case of a permanent magnet with a linear demagnetization curve which is a property of rare-earth permanent magnet materials at room temperature. The inverse length scale dependence is evident and the plot in Fig.1 of this relationship for various maximum current densities shows the dimensional regimes where permanent magnets gain a significant advantage.

Fig. 1 Plot of Eqn. (1) for various maximum coil current densities with $B_r = 1.2$ T, $\mu_m = 1.05$, and $\alpha = 0.75$.

The merits of permanent magnets for microactuators are best exhibited by the family of rare-earth based materials including the Nd-Fe-B and Sm-Co forms. The permanent magnet figure of merit most pertinent to actuators is their maximum energy product. Rare-earth based permanent magnets possess by far the largest energy products and have the additional benefit of a linear demagnetization curve that makes possible intricately tailored multipole magnetic structures

[1,2,3]. This linear demagnetization character leads to a nearly constant magnetization thereby yielding a nearly ideal mmf (magneto-motive-force) source which behaves like a "magnetic battery"[4]. Since this mmf source is realized without ohmic losses it is ideally suited to microdevices.

Despite their merits, there has been very little attention to batch planar processed rare-earth permanent magnets in MEMS work. Conventionally fabricated rare-earth permanent magnets have been reported to be used in conjunction with silicon micromachining [5,6]. Photopatternable ferrite magnets [7] and electroformed Co-Ni-Mn-P magnetic materials [8] have also been reported to be used in batch processed microactuators.

An additional issue in permanent magnet based devices pertains to their orientation or magnetization direction. The previously cited work utilizes magnets with a single orientation relative to the substrate. Obtaining multipole or multiple orientation magnet arrays within a device requires either a localized magnetization scheme or an assembly technique which may include batch assembly approaches. Locally magnetizing permanent magnet materials with alternating structure, however, becomes prohibitively difficult on the microscale. This is particularly true for rare-earth based permanent magnets which require magnetizing fields in excess of 35,000 Oe. An assembly approach has therefore been taken similar to that of the LIGA assembly approach [9]. Two fabrication schemes are being pursued which are outlined in the following section. The design and fabrication of a brushless DC motor utilizing a multipole permanent magnet rotor constructed with precision batch fabricated rare-earth permanent magnet components is then described.

DXRL PERMANENT MAGNET PROCESS

Deep x-ray lithography (DXRL) is commonly used to form precision electroforming molds using PMMA as the x-ray photoresist. The unique attribute of this mold is its submicron tolerances that it obtains for any prismatic geometry to thicknesses of over 1 mm. This mold is typically transferred to a metal component via electroforming. This type of mold, however, may also be used as a starting point for forming other pressed materials [10]. A bonded form of rare-earth permanent magnet material was found to be well suited to this type of forming and the resulting process is outlined in cross-section in Fig. 2. Initially, a composite of rare-earth permanent magnet powder and a binder such as epoxy is calendered and pressed in a low viscosity state into a DXRL defined PMMA mold. Unmagnetized meltspun isotropic $\text{Nd}_2\text{Fe}_{14}\text{B}$ powder particle sizes of between 3 and 20 microns are commonly used and are prepared with

the epoxy in an inert ambient. The preferred epoxy is Master Bond EP41S-1 which has chemical resistance to organic solvents. Subsequent to curing in a press at 7 ksi at room temperature a grinding/lapping step planarizes this pressed composite to a desired thickness. All magnets are then batch magnetized directly on the substrate with a field greater than 35,000 Oe. The magnetization will accommodate any batch alignment desired including normal to the substrate as well as any direction in the plane of the substrate. The PMMA mold material is then dissolved away without perturbing the permanent magnet arrays and the magnets are batch released from the substrate by etching the sacrificial copper layer.

Fig. 2 Batch DXRL-based process for bonded rare-earth permanent magnets.

Figure 3 shows typical resulting bonded NdFeB permanent magnets before release. Energy products between 3 and 8 MGOe (Mega-Gauss-Oersted, $1 \text{ MGOe} = 100/4\pi \text{ kJ/m}^3$) have been achieved for an epoxy volume fraction between 50 and 20% which is typical for bonded rare-earth permanent magnets. An increased mechanical strength is obtained with higher volume fractions of epoxy with a concession in energy product due to decreased magnetic flux density. A M-H curve resulting from a 20% epoxy bonded $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnet is shown in Fig. 4. Bonded magnets with lateral dimensions as small as 5 microns with hundred micron heights have been produced in this fashion.

