Permanent magnetic NdFeB thick films

B. PAWLOWSKI
Hermsdorfer Institut für Technische Keramik e.V., M.-Curie-Str. 17, 07629 Hermsdorf, Germany

J. TÖPFER*
Hermsdorfer Institut für Technische Keramik e.V., M.-Curie-Str. 17, 07629 Hermsdorf, Germany; Fachhochschule Jena, FB Werkstofftechnik, Carl-Zeiss-Promenade 2, 07745 Jena, Germany
E-mail: joerg.toepfer@fh-jena.de

NdFeB films of thickness between 10 to 50 μm were prepared by screen printing of inks containing 70–87 wt% of commercial NdFeB powder (MQP). After curing the printed films at 120°C magnetic films with a remanence of 200 to 400 mT are obtained. The coercivity varies from 300 to 900 kA/m depending on the type of MQP powder used. Magnetic inks with higher loading of NdFeB powders (80–91 wt%) are used for filling of vias or patterned substrates.

NdFeB thick films are promising for applications in micro-electromechanical systems or miniature actuators. Printed films were magnetized with a multi-pole stripe pattern with 1 mm pole pitch using short current pulses. The induction at the surface of the films was compared to theoretical calculations. The results indicate a complete magnetization of the films. Other applications include encoders.

1. Introduction

The application of magnetic components in micro-electro-mechanical systems (MEMS) or micro-systems requires miniaturized permanent magnets. Usually small magnets are cut out of sintered blocks, magnetized and then the individual magnets are placed into their final positions. This is a cost-intensive manufacturing process and moreover, due to the fact that the magnets are magnetized before assembly, handling and positioning of such miniature magnets is not straightforward. Permanent magnetic thick films deposited on micro-system components acting as substrate represent an alternative manufacturing technology. Multi-pole magnetization of the films after assembly is an additional advantage, many quasi-individual micro-magnets can be fabricated within one magnetization step.

For such applications rare-earth magnets with large remanent induction and energy product are used. To date, techniques for the preparation of rare-earth magnetic thick films with appropriate magnetic properties are not well investigated. Thin film techniques like laser deposition, sputtering and molecular beam epitaxy have been successfully tested for the preparation of NdFeB thin films [1–3], but such films are too thin for most actuator applications. The current status of permanent magnetic films for micro-systems is summarized in [4, 5]. Only very few papers on the preparation of NdFeB thick films have appeared: arc-plasma spraying has been used for the preparation of films of up to 3 mm in thickness [6]. A different approach consists of the compaction of granulated NdFeB-powders from the powder-metallurgical process. Thin plate magnets of 200 μm thickness with excellent magnetic properties have been prepared [7]. The fabrication of NdFeB films of 100–800 μm thickness by tape casting has been reported [8]. Recently, screen printing of Sr hexaferrite inks has been suggested for the preparation of permanent magnet thick films [9]. Screen printing of rare-earth magnet films has not been reported yet.

In this contribution we describe the preparation of magnetic thick films by screen printing of NdFeB inks. Commercial NdFeB powders were used as starting materials for the ink preparation. After printing and curing at 120°C films of 10–50 μm thickness with a remanence of 200–400 mT and coercivity of 300–900 kA/m are obtained. The films can be magnetized with a multi-pole stripe pattern with a pole pitch of 1 mm. The induction at the film surface was measured and compared to calculations. It is demonstrated that the printed films are applicable in actuators or magnetic encoders.

2. Experimental

Commercial NdFeB powders (Magnequench International [10]) prepared by melt-spinning (MQP-B and -Q) or atomization (MQP-S) were used as starting materials. The powders were wet milled for several hours in a porcelain mill with steel balls (7 mm diameter) in a mixture of organic solvents.

* Author to whom all correspondence should be addressed.

0022–2461

For the preparation of inks the NdFeB powder was dispersed in a resin dissolved in a mixture of organic solvents. The loading of magnetic powder in the ink was varied from 70 to 91 wt%.

The inks were screen printed onto several substrate materials (soft iron, alumina, glass, silicon). Several test geometries were printed: from areas of size 50 × 50 mm^2 to several fine structures with a width of the printed magnet track of 100 μm. The thickness of the obtained film after a single printing step was about 10 μm, multiple printing gave films of 50 μm thickness. Inks of larger viscosity were used for filling of vias or cavities in patterned substrates. The films were annealed at 120°C for 2 h; curing of the resin yields mechanically stable films.

