Ferrite Filled Polymers for Integrated Power Conversion Devices

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Abstract

Passive magnetic devices such as inductors, transformers, and square loop core devices, are often currently mounted on MCM substrates in a hybrid fashion. Integrating these devices in a fashion compatible with MCM-L substrates is desirable in many applications. Materials and methods for deposition of both soft and hard mag- netic materials onto MCM-L compatible substrates are currently under investigation. In this work, we assess the suitability of hard ferrite-filled polymers in the realization of integrated passive magnetic elements. Strontium fer- rite powder (particle size between 1.15 and 1.5 *J1m) has been mixed with various polyimides, and the resulting materials have been studied in terms of processabiliry, magnetic and mechanical properties. The range of concen- tration loading of the ferrite powder was between* 55 *and 80 vol.* %. *11ze resultant material has been deposited and patterned both by screen-printing and spin-coating followed by photolithography. The 80 vol. % ferrite load- ing samples exhibit square loop behavior with high intrinsic coerciviry Hci = 4000 De, residual magnetic flux Br - 3000 Gauss, and maximum energy product (BH)max=* 1.8 *MGDe, approaching bulk ferrite properties. Mechanical properties of the films have also been measured, with residual stresses ranging from* 31-33 *MPa anli biaxial moduli ranging from* 6-16 *GPa depending on the volume loading of ferrite material. Based on these results, the material is a promising candidate for realization of MCM-L compatibi.e integrated magnetic components.*

Key words: polymer composite, magnetic materials, low temperature

Introduction

Recently there has been much interest in both hard and soft magnetic materials in microelectronics. Applications such as integrated square-loop core devices [1], [2], integrated inductive components [3], electromagnetic interference shielding, and others have been proposed. However, materials which have suitable magnetic properties are often not compatible with microelectronics processes, since they require processes such as pressing, molding, sintering, or other processes. In packaging technologies, one successful approach for integrated MCM-compatible magnetic components is firing feITite-filled inks on ceramic substrates. However, the required processing temperatures are not compatible with the organic substrates such as those used for MCM-L. In these cases, the use of a filled polymer approach is desir- able.

In this work, we describe and characterize a mate- rial suitable for integration of magnetic components with low temperature MCM substrates. This mate- rial is based on a filled polymer approach to realize low temperature, processable films with hard magnetic properties. The characteristics of interest include the magnetic properties (intrinsic coercivity Hc, residual magnetic flux density Br, and maximum energy product (BH)max), the mechanical properties (Young's modulus, and residual tensile stress), and the processing characteristics of these materials.

Materials Considerations

Materials which have suitable hard magnetic properties are usually not compatible with microelectronics processes. For example, the preparation cf samarium-cobalt and neodymium-iron-boron magnets usually requires high temperature, pressing, and sintering. Magnetic polymer composites, in which small particles of magnetic material are suspended in a nonmagnetic (e.g., polymeric) matrix or binder, are a good compromise to combine the favorable properties of the magnetic material with the simple processing sequences of the polymer. Three types of polymer binder can be used: elastomeric, thermoplastic, or thermoset. The choice of the polymer matrix depends on its ability to accept large volume loading of magnetic powder, and the type of process used to make the finished magnet. In this work, it was also impor- tant to consider the temperature limitations of MCM- L. As a first step, the polyimide PI-2555 ftom DuPont, was used as the binder material. It consti- tutes a favorable binder material, due to its common use in microelectronics (and therefore its well-known processing characteristics), and also due to its favor- able chemical and mechanical properties. Although the recommended curing temperature of this poly- imide (300 °C) is still higher than the required tem- perature for the MCM-L substrates «200 °C), longer cure times at lower temperatures, or use of a pre- imidized material, can lower the required processing temperatures into the acceptable range. Other microe- lectronics-compatible materials, such as benzocy- clobutene, polyolefin materials, or epoxies, can also be suitable binder materials.

There are a variety of pennanent magnet materi- als commercially available which might be candidates for the powder in a magnetic composite. Such materi- als can be characterized by their magnetization curve, 47tM vs. H, and the curve of the external energy product, (BH) vs. B. These curves yield the residual induction Br, the intrinsic coercive force Hci, and the maximum energy product (BH)max. Table I (taken from [4]) shows a comparison of some common per- manent magnet materials available commercially in powder fonn.

