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COMPOSITES THEORY AND PRACTICE

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SEGREGATION OF Nd-Fe-B POWDERS DURING INJECTION MOLDING OF GEARS WITH MAGNETIC PROPERTIES

The possibility of producing polystyrene-bonded micro-magnets with the use of two commercial nanocrystalline Nd-Fe-B powders (delivered by the Magnequench Co) was examined. The micro-magnets were formed by the injection method to obtain gear-shaped samples. The powders were produced by two different methods, namely the crushing of a rapidly cooled ribbon (MQP-16-7) or spraying (MQP-S). Depending on the production method, the powders differ in their particle shapes: the former method gave flaky particles whereas the latter - spherical particles. The particle shape has an essential effect upon the technological properties of the powder. The gear-shaped micro-magnets with a diameter of 2.6 mm were examined by X-ray tomography. An analysis of the 3D images has shown that during their injection with polystyrene, the powders undergo segregation. The largest particles of the MQP-S powder (spherical) are located at the greatest distance from the injection point which was positioned in the axis of the gear. They are also relatively numerous near the side surfaces of the gear. The filling ratio of this composite decreases as we pass along the radius of the gear. Taking into account the fact that the properties of the sprayed powders depend on their particle size, these observations will permit the of designing pieces with a gradient of magnetic properties. In the gears produced from the powder with flake-shaped particles, the flake surfaces tend to position themselves perpendicularly to the gear radius. The shape, therefore, of the powder particles determines the anisotropy of their distribution within the product. This observation can be utilized in the design of bonded magnets.

Keywords: **Nd-Fe-B magnets, polymer bonded magnets, hard magnetic composite**

SEGREGACJA PROSZKÓW Nd-Fe-B W PROCESIE WYTWARZANIA PRZEZ WTRYSKIWANIE MINIATUROWYCH KÓŁ ZĘBATYCH O WŁAŚCIWOŚCIACH MAGNETYCZNYCH

Zbadano możliwość wykorzystania dwóch handlowych proszków nanokrystalicznych Nd-Fe-B firmy Magnequench do wytwarzania mikromagnesów wiązanych polistyrenem. Jako metodę formowania wykorzystano wtryskiwanie. Proszki te były wytwarzane różnymi metodami: poprzez kruszenie szybko chłodzonej taśmy (MQP-B) i poprzez rozpylanie (MQP-S). Proszki te różniły się kształtem cząstek: pierwszy z nich posiadał cząstki płatkowe, a drugi kuliste. Ma to zasadniczy wpływ na właściwości technologiczne proszku. Mikromagnesy w postaci kół zębatych o średnicy 2,6 mm badano m.in. za pomocą tomografu rentgenowskiego. Analizując obrazy 3D, stwierdzono, że proszki w czasie wtryskiwania z polistyrenem ulegają segregacji. Największe cząstki proszku MQP-S (kulistego) są najbardziej oddalone od punktu wtryskiwania, który był w osi koła zębatego. Jest ich też stosunkowo więcej przy powierzchniach bocznych koła. Współczynnik napełniania takiego kompozytu zmniejsza się wzdłuż promienia koła zębatego. Biorąc pod uwagę, że właściwości proszków rozpylanych zależą od ich wielkości, poczynione obserwacje pozwalają na projektowanie wtryskiwanych kształtek o gradiencie właściwości magnetycznych. W kołach zębatych zawierających proszek płatkowy, poszczególne płatki mają tendencję do układania się powierzchnią prostopadle do promienia koła. Zatem kształt cząstki proszku determinuje anizotropię rozmieszczenia cząstek w wyrobie. Fakt ten może być również wykorzystany przy projektowaniu magnesów wiązanych.

Słowa kluczowe: **magnesy Nd-Fe-B, magnesy wiązane polimerem, kompozyty magnetyczne twarde**

INTRODUCTION

A great need for the miniaturization of micro-electromechanical devices has been observed for many years and has found its reflection in the great interest in micro-electromechanical systems (MEMS). This entails the demand for miniaturized sources of magnetic field, such as magnets or induction coils.

It follows from ref. [1] that with the decreasing dimensions of components able to generate a magnetic field, magnets are gaining superiority to induction coils. Fig. 1 shows the current density necessary to generate a magnetic field of 1T depending on the coil size. Since with the decreasing size of these devices the current density substantially increases, micro-magnets are more advantageous where miniature magnetic components are needed. Moreover micro-magnets have the obvious advantage that they can generate magnetic fields and forces without the need for external energy sources.