The particle size of the magnetic powder was determined by laser diffraction with a SYMPATEC system with a dry dispersion unit RODOS. The density of the test samples made from cured inks was determined by Archimedes' method in water.

Demagnetization curves of the films were measured with a vibrating sample magnetometer (corrected for demagnetizing fields). Temperature coefficients were obtained by measuring the hysteresis at different temperatures between 20 and 125°C.

To study the multi-pole magnetization films 50 × 50 mm^2 in size were printed onto soft iron substrates of 0.5 mm thickness. After curing at 120°C the magnetic films show a good adherence to the substrates. Multi-pole magnetization with a stripe pattern of 1 mm pole pitch was realized by pulse magnetization (Plötner & Hoffmann, IM142/35); a capacitor discharge battery provides a high current pulse of short duration (semi-sinusoidal impulse). The desired multi-pole pattern is obtained by placing the magnetic film on top of a copper wire wound fixture and discharging the capacitor bank through the fixture. The discharge voltage was 3.0 kV at maximum and a current of about 18 kA was observed. The induction at the surface of the films was measured with a magnetic field scanning system (Plötner & Hoffmann, SMP386/01) with a Hall probe scanning the surface of the film at a distance of 250 μm.

3. Results and discussion

The NdFeB powders used in this study are MQP-B, -Q and -S powders. After wet milling the MQP-B powder for 20 h a final medium particle size of about 10 μm is reached. MQP-Q powders were milled for about 50 h to obtain a fine powder. The details of NdFeB powder (MQP) milling are given elsewhere [8]. REM micrographs of the powders before and after wet milling show a transition from melt-spun coarse flakes to fine powders (Fig. 1a–b). Spherical MQP-S powder which is manufactured by an atomization process has a mean particle size of about 50 μm (Fig. 1c), hence only large particles were removed by sieving.

Films of various thickness ranging from 10–50 μm were prepared by screen printing. A typical composition contains about 80 wt% MQP and 20% resin and organic vehicle.

In order to obtain a large remanence of the inks and films the content of magnetic powder in the ink was

