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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 $Nd_2Fe_{14}B$, 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 processes[1-4] 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 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,

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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.

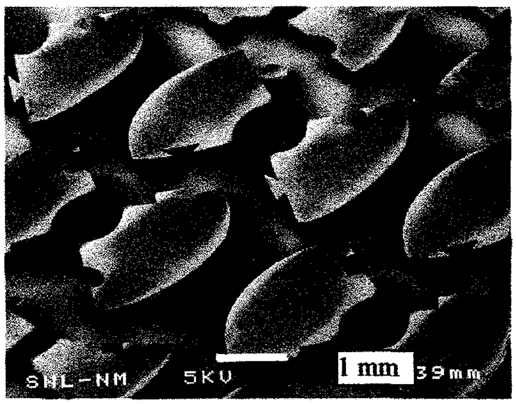

Figure 1. Bonded $Nd_2Fe_{14}B$ magnet.

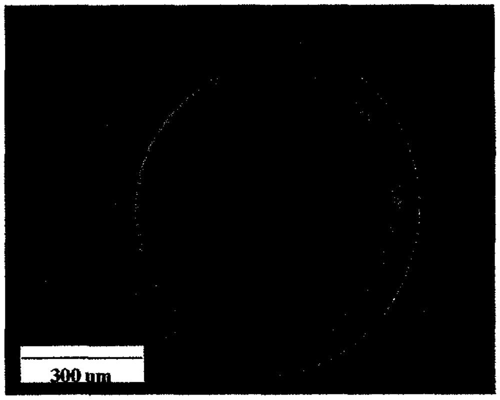

Figure 2. Sintered Mn-Zn ferrite toriod.

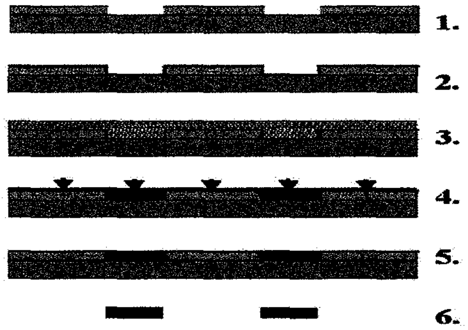

Figure 3. The process for fabricating micro-magnets by hot-pressing: 1) Ceramic mold 2) Coat with release layer 3) Apply powder 4) Hot-press 5) Lap and magnetize and 6) Dissolve release layer.

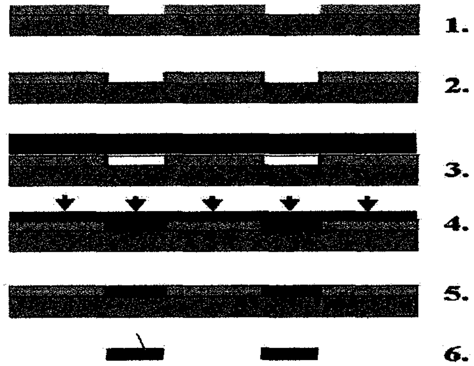

Figure 4. The process for fabricating hot-forged micro-magnets: 1) Ceramic mold 2) Coat with release layer 3) Apply slice of bulk magnet 4) Hot-forge 5) Lap and magnetize and 6) Dissolve release layer.

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.

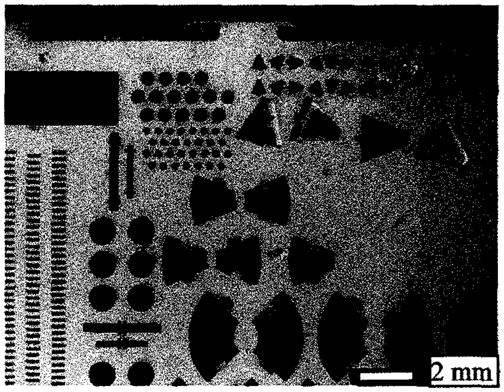

Figure 5. A 94% alumina mold before bisque firing.

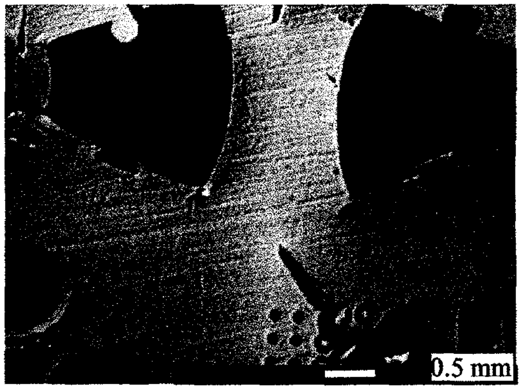

Figure 6. A 94% alumina mold after bisque firing showing distortion and cracking.

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% SiO_2) 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 $Nd_2Fe_{14}B$. 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 MQ2-F magnet (Magnequench, Anderson, IN) 2 mm thick using the process previously developed (see Figure 4). This is a high energy-product, anisotropic $Nd_2Fe_{14}B$ 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 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.