In tenns of large Br, Hci, and (BH)max. the materi- als with the best magnetic properties are the rare earth alloys. However, it is not easy to use these materials in powder fonn suitable for composites since they are susceptible to oxidation and corrosion and the resul- tant loss of their desirable magnetic properties. Ce:. ramic ferrites, although possessing lower valuesofBr, Hci, and (BH)max, do not suffer from these problems. In addition, they are by far the most widely used type of pennanent magnet material. During their produc- tion the powder may be milled to particles that are approximately single domain size (about I I1m-l.5 11m diameter). This fabrication process results in the magnetic properties of these ceramic ferrites being based on magnetic anisotropy, which produces a magnet with a high coercivity and an almost square second quadrant B versus H characteristic. Their great popularity is mainly due, though, to the low cost and great abundance of their raw materials. In particular, strontium ferrite can be easily found in powder fonn with low particle size (1-1.5I1m), at low cost. In ad- dition, these ferrites are very stable chemically. Due to these reasons, strontium ferrite powder was se- lected as the filler material in the magnetic compos- ite.

Composite Fabrication

The polymer magnet is composed of 1.5 11m strontium ferrite particles produced by Hoosier Mag- netics and Dupont PI-2555 polyimide (a benzophe- none tetracarboxylic dianhydride-oxydianiline / meta- phenylene diamine formulation). The composite is then formed by introducing various quantities (loading) of the magnetic particles into the poly- imide. Various additives are optionally used to im- prove particle dispersion. The materials are mixed using a ball mill rotating at 4-5 rpm. The mixing period is held for at least 72 hours in order to insure homogeneity of the mixed composite solution. The polymer/ferrite liquid suspension is then deposited on a suitable substrate and patterned, e.g., by screen printing or spin-casting followed by photolithogra- phy. After deposition and patterning, the magnetic composite is cured to achieve its final properties. The composition of the magnets described below is based upon the weight percentages of the two constituents in the fully cured film. The weight of the polyimide in the cured film is calculated using the average per- cent solids of the polyimide solution.

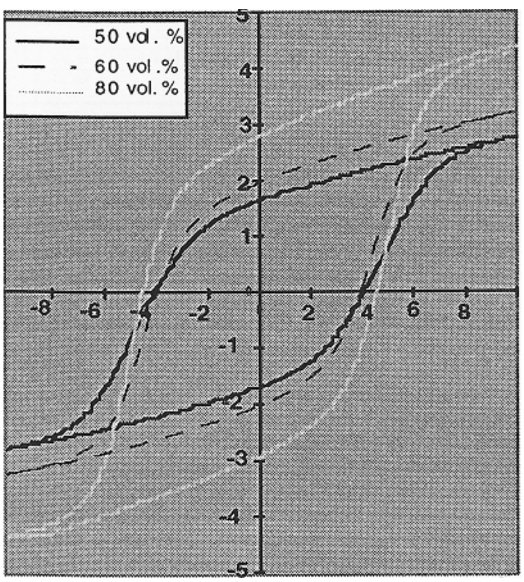
Table 1. Material comparison chart [4]

Materials Class	Rare Earth Alloys	Ceramic Ferrites
Residual fu- duction Br	- 9,000 Gauss	3,600 Gauss
futrinsic Co- ercivity RCf	- 15,000 Oe	> **4,000 Oe**
Maximum Energy Prod- uct(BR)max	- 19.5 MGOe	- 3.05 MGOe
Chemical Stability	Corrosion Sen- sitive	Very Stable
Powder Form	Not Commonly Utilized	Commonly Utilized
Particle Size	50 - 250 ~m	LU~~- ~
Relative Cost -	High	

Magnetic Properties

Test specimens were composed of square mag- netic polymer sheets of - 9 11m thickness, and 2x2 cm area, deposited on square glass slides by using a multi coat procedure. A single coat of the multicoat procedure consists of a 3000 rpm, 40 s spin cycle followed by a 15 min soft bake at 120 °C. The mul- ticoat is then fully cured at 300 °c for one hour in a conventional oven. Upon final cure, the films are ex- posed to an external magnetic field, to compensate the reversible remanence loss due to the high tem- perature exposure during the polyimide curing. Fi- nally the polymer magnet films are removed from their glass substrates for testing.