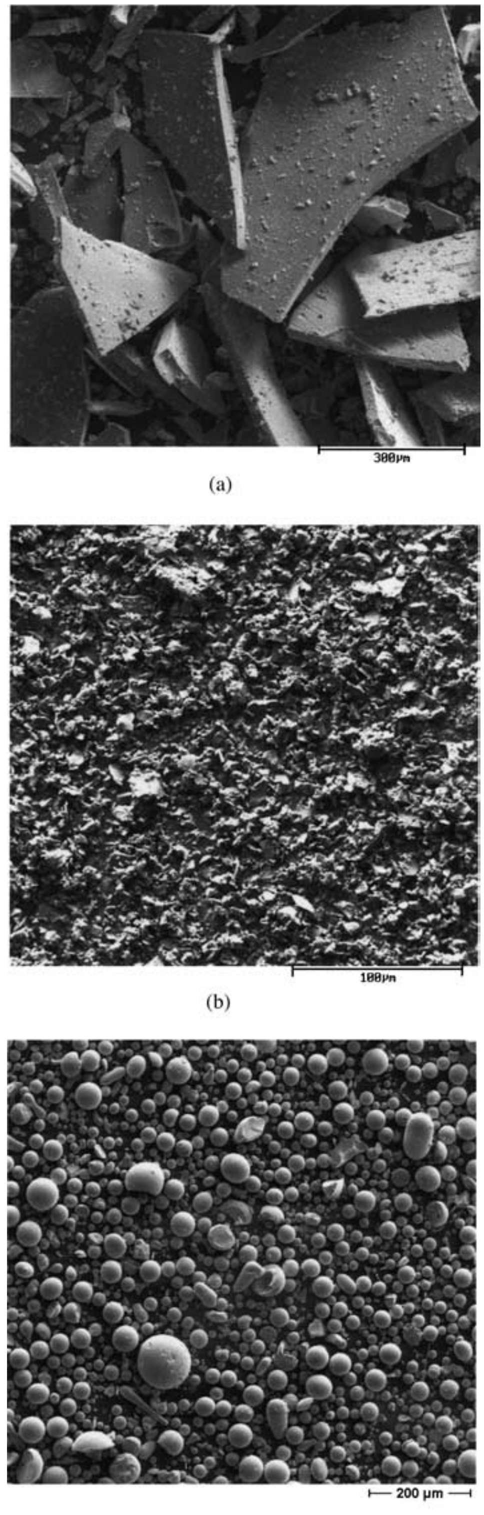

Figure 1 REM micrograph of powders MQP-B, original flakes (a) and powder milled for 24 h (b) and MQP-S original spherical powder (c).

optimized. The maximum loading of the fine-milled MQ powder is about 85 wt%; samples with a higher concentration of powder can not be processed to homogeneous inks. An estimate of the maximum powder content is obtained if the tap density of the fine-milled MQP-B powder ($\rho_{tap} = 3.34$ g/cm^3) is compared to the materials density ($\rho = 7.64$ g/cm^3). From this particle packing situation a total interparticle volume of 56% results. This represents the space that is available to be filled with polymer. Lower organic content will result in

TABLE I Remanent induction B_R of cured MQP-B inks with different powder loading

Ink	wt-%	vol-%	B_R (mT) calc	B_R (mT) exp
MQP-B	70	24.6	212–220	200
	75	29.6	254–264	237
	80	35.9	308–321	305
	85	44.2	380–396	388

pores and powder agglomerates in the ink. Powders of grade MQP-S with a mean particle size of about 50 μm have a tap density of 4.42 g/cm^3, therefore the empty interparticle volume is smaller. Consequently, less organic medium is required to fill this space. Inks from MQP-S powder are prepared with a maximum content of 91 wt% powder.

From the volume loading of magnetic powder the remanence of the cured ink is calculated. For MQP-B based samples (B_R of the powder: 860–895 mT [10]) the remanent induction of the cured ink is expected to be between 250–400 mT (Table I). The calculated B_R agree well with measured ones. The demagnetization curves of MQP-B inks with different powder loading are shown in Fig. 2. Demagnetization curves of inks with standard composition of 80 wt% MQP-B, -O and -Q are compared to that of an ink with 91 wt% MQP-S in Fig. 3. The coercivities $_IH_C$ are 700, 350, 900 and 750 kA/m for MQP-B, -Q, -O and -S inks, respectively. Temperature coefficients (Fig. 4) are $TK(B_R) = -0.13\%/K$ and $TK(_IH_C) = -0.37\%/K$.

Inks for screen printing need to have an appropriate viscosity to be processable without problems. The maximum powder loading for NdFeB inks for screen printing based upon the fine-milled powders MQP-B and -Q is about 80 wt%, which corresponds to a B_R of 320 mT. In the case of spherical MQP-S powder a beneficial effect appears: inks with about 87 wt% powder ($B_R = 400$ mT) have a viscosity that allows processing

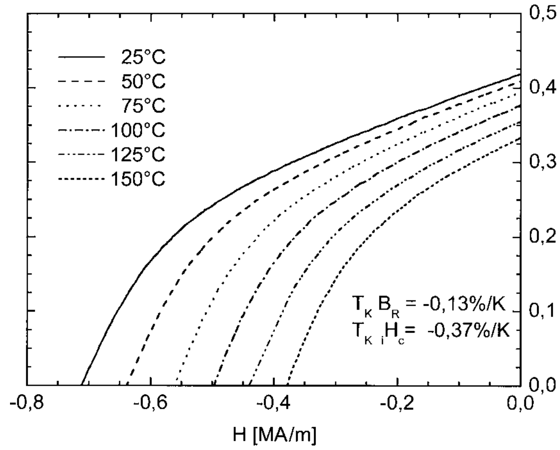

Figure 4 Demagnetization curves of a cured MQP-S ink (91 wt%) at different temperatures.