A second process flow has been attempted to make use of anisotropic rare-earth magnet powder which requires application of a magnetic field near 1 Tesla

Fig. 3 Bonded $\text{Nd}_2\text{Fe}_{14}\text{B}$ permanent magnets fabricated with the use of DXRL defined PMMA molds. (a) quadrant sections of a 4 pole rotor for a brushless DC motor with the direction of magnetization shown by the arrow, some distortion is present in the SEM image due to the magnetic field present from the permanent magnets. (b) octant sections for the same rotor and trapezoidal shapes aligned in orientations suitable for an octagonal representation of a magic ring or a Halbach array (c) bar magnets $75\mu\text{m}$ wide with a thickness of $250\mu\text{m}$.

Fig. 4 M-H characteristic of 20%/volume epoxy bonded $\text{Nd}_2\text{Fe}_{14}\text{B}$ permanent micro-magnet with 0.64 T remanence and 1.4 T coercivity.

before and during pressing of the composite material to align the powder. This technique has reported possible ultimate energy products of 19 MGOe in bonded form. Only a fraction of this has been achieved to date, however, presumably due to the difficulty of alignment.

APPLICATIONS: BRUSHLESS DC MOTOR

Design

In order to demonstrate the implementation of multipole DXRL fabricated permanent magnets, an 8 mm diameter brushless DC motor was fabricated. A 9-slot, 4-pole inner rotor configuration was chosen with the layout depicted in Fig. 5. This particular design accommodates low-profile motor geometry similar to that used in portable magnetic hard disk drives [11,12]. In order to minimize cogging torque,

Fig. 5 9-4 Brushless DC motor layout. The multipole permanent magnet rotor is 5 mm in diameter.

a slotless stator was implemented. Due to the precision miniature nature of the individual permanent magnet sections, a radial-anisotropic magnet rotor is possible as opposed to pole-anisotropic magnets that have been implemented in multipole rotors as small as 1cm [11] and that appear to have reached the dimensional limit for continuous multipole rare-earth permanent magnets. The target maximum torque for this motor is 50 μ N-m at 1.5 mm thickness.

Fig. 6 Plan view (a) and close-up (b,c) photographs of the 4 pole brushless DC motor.

Fabrication

The motor is fabricated on a DXRL defined PMMA base-plate that is used to accommodate the press fit of a miniature ball bearing (RMB Miniature Bearings, Inc.) and alignment gauge pins. The permanent magnet rotor is

press fit on a 80 Permalloy hub attached to a 320 micron diameter shaft. The coils are wound on DXRL defined PMMA forms with 100 turns of 47 AWG magnet wire, potted, and then freed by dissolving the PMMA. The coils are then inserted in a radial orientation on a stator winding form also patterned via DXRL processing which is inserted inside the outer magnetic return path ring. The completed motor is shown in Fig 6. An excitation of 1 mA is found to be sufficient to induce rotor rotation. Complete motor characterization is underway.

CONCLUSIONS

Bonded rare-earth permanent magnets defined by DXRL processing are well suited for multipole magnetic microactuators. Further work to achieve fully dense rare-earth magnets is being attempted with alumina mold forms replicated from PMMA. Batch wafer transfer techniques are the next step in further integrating the magnet arrays.

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THE DEVELOPMENT OF A CERAMIC MOLD FOR HOT-FORGING OF MICRO-MAGNETS

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ABSTRACT

A new mold material has been developed for use in making rare-earth permanent magnet components with precise dimensions in the 10 to 1000 μm range by hot-forging. These molds are made from molds poly(methyl)methacrylate (PMMA) made by deep x-ray lithography (DXRL). An alumina bonded with colloidal silica has been developed for use in these molds. This material can be heated to 950°C without changing dimensions where it develops the strength needed to withstand the hot-forging conditions (750°C, 100 MPa). In addition, it disintegrates in HF so that parts can be easily removed after forging.

INTRODUCTION

Most components for meso- and micro-electromechanical systems have to date been fabricated using either silicon micromachining or the LIGA process. These processes limit the choice of materials to silicon and silicon dioxide for silicon micromachining and to metals that can be electroplated for LIGA. However, a variety of applications exist where other materials are desired. Examples include hard magnetic materials such as $\text{Nd}_2\text{Fe}_{14}\text{B}$, soft magnetic materials for high frequency applications, such as ferrites, and potentially other ceramics for, for example, high temperature applications.