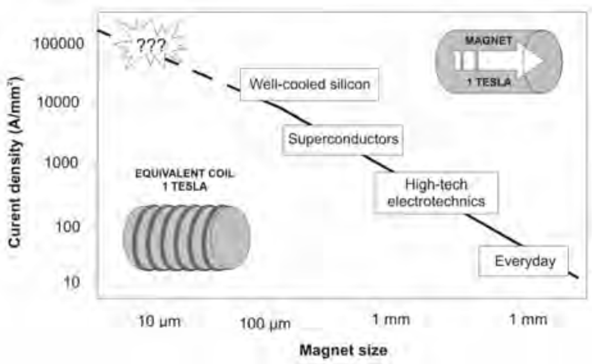

Fig. 1. Electric current density necessary to generate magnetic field of 1 T depending on coil dimensions, compared to that of permanent magnet of same size

Rys. 1. Gęstość prądu potrzebna do wytworzenia pola magnetycznego 1 T w zależności od wymiaru cewki indukcyjnej, w porównaniu do magnesu o tych samych wymiarach

The possibility of producing magnets of a desired size depends on the technology employed. The basic technology used for fabricating Nd-Fe-B magnets, which in view of their properties are the most interesting, is powder metallurgy. Nd-Fe-B magnets and ferrites are also produced in the form of so-called bonded magnets by joining magnetically hard magnetic powders with a plastic material. Powder metallurgy is suitable for fabricating larger products, about 1 mm in size, especially when Nd-Fe-B magnets are in question, since the surface oxidation which takes place during sintering degrades their properties, especially in magnets of small sizes.

There are also methods which permit producing magnetically hard materials in the form of 'thick' layers. Miniature magnets are most often fabricated by ion sputtering [2] and pulse laser deposition (PLD) [3]. There are publications which report on magnets with a thickness of 100 μm produced by these methods. These techniques have, however, a drawback in that they require a longer time to obtain such thick layers.

We can see that magnets of sizes within the range from, let us say, 100 to 1000 μm are too small to be fabricated by conventional powder metallurgy methods and too large to be suitable for layer deposition techniques. At the same time, however, the literature indicates that the constructors of electric micro-machines equipped with magnets are greatly interested in magnets sized just within this range.

A solution to this problem may be composite magnets bonded with a plastic binder, which can be produced by e.g. pressing a mixture of the two components, cutting from thin foils [4], or by injection. The great variety of commercially available magnetically hard powders permits the properties of the magnet to be tailored according to the current needs. The maximum filling ratio possible to achieve ranges from 50% to 65%, depending on the production method and the powder parameters.

In general, bonded magnets are less expensive than sintered magnets, since the sintering operation, with the necessity to use protective atmospheres or vacuum and with its high power consumption, substantially increases the production costs. These costs can probably be reduced even more thanks to the micrometric sizes possible to achieve in the fabrication of bonded magnets.

An interesting method of producing bonded micromagnets is injection [5]. An advantage of this method is the high precision of the products and the high effectiveness of the process. Thanks to these advantages, it is possible to fabricate magnets with complicated shapes, such as gears which, in an electric engine, can perform a double function, namely, as the source of magnetic field and, at the same time, a drive transmitting component.

It is a complex task to develop the technology of miniature magnets in the form of gears, since it requires studies on the technological process itself and also proper selection of the powders and methods of their preparation. The present study describes and discusses the results of examination of magnetic gears with a module pitch of 0.2 and diameters of 3.2 and 2.0 mm, produced by injection, in particular examination of whether and how the powder is segregated during the injection process. The examination methods employed permit the internal structure of the gears to be analyzed in three directions (3D analysis).

EXPERIMENTAL PROCEDURE

In the present experiments, magnetic gears were produced using powders delivered by the Magnequech Co, designated by the trade marks MQP-S and MQP-B (Fig. 2).

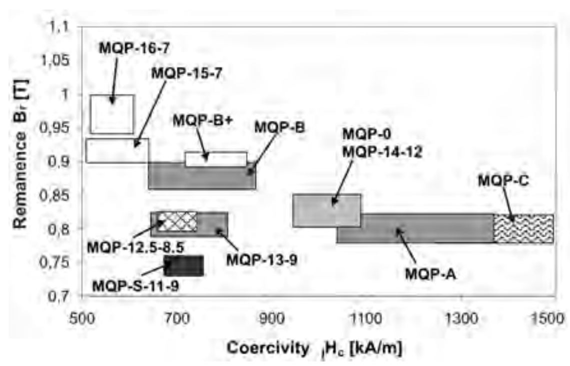

Fig. 2. Remanence and coercivity of high-coercivity powders produced by Magnetquench Co. Symbols given in the figure are trade marks of the powders (from: www.magnetquench.com)