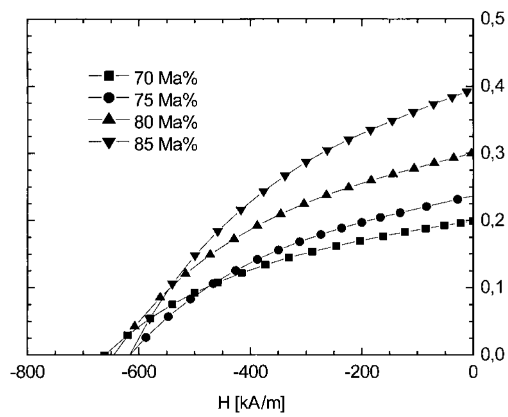

Figure 2 Demagnetization curves at room temperature of cured MQP-B inks with various loading of magnetic powder.

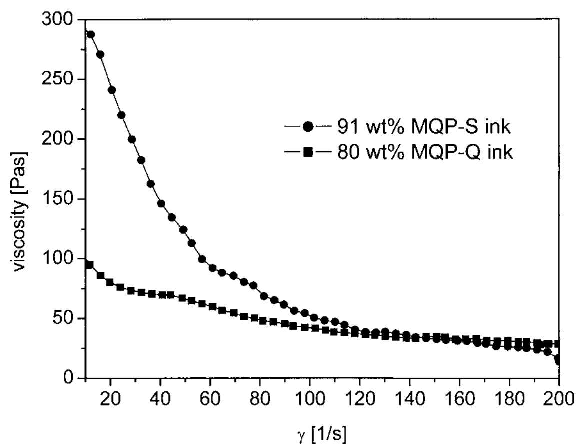

Figure 5 Room temperature viscosity as a function of shear rate of inks with 80 wt% of MQP-Q and 91 wt% of MQP-S.

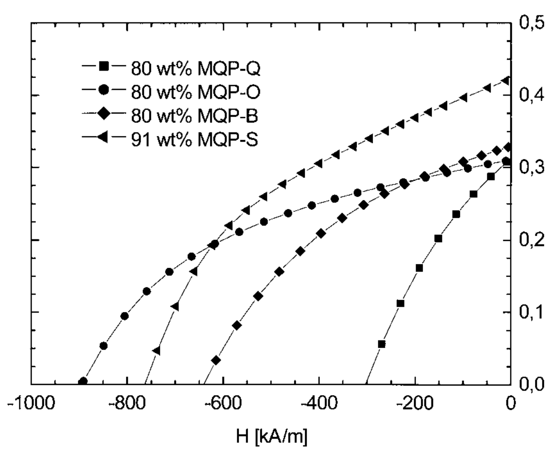

Figure 3 Demagnetization curves at room temperature of MQP-B, MQP-Q, MQP-O inks (80 wt%) and MQP-S ink (91 wt%).

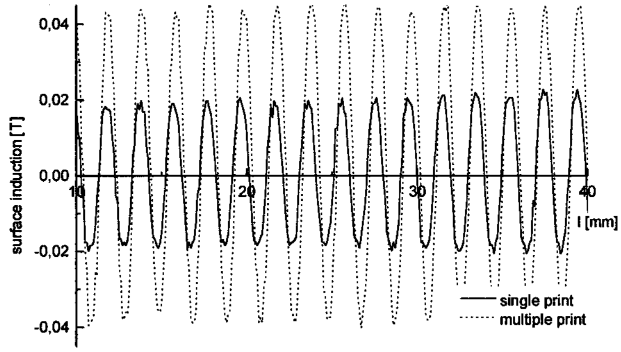

Figure 6 Induction across the surface of a printed MQP-B film magnetized with 1 mm pole pitch (measured 250 μm above the surface): single print with about 10 μm film thickness (solid line) and multiple print with about 50 μm film thickness (dotted line).