Although limited to metals that can be plated, the LIGA process has a number of attractive attributes. The deep x-ray lithography (DXRL) process used in LIGA allows parts with arbitrary shapes in two dimensions in the 10 to 1000 μm size range with very high dimensional precision to be readily produced. Sidewalls are vertical and up to >1mm thick, allowing for high aspect ratio parts to be fabricated. The batch nature of the process is also an advantage in comparison to techniques where each part must be machine individually.

Several processes¹⁻⁴ have been reported recently that are modifications of the LIGA process that can fabricate micro-components of magnetic and other materials that cannot be electroplated. Some of these processes^{3,4} are based on filling DXRL formed poly-(methyl)methacrylate (PMMA) molds with powders. Bonded parts were made simply by filling the mold with a mixture of powder and epoxy, lapping off the excess after curing and then releasing the parts by dissolving a thin metal release layer (see Figure 1). Dense parts were made by filling the mold with powder and then sintering (see Figure 2). However, the

Figure 1. Bonded $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnet.

Figure 2. Sintered Mn-Zn ferrite toroid.

Figure 3. The process for fabricating micro-magnets by hot-pressing: 1) Ceramic mold 2) Coat with release layer 3) Apply powder 4) Hot-press 5) Lap and magnetize and 6) Dissolve release

Figure 4. The process for fabricating hot-forged micro-magnets: 1) Ceramic mold 2) Coat with release layer 3) Apply slice of bulk magnet 4) Hot-forge 5) Lap and magnetize and 6) Dissolve release layer.

shrinkage that occurs during sintering requires molds to be designed oversize and the shrinkage variability decreases dimensional precision. These drawbacks were overcome by either by hot-pressing metal or ceramic powder (Figure 3) or by hot-forging (Figure 4) metal slabs into a ceramic mold that was made from a DXRL PMMA mold. The ceramic mold was made by pressing a 94% alumina powder into the PMMA mold, lapping off the excess, burning off the PMMA and then bisque firing to develop the required strength. A layer of copper was then sputter coated on the mold to serve as a release layer.

Although somewhat successful, several problems were encountered during hot pressing and forging using these molds. The first problem occurred during the bisque firing required to develop adequate strength in the mold. Although the ceramic molds were highly precise replica of the PMMA mold prior to bisque firing, as shown in Figure 5, cracking and deformation of the alumina at the sidewalls due to shrinkage and delamination between the mold alumina and the dense alumina substrate took place (see Figure 6). Thus parts produced with such a mold did not have desired dimensions. The second problem was that the release of parts, especially the smaller ones, was difficult

because of the difficulty in coating vertical sidewalls in narrow gaps with the copper release layer using sputtering. The final problem was the production of theoretically dense parts by hot pressing. The pressing was done by covering the mold with a uniform layer of powder and then pressing with a flat plunger. In other words, there was not a matching shaped plunger for each part, as this would have required a difficult blind alignment of the two parts of the mold. Therefore, densification of the parts was impeded both by the limited stress transfer into the cavities and because when the powder regions between the parts reached theoretical density, further densification of the parts was stopped.

Figure 5. A 94% alumina mold before firing.

Figure 6. A 94% alumina mold after bisque firing showing distortion and cracking.

In the present work, a new mold material was developed to overcome first two problems mentioned above. This material consisted of an alumina that was bonded using colloidal silica instead of by bisque firing. The hope was that the colloidal silica particles would deposit at the contact regions between the alumina particles and then densify there during heating to increase the strength of the alumina skeleton without causing shrinkage. Such a mold would be usable up to the temperature at which the silica would start to become deformable, about 1000°C. Since the alumina would be bonded by the silica, dissolution of the silica in HF would then cause the mold to disintegrate, thereby facilitating the release of the parts.

EXPERIMENTAL PROCEDURE

Several compositions of different alumina powders and colloidal silica suspensions were initially investigated to determine which was most suitable for making molds. The alumina powder that worked the best was Alcoa A-14 (Alcoa,), with a 2 μm average particle size, since cracking during drying occurred with smaller particle size alumina and the A-14 gave better strength and surface finish than larger particle size alumina powders. The colloidal silica with the smallest particle size of those studied (2 nm), Nyacol 215 (Nyacol Products, Ashland, MA), gave the highest strength films when mixed with the A-14.