Rys. 2. Remanencja i koercja wysokokoercyjnych proszków produkowanych przez Magnetquench Co. Symbole wykorzystane na wykresie są nazwami handlowymi (na podstawie www.magnetquench.com)

One powder had spherical particles (MQP-S) and the other 'flaky' particles (MQP-B). The particle size distributions in the powders determined by sieve analysis are shown in Figure 3, and their magnetic properties are given in Table 1. The matrix of the composites was polystyrene with high impact strength (SX-25), delivered by DWORY S.A. The powder granulates were prepared by introducing the Nd-Fe-B powder into a polystyrene solution in toluene. After the toluene evaporated, the material was subjected to mechanical granulation.

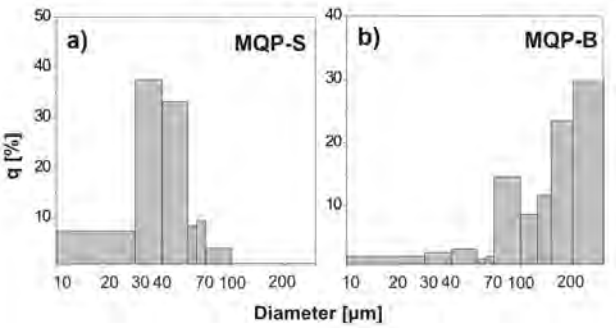

Fig. 3. Particle size distributions in MQP-S powder (a) and MQP-B powder (b)

Rys. 3. Rozkłady wielkości cząstek proszków MQP-S (a) i MQP-B (b)

The injection molding process was conducted in an injection molding micro-machine constructed at the IMiIB PW. Its mold is equipped with a thermostatic system which permits the of forming materials with high viscosity and high thermal conductivity.

The injection parameters were: the temperature of the molding mass T_m = 210°C, mold temperature T_f - 135°C and injection pressure p = 80 MPa. The material was introduced along the axis of the gear (central injection molding).

The magnetic properties of the samples were measured using a VSM magnetometer (Lake Shore). The morphology was analyzed in a redia Micro-XCT-400 X-ray micro-tomograph (accelerating voltage of X-ray tube 120 kV, power 10 W, acquisition time of single projection 8 s, total projections for scan 1600, total scanning time 4 h 20', magnification 10X, pixel size of single projection 2.44 μm).

RESULTS

The magnetic properties of the produced gears are given in Table 1. The proportion of the metallic particles was assumed to be 48%. Their real proportion was calculated based on the density of the composite. The densities of the Nd-Fe-B powders were given by their manufacturers, whereas the density of the polystyrene was determined experimentally (using a helium pycnometer) to be 1.0511 Mg/m^3. The densities of the powders and the composites together with the filling ratios are given in Table 2.

TABLE 1. **Magnetic properties of powders compared to properties of composites obtained during present experiments**

TABELA 1. **Właściwości magnetyczne użytych proszków i otrzymanych kompozytów**

POWDER	POWDER DENSITY [g/cm³]	COMPOSITE DENSITY [g/cm³]	FILLING RATIO [%]
MQP-S	7,43	4,0098	46,4
MQP-B	7,64	4,0730	45,9

TABLE 2. **Measured densities of composites and calculated values of filling ratios**

TABELA 2. **Zmierzone gęstości kompozytów i obliczone współczynniki napełnienia**

MAGNETIC PROPERTIES		POWDER	COMPOSITE
MQP-S	Coercivity [kA/m]	670-750	755
	Remanence [T]	0,73-0,76	0,334
	$(BH)_{max}$ [kJ/m³]	80-92	19,2
MQP-B	Coercivity [kA/m]	640-800	730
	Remanence [T]	0,86-0,895	0,361
	$(BH)_{max}$ [kJ/m³]	111-126	22,16