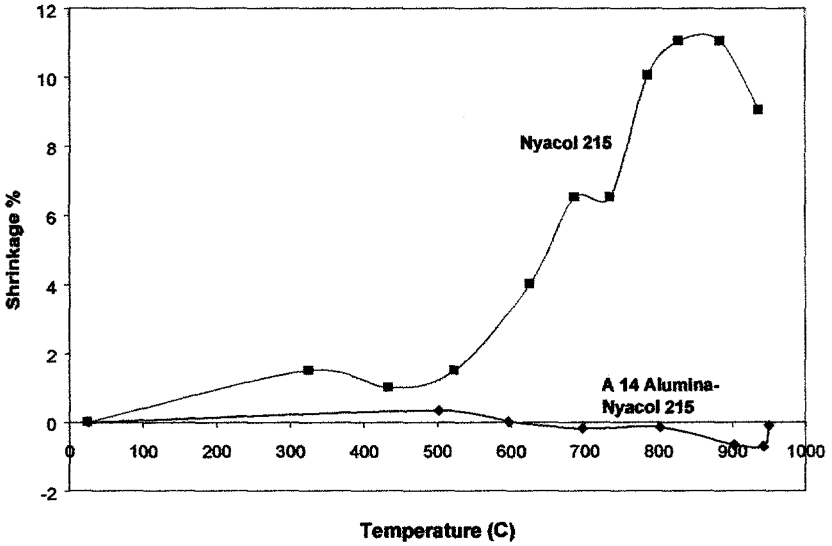

Figure 7. The shrinkage of the colloidal silica and the alumina-silica mixture during heating.

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.

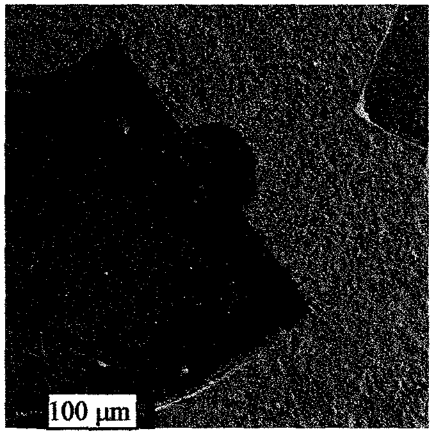

Figure 8. SEM image of a heat-treated mold showing no sidewall distortion.

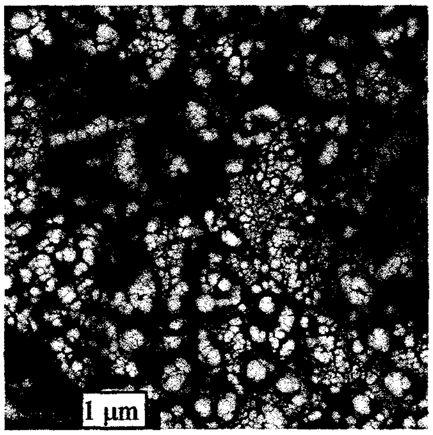

Figure 9. The microstructure of the mold after heating.

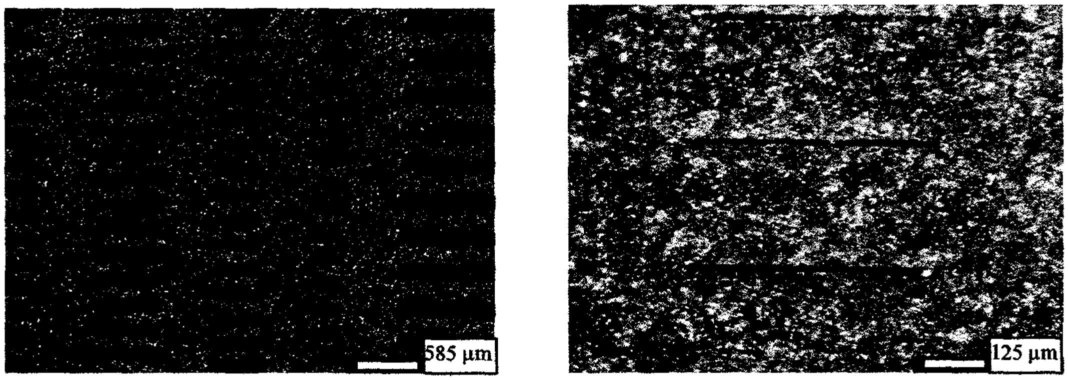

Figure 10. Optical micrographs of a fired mold.

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.

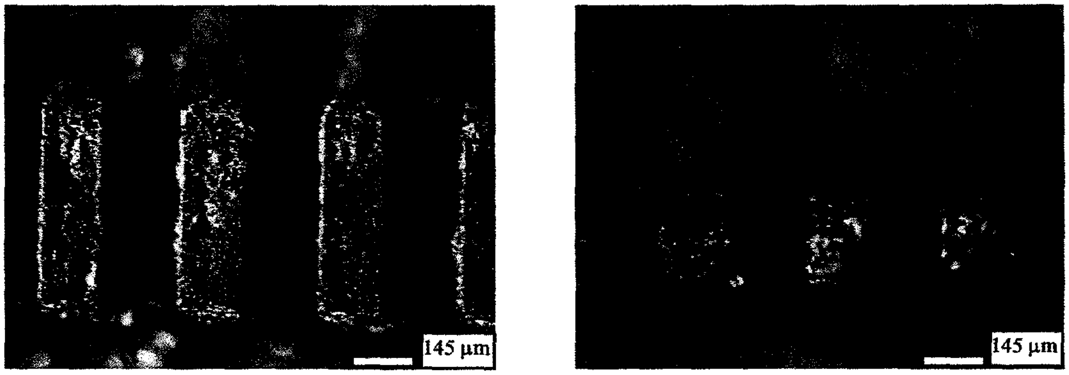

Figure 11. Optical micrographs of hot-forged $Nd_2Fe_{14}B$ bars in top and side views.

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 work[4], 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 μm could be formed in a 200 μm thick film.

The $Nd_2Fe_{14}B$ 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 μm 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 colloidally 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 $Nd_2Fe_{14}B$ 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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