To make a mold for hot-forging, a PMMA DXRL mold with the PMMA in the shapes of the desired magnetic parts on an alumina substrate was first fabricated. The PMMA layers ranged in thickness from 100 to 500 μm and the minimum feature size was 10 μm .

The PMMA molds were exposed under x-ray masks using the synchrotron facilities at the Center for Advanced Microstructure Devices at Louisiana State University, Baton Rouge. This mold was then coated with a slurry containing 1 ml of Nyacol 215 (15 wt% SiO₂) for every 2 g of alumina. After drying the slurry on a hotplate at 100°C, the colloidal suspension was applied directly to the coating and it was dried again, after removal of the excess suspension. This process was then repeated until no more of the suspension could wick into the layer. The shrinkage of a piece of this material was measured during heating at 10°C/min to 950°C in air using a video system. In the same way, the shrinkage of a dried piece of the colloidal silica was measured.

The excess alumina was removed by lapping and the PMMA was then burned out. The sample was then heated to 950°C in air for 1 hr to give the mold sufficient strength to withstand the pressure used to hot-forge Nd₂Fe₁₄B. Molds were examined with both optical and scanning electron microscopy. The dimensions of features were carefully compared before and after heating using the SEM.

The heated alumina-silica mold was then used to form parts by hot-forging using a disk of a Magnequench MQ3-F magnet (Magnequench, Anderson, IN) 2 mm thick using the process previously developed (see Figure 4). This is a high energy-product, anisotropic Nd₂Fe₁₄B material. The disk was placed inside a graphite die of the same diameter to constrain lateral motion of the metal during forging. The slice was oriented with its preferred direction parallel to the plane of the substrate in the desired direction with respect to the shapes of the parts. The forging was done in high vacuum at 750°C and 100 MPa for 30 min. After forging, the magnetic material and the mold separated from the alumina substrate. The sample was then soaked in concentrated HF for 1 min to cause the mold to disintegrate.

RESULTS

The results of the sintering experiments are shown in Figure 7. The colloidal

Figure 7. The shrinkage of the colloidal silica and the alumina-silica mixture during heating.

silica began to shrink around 550°C and reached a maximum shrinkage of 11% by 850°C. The mixture, on the other hand, did not change dimensions, to within the accuracy of the measurement (+/- 1%). The SEM micrographs taken before and after heating confirmed that the mold did not measurably change dimensions with heating to 950°C. At room temperature, the mold material that was previously heated to only 700°C could withstand a compressive load of 200 MPa without deformation.

Two SEM images of a fired alumina mold with a 250 μm thick alumina layer are shown in Figures 8 and 9. Figure 8 shows that the sidewalls were vertical and that no delamination occurred where the sidewall contacts the substrate. However, some voids were present in the sidewalls. The micrographs in Figure 10 show that the mold was uniform over a large areas and that features as narrow as 10 μm could be accurately produced in a 200 μm thick mold.

Figure 8. SEM image of a heat-treated mold showing no sidewall distortion.

Figure 9. The microstructure of mold the mold after heating.

Figure 10. Optical micrographs of a fired mold.

The optical micrographs shown in Figure 11 show that after soaking in the HF, the mold disintegrated leaving the forged parts. However, the sidewalls of the parts were somewhat rough.

Figure 11. Optical micrographs of hot-forged $\text{Nd}_2\text{Fe}_{14}\text{B}$ bars in top and side views.

DISCUSSION

The shrinkage data in Figure 7 indicate that even though the silica material reaches high density by 950°C , the alumina-silica mixture undergoes essentially no shrinkage during heating to this temperature. This implies that the alumina particles form a network of contacts that is not deformed by the densification of the silica regions. The fact that the densification of the silica substantially increases the strength of the material without producing any overall shrinkage implies that the silica is in large part located at the necks between the alumina particles. This conclusion is supported by the fact that the material disintegrated in HF. This would be expected since the drying suspension would form menisci there, deposited the silica particles there. Although the silica did not densify further above 900°C , it may be possible to further increase the strength of the mold by higher temperature heating through the formation of mullite around 1000°C . However, this could produce a dimensional change and would affect the ability of the material to disintegrate in HF.