Magnetic properties were measured by a vibrat- ing sample magnetometer recording the magnetiza- tion curve of the material, or the magnetization 47tM (Gauss) as a function of the applied magnetic field H (Oe). Figure I shows a typical 47tM-H curve of this material, for different ferrite loading concentrations. For a 80 vol. % sample, an intrinsic coercivity Hci cf approximately 4000 Oe, a residual induction Br ap- proaching 3000 Gauss, and a maximum energy prod- uct (BH)maxof 1.5 MGOe, were achieved. The overall 47tM-H curve also demonstrates the typical square loop behavior of a permanent magnet., and the mag- netic properties of the 80 vol. % sample are very closed to those of the bulk ferrite.



C/)

C/)

H **(KOe)**

Figure 1. Magnetization curves for different ferrite loading concentrations.

Mechanical Properties

The mechanical characteristics are determined by an analysis of the load / deflection behavior of a membrane [5] made from the ma~etic polymer mate- rial. The load / deflection testing is performed using square membranes of the thin films which are fabri- cated on a silicon substrate. The suspended mem- branes are realized using standard micromachining techniques. In the fabrication process, 2 inch < 100> wafers are heavily boron doped (> 1020 / cm3) to form a p+ etch stop layer approximately 3-5 11m thick. Silicon nitride, 5000 A thick, is deposited using plasma CVD and is used as an etch mask for both sides of the wafer. Square windows are patterned in the Si3N4 on the unpolished side of the wafer using a buffered oxide etchaht. The wafer is then anisotropi- cally etched from the unpolished side in a 20 wt % potassium hydroxide solution heated to 56 °c to produce a silicon membrane. After the silicon etch is complete, a thin film of magnetic composite material is applied on the Si3N4 on the polished side of the wafer using the same single coat procedure as for the preparation of the composite samples for the magnetic measurement. When the composite film has been fully cured at 300 °c for one hour, the p+ etch stop layer and Si3N4 layer are removed from the membrane regions using wet etching: 20 % H2O, 75 % nitric acid and 5 % HF, resulting in the finished composite membrane. At this point, the mechanical properties (plane strain modulus and residual stress) can be de- termined from the load/deflection behavior of the membrane.

The mechanical characteristics are determined by an analysis of the load/deflection behavior of a mem- braneusing an energy minimization approach [6], but modified to account for the presence of residual tensile stress and square dimensions [7-8]. For a membrane of side length 2a and thickness t, it can be shown that the relationship between the applied pressure and the deflection at the center of the membrane is given by:

(1)

(2)

where P is the applied pressure, K is the plain strain modulus of the composite film, 0"0 is the residual stress in the film, and E and \) are the Young's modulus and Poisson's ratio of the film, respectively.

The characterization of the thin films is carried out using a material characterization station which allows application and simultaneous measurement cf a pressure or vacuum load to the membrane. The apparatus is placed on a microscope stage. The deflec- tion of the membrane at its center is measured by focusing on the membrane center and measuring the amount of microscope head travel necessary to keep the membrane in focus using a a z-axis digimatic indicator mounted on the microscope head. Figure f shows typical load-deflection data taken in this man- nelofa 55 vol.% composite membrane. Examination of equation (1) shows that if pressure-deflection data are plotted with [pa2/dt] on the y-axis and [(d/a)2] on the x axis, the data should fall on a straight line. The residual stress can then be determined from the y- intercept of the line, and the plane strain modulus from the slope of the line. Figure 3 shows the load- deflection data of figure 2 plotted in accordance with equation (1). Similar measurements and data analysis on several membranes for each composite give the average values ofK and 0'0 for loadings ranging from 55 - 80 vol %. and are shown in Table 2. It appears that the biaxial Young's modulus increases with the ferrite loading concentration. The residual stresses in the film remain constant with values similar to that of unloaded polyimide. From these data it is reasonable to assume that the coefficient of thermal expansion, which is related to the residual stress, will b the same order of magnitude of the polyimde PI-2555 (40 ppm/oC).