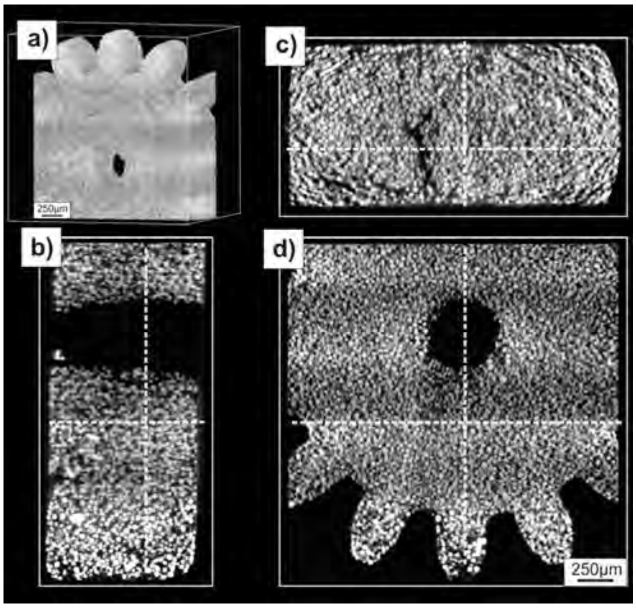

Fig. 4. Images (X-ray tomography) of miniature magnetic gear produced from MQP-S powder (a) and images of its cross-sections analyzed in the study (b, c, d)

Rys. 4. Widok (a) i analizowane przekroje (b, c, d) koła zębatego z proszku MQP-S

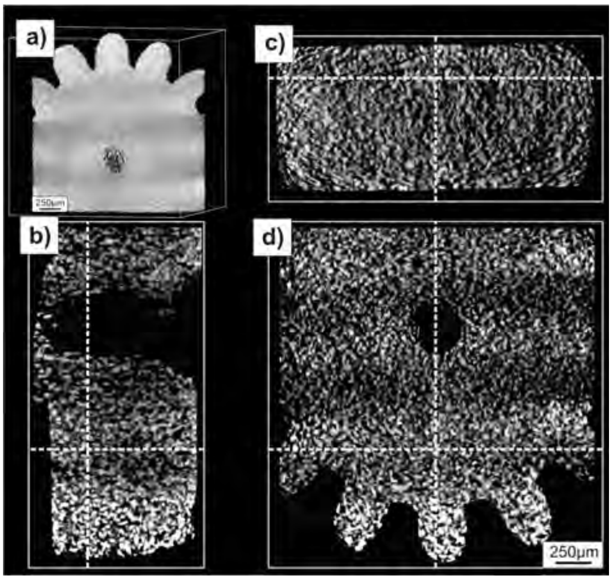

Fig. 5. Images (X-ray tomography) of miniature magnetic gear produced from MQP-B powder (a) and images of cross-sections of this gear (b, c, d)

Rys. 5. Widok (a) i analizowane przekroje (b, c, d) koła zębatego z proszku MQP-B

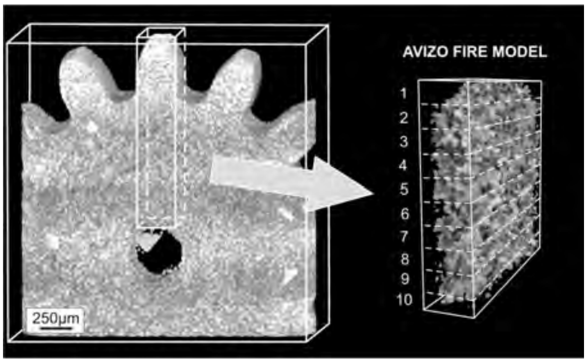

Fig. 6. Image of cross-section of gear showing places where it was cut out (numbers refer to sectors selected for calculating filling ratio and average particle size)

Rys. 6. Analizowane przekroje kół zębatych (numery odpowiadają sektorom analizowanym pod względem wsp. napełnia i średniej wielkości cząstek)

Taking into account the real values of the filling ratios and the properties of the powders, magnets obtained can be considered to be in agreement with those anticipated. The coercivity has practically not changed, whereas the remanence and correspondingly the energy $(BH)_{max}$ have decreased in proportion to the volumetric share of polystyrene.

The microstructure of the magnetic gears was examined by X-ray tomography. Figures 4 and 5 are images of fragments of the gears produced from the two powders: with spherical and 'flaky' particles. In both Figures 4 and 5, (a) denotes 3D images, whereas (b, c, and d) show cross-sections of the gears. Figure 6 is an image of a cross-section indicating where the cross-sections were cut out. In X-ray tomography, the image contrast depends on the density of the sample. In Figures 4 and 5 we can see the particles of the Nd-Fe-B powders, but we cannot see the polymer matrix. A preliminary analysis indicates that the distribution of the powders within the matrix is not uniform. Therefore we examined the variation of the filling ratio and the average particle size along the radius of the gear (Figs. 4d and 5d). Figure 6 shows a cubicoidal fragment of the gear volume cut out beginning from the central opening of the gear to the tooth tip. This gear fragment was divided into 10 sub-volumes, the filling ratio and the average particles size were determined in each of them. It should be noted that the filling ratio determined near the tooth tip is lowered misleadingly since this region also comprises some space beyond the tooth. Moreover, the filling ratios determined by X-ray tomography are smaller than those calculated from the density of the composites. This is so since the program neglects the particles positioned on the boundary of the analyzed sub-volume (the particles cut through with the plane that separates this sub-volume). Figure 7 shows the variation of the filling ratio along the gear radius in the gears produced with each of the two powders.