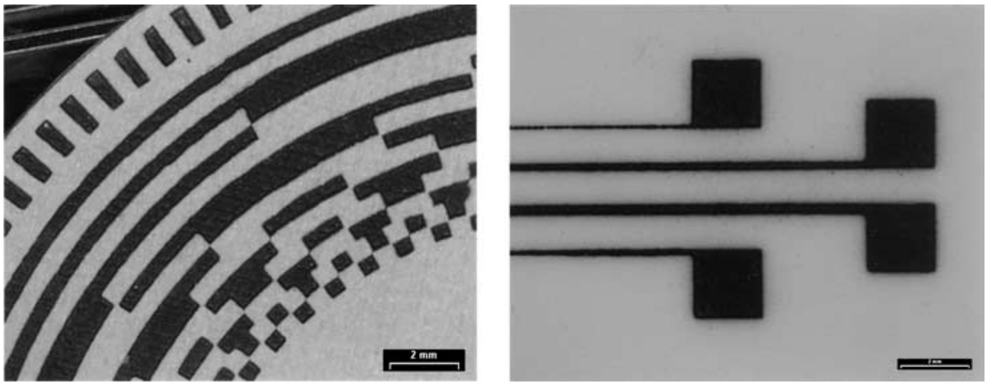

Figure 7 Micrographs of screen printed MQP-B films on an iron (left) and ceramic (right) substrates for sensor applications.

via screen printing. Inks of >80 wt% fine-milled MQP-B and -Q or >87 wt% MQP-S exhibit larger viscosity. These pastes are applicable for filling of vias or patterned structures. Two examples of ink viscosities are shown in Fig. 5.

For actuator applications the printed films can be magnetized with several magnetic poles at one magnetizing step. For such experiments MQP-B films on iron substrates were magnetized with a stripe pattern and 1 mm pole pitch. After multi-pole magnetization the induction on the surface of the films was measured and compared to data calculated with the software MAGFIELD [11] using BEM algorithms. A horizontal scan above the film surface is shown in Fig. 6. At the pole maximum an induction of 20–50 mT is observed. This agrees well with calculated data indicating that the printed films are completely magnetized during the impulse magnetization process.

The printed thick films represent alternative magnetic components for micro-systems: application in MEMS, e.g., in micro-motors [12, 13] or magnetic encoders [14] have been successfully tested. Fig. 7 shows micrographs of two examples of printed structures for sensor applications.

Acknowledgements
This work was supported by a grant (16SV439/8) from the Bundesministerium für Forschung und Bildung (BMBF), Germany. We thank M. Morauer and D. Schabbel for help in the preparation of inks.

References
1. S. YAMASHITA, J. YAMASHITA, M. IKEDA and N. IWABUCHI, *IEEE, Trans. Magn.* **7** (1992) 45.
2. H. LEMKE, G. THOMAS and D. L. MEDLIN, *Nanostruct. Mater*. **9** (1997) 371.
3. D. J. KEAVNEY, E. FULLERTON, J. PEARSON and S. BADER, *J. Appl. Phys*. **81** (1997) 4441.
4. T. S. CHIN, *J. Magn. Magn. Mater*. **209** (2000) 75.
5. O. CUGAT, in Proceedings of the 17. Symposium on Rare Earth Magnets and their Application, Newark, USA, August 2002, edited by G. C. Hagjipanayis and M. D. Bonder (Rinton Press, Princeton, 2002) p. 478.
6. J. J. WYSLOCKI, *J. Mater. Sci*. **27** (1992) 3777.
7. K. MAKIDA, O. YAMASHITA and A. NAKANISHI, *Jpn. J. Powder Metall*. **22** (1998) 365.
8. B. PAWLOWSKI, S. SCHWARZER, A. RAHMIG and J. TÖPFER, *J. Magn. Magn. Mater*. **265** (2003) 337.
9. Z. YUAN, A. J. WILLIAMS, T. C. SHIELDS, S. BLACKBURN, C. B. PONTON, J. S. ABELL and I. R. HARRIS, *ibid*. **247** (2002) 257.
10. Product catalogue, MAGNEQUENCH INTERNATIONAL, INC.
11. V. CHRISTOPH, HTW Dresden, software package "MAGFIELD".
12. E. KALLENBACH, H. KUBE, V. ZÖPPIG, K. FEINDT, R. HERMANN and F. BEYER, *Mechantronics* **9** (1999) 769.
13. H. KUBE, E. KALLENBACH, B. PAWLOWSKI and J. TÖPFER, in Proceedings of the 14th ASPE Annual Meeting, Monterey, CA, USA (1999) 579.
14. J. TÖPFER, in Proceedings of the 5th Symposium "Magnetoresistive Sensors: Basics, Preparation and Applications," 10./11.03.99, Wetzlar, Germany.

Received 20 January
and accepted 26 September 2003