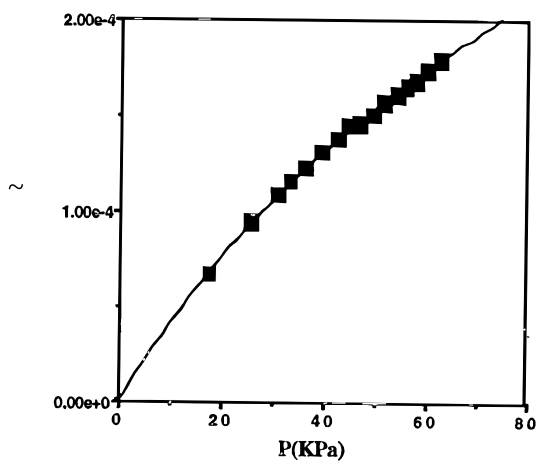


Figure 2. Typicalload/deflection characteristics for the magnetic polymer composite (55 vol. 0/0 loading)

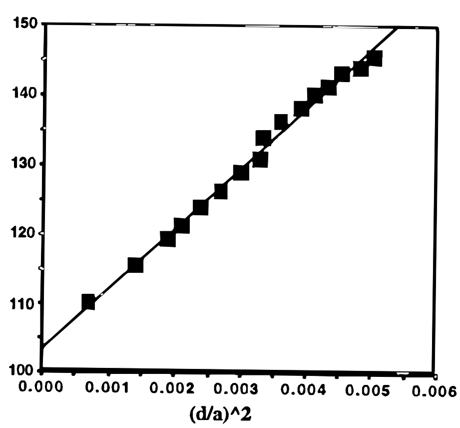


Figure 3. Load/deflection data plotted in accor- dance with Eq.(l) for the magnetic polymer com- posites composite (55 vol. % loading)

Processability

The realized polymer magnetic composite can be patterned in several ways. Screen-printing deposition is an attractive technique due to its capacity to produce thick devices (100 **I.Lm** thick devices have been produced without difficulty). Using this technique, magnets ranging from 250 microns to centimeters in width can be easily achieved. Figure 4 shows a screen-printed film with 250 micron features. Moreo~ ver it has also been demonstrated that this material can be patterned by using standard photolitography and wet etching of the polyimide composite. The process consits of depositing by spin-coating the magnetic polymer composite, soft-baking it, then depositing on the top of the composite standard pho- toresist, and soft-baking the photoresis and exposing using ultraviolet lithography. Upon developing, the photoresist developer also etches exposed regions cf the composite. After etching and residual photoresist removal, the composite is fully cured. Devices on the order of 200 microns in width have also been fabricated using this technique.

Table 2. Summary of composite mechanical properties as a function of loading

Ferrite Concentration vol. %	55	65	80
E k = 1=;: GPa	6..57 0.6	8.44 0..2	16.725 1.1
D,MPa	33.33 0.3	34.86£ **0.41**	31.85 £ 0.2

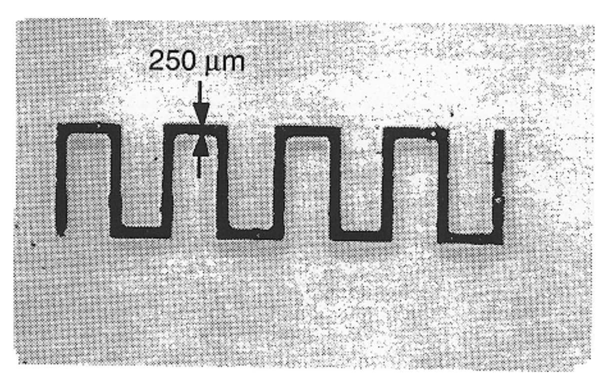


Figure 4. Screen-printed polymer magnet

Conclusions

Filled polymer magnetic materials possessing hard magnetic properties, and good mechanical prop~ erties, have been prepared, deposited, and patterned using a variety of techniques. The magnetic properties of the polymer composite with 80 vol.% loading concentration in ferrite, approach those of the bulk ferrite. The biaxial Young's modulus of the composites increases with the ferrite concentrations, but still remains reasonable even for high ferrite loadings. The residual stress in the composite films is stable, and approaches that of unloaded polyimide; thus, it is reasonable to assume that the coefficient of thermal expansion of the composites is reasonably independent of loading. The processing of these composites is compatible with organic MCM-L substrates. Other microelectronics-compatible materials, such as benzocyclobutene, polyolefin materials, or epoxies, can also be suitable binder materials. Current work in- volves the application of these materials in combina- tion with analogously-prepared soft magnetic polymer composites to integrated magnetic components.

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