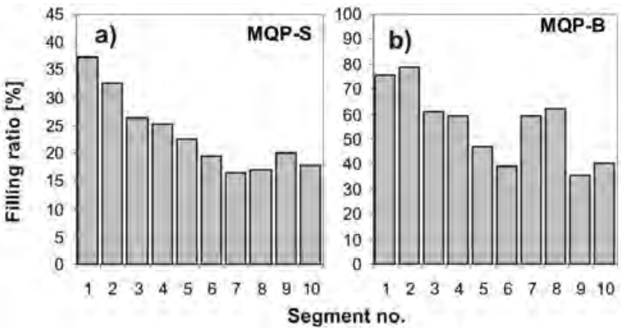

Fig. 7. Variation of filling ratio along gear radius in gear produced with (a) spherical powder, and (b) 'flaky' powder

Rys. 7. Zmiany współczynnika napełnienia wzdłuż promienia koła zębatego z proszkiem kulistym (a) i płatkowym (b)

Neglecting region 1 for the reasons described above, it can be seen that along the gear radius, the values of the filling ratio differ by more than 5%, and these differences are more marked in the powder with flaky particles. Since the measurement error is 1.8% with the spherical particles and 2.3% with the flaky particles, these differences are significant. Within the tooth region (regions 2 and 3), the filling ratio decreases in the powder with spherical particles and is unchanged in the powder with flaky particles, but in the latter powder its changes along the gear radius are even more visible. When observing the particle distribution in the plane perpendicular to the gear radius (Figs. 4b and 4b), we can suppose that during the injection when the powder was moving from the center of the gear along its radius, it underwent segregation.

The variations of the particle size along the gear radius are shown in Figures 8a and b. We can see that the particle size increases gradually along the gear radius as we pass towards the tooth tip in both the 'spherical' and 'flaky' powders, except in the latter powder in regions 7 and 8 where the particle size is increased (just as the filling ratio).

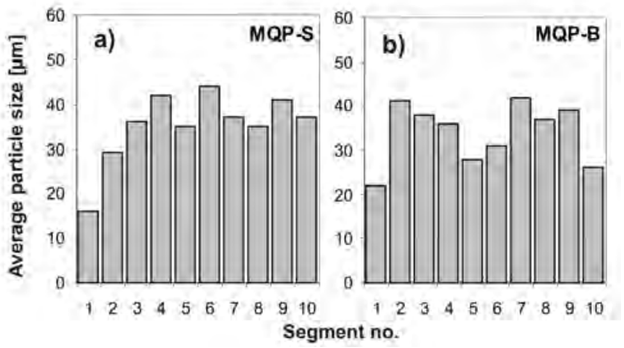

Fig. 8. Variation of average particle size along radius of gear produced with 'spherical' powder (a) and 'flaky' powder (b)

Rys. 8. Zmiany średniej wielkości cząstek proszku wzdłuż promienia koła zębatego z proszkiem kulistym (a) i płatkowym (b)

This difference in the behavior between the two materials is probably associated with the higher viscosity of the composite produced by the 'flaky' powder (its particles are much greater and their surfaces are strongly developed). A 3D image analysis of the composites with the 'flaky' powder shows that the flakes tend to arrange themselves so that the direction of propagation of the material being injected is perpendicular to their surfaces. This observation is in agreement with the results presented in [6].

CONCLUSIONS

Composite (Nd-Fe-B with a polystyrene matrix) gears with magnetic properties were produced with 2 magnetic powders differing in particle shape (spherical and flake-shaped). The distributions of the filling ratio and particle size within the samples appeared to be non-uniform (as examined by X-ray tomography). The average particle size examined along the gear radius increased with increasing distance from the point of injection, except in some regions of the composite produced with 'flaky' powder where the average particle size was greater. In both the composites, the filling ratio varied along the gear radius, with a well-marked decrease within the gear tooth region in the composite produced with the powder with spherical particles. The results obtained in the present study clearly show that the structure of the composite along the directions of material movement during the injection is inhomogeneous.

Acknowledgments

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