As in the previous work⁴, the PMMA mold could be filled with the mold material to form precise features after pyrolysis of the PMMA. However, in the case of the colloidally bonded silica mold, no cracking or distortion was apparent after heating to 950°C , as shown in Figures 10 and 11. Features as fine as 10 by $400\ \mu\text{m}$ could be formed in a $200\ \mu\text{m}$ thick film.

The $\text{Nd}_2\text{Fe}_{14}\text{B}$ material used in the hot-forging experiments had previously been plastically deformed at high temperature in the die-upsetting process to induce magnetic anisotropy.⁵ In this process, the preferred magnet direction is parallel to the direction that the material is compressed. In the present case, it was desired that the preferred direction be in the plane of the mold so that the material was orientated that way initially. During the forging process, some of this anisotropy will be destroyed since the material is being compressed perpendicular to the desired direction in order to force it into the cavities in the mold.

The hot-forging was successful at formed parts down to the $50\ \mu\text{m}$ range, as shown in Figure 11. However, the surfaces of the parts were rough. At least two causes for this behavior can be suggested. First, the magnetic material could have been forced into the pre-existing voids in the sidewalls of the mold. Second, addition sideways motion of the magnetic material could have occurred into locally weak areas of the mold. A combination of these processes likely occurred.

CONCLUSIONS

A colloiddally bonded alumina material has been developed for use in molds for fabricating micro-magnets by hot-forging. The material did not undergo any shrinkage during heating so that molds with precise dimensions down to 10 μm could be created. Heating the mold to 950°C gave it enough strength to be able to successfully hot-forge $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets with feature below 100 μm . The mold could then be disintegrated by an HF treatment to aid in removal of the part from the mold. However, some surface imperfections were apparent on the forged parts due either to low mold strength or to voids in its surface.

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Microfabrication of Fully-Dense Rare-Earth Permanent Magnets via Deep X-ray Lithography and Hot Forging

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The impact of permanent magnets on microactuator designs would be greater if a convenient means to integrate them into a batch fabrication scheme could be realized. The scale independent magnetic field produced from a permanent magnet is a property possessed by permanent magnets which makes their use appropriate in the micro-domain. As a result of this scaling, magnetic microactuators using permanent magnets are more efficient than those using only coils. The characteristics of rare-earth based permanent magnets with high resistance to demagnetization and high remanence makes their use the most compelling. Fabricating these magnets to MEMS scale with corresponding micron size tolerances is difficult, however, particularly when more complex multipole arrangements are desired.

The magnet microfabrication approach which has been studied involves the use of a deep x-ray lithography (DXRL) defined PMMA mold as a precision starting geometry to transfer to rare-earth permanent magnet (REPM) material. Initial work centered on bonded rare-earth magnets composed of rare-earth permanent magnet powder and a binder epoxy which may be replicated directly from the PMMA mold by calendaring and pressing techniques.[1] The magnet geometry is subsequently batch magnetized and released. The results are precision bonded REPM with energy products from 6 to 10 MGOe (Mega-Gauss-Oersted) depending on epoxy binder concentration. In order to achieve higher density and thus higher energy product magnets, higher temperature processing is required which requires a mold that can withstand higher temperatures. This mold may then be used for hot pressing of powders or hot forging. The technique is outlined in Fig. 1.

Alumina is used as an intermediate mold and is obtained by applying alumina powder in a uniaxial press or by slurry casting into a PMMA mold residing on an alumina substrate in a manner similar to that described by [2]. The excess alumina is removed by lapping and the PMMA is burned out during an intermediate sintering step. The substrate is then heated to 1250 deg. C in air for 2 hours to yield an alumina mold without significant densification in order to avoid geometry distortion. The results are shown in Fig. 2. The mold is then coated with evaporated copper to provide a release layer for the permanent magnets. Two techniques have been pursued to fill the mold with permanent magnet material. Hot pressing is more suited for isotropic REPM material since anisotropic powder must be aligned during the pressing via a die upset-like technique. The ability to hot forge pre-aligned REPM material has been most recently demonstrated and has the advantage that alignment is already provided and the handling of powder is not

necessary. Strain rates for Magnequench MQ2-F die upset NdFeB material were measured to be 22% at 2000 psi and 700 deg. C for 500 sec. which is well suited for the 200 to 500 micron magnet thickness of interest. The result is batch microfabricated permanent magnets with energy products approaching 35 MGOe.

Fig. 1 Hot forged REPM microfabrication process.

Fig. 2 Alumina hot forge molds replicated from DXRL defined PMMA.

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