Hindawi Publishing Corporation
Advances in Materials Science and Engineering
Volume 2014, Article ID 651062, 8 pages
http://dx.doi.org/10.1155/2014/651062

Research Article

Material Characterization of Strontium Ferrite Powders for Producing Sintered Magnets by Ceramic Injection Molding (MagnetPIM)

Dietmar Drummer and Susanne Messingschlager

Institute of Polymer Technology (LKT), Friedrich-Alexander-University of Erlangen-Nuernberg, 91058 Erlangen, Germany

Correspondence should be addressed to Susanne Messingschlager; messingschlager@lkt.uni-erlangen.de

Received 7 May 2014; Revised 26 August 2014; Accepted 20 September 2014; Published 13 October 2014

Academic Editor: Jun Zhang

For this study, different strontium ferrite powders were mixed with a filling ratio of about 60 vol% in a binder system and formed into green compacts. During the process of injection molding, a magnetic field was generated in the tool via a magnetic coil, which enables magnetization and orientation of the ceramic particles. All powders were successfully processed by MagnetPIM. The investigations identified that it is impossible to extrapolate from the magnetic properties of a green compact to the magnetic properties of a sintered part. It became obvious, though, that, when producing very strong magnetic parts by MagnetPIM, the best results can be obtained by using powders with small particle sizes.

1. Introduction

The process of powder injection molding can be classified into ceramic injection molding and metal injection molding. Both benefit from the geometric freedom of the injection molding process, making it possible to produce complex, precise, and small ceramic or metal components in high volumes [1].

By using magnetically ceramic [2–7] or metal powders [8] in the process of powder injection molding sintered magnets can be produced. In [3], Lee and Jeung developed a new binder system for injection molding of strontium ferrite and Murillo et al. investigated in [2] the microstructures of strontium ferrite permanent magnets via SEM and metallographic micrographs and the magnetic properties. Zlatkov et al. analyzed in [4] the sintering process of isotropic PIM parts. The use of magnetically anisotropic strontium ferrite powder in the powder injection molding process represents an option to produce three-dimensional, multipolar sintered magnets that combine the benefits of the injection molding process to those of sintered magnets. When producing multipolar magnets, the number of assembly steps can be diminished. In some cases, assembly can be avoided or even included in the molding process. For the injection molding of highly filled powder compounds, low viscosity is required, and the melt must be shear-thinning.

Powders with bigger particle sizes are better suited for the production of plastic-bonded permanent magnets, due to their better dispersion in plastic matrices. This results in a homogeneous compound with good flow properties. As these flow properties are very important, Kim et al. investigated in [9] the magnetorheological property of magnetic PIM feedstock.

For the injection molding process under consideration, several powders were developed and optimized. For the development of sintered magnets with anisotropic strontium ferrite powders, the magnetic properties of "remanence" and "coercivity" are important. In order to achieve a high level of remanence, high density is required, and, in order to obtain high coercivity, small grain sizes are also necessary after sintering [10]. The impact of the heating rate during sintering is shown by Zlatkov et al. in [4].

In order to achieve small grain sizes, small powder particle sizes are needed. However, processing of these

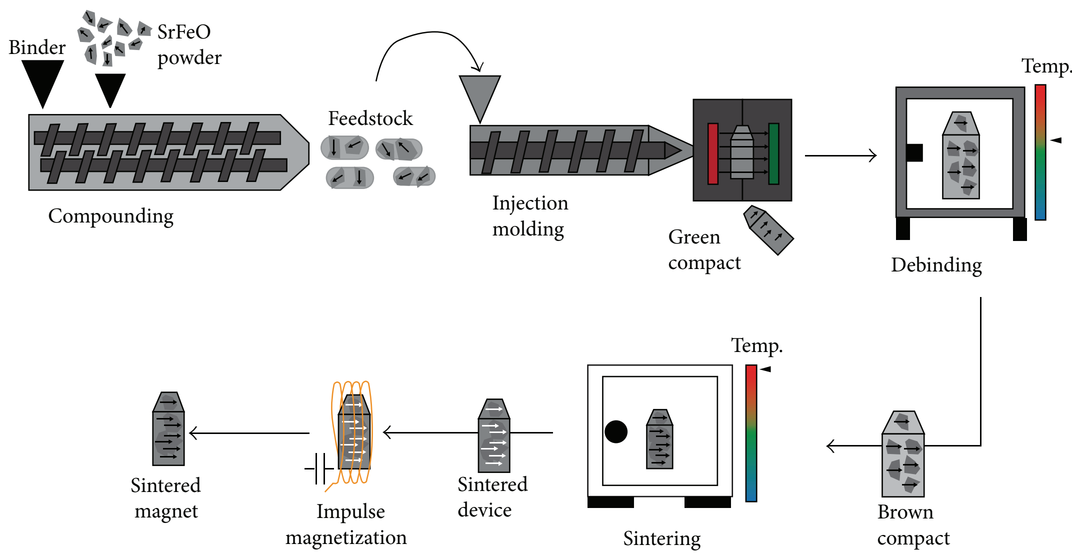


Figure 1: Process chain of ceramic injection molding to build a magnetic device (MagnetPIM).

powders is very challenging. The aim of this study is to characterize powders with different particle size distributions for MagnetPIM application.

2. Basics

The process chain of MagnetPIM starts with mixing a ceramic powder and a binder to form a feedstock (filling degree: approximately 60 vol%). The powder is not magnetized at this stage of the process. By using a powder with anisotropic magnetic properties and a mold with a magnetic field applied, the particles can be magnetized and oriented during injection. Orientation of the field can also be applied in three dimensions, allowing components with complex multipolar structures to be produced. Before sintering, the binder has to be removed. This can be accomplished by solvent debinding, thermal debinding, or a combination of the two. Then, the powder is sintered. By using impulse magnetization, the device can be magnetized (Figure 1).

Temperature is very important for the magnetic properties resulting. As temperature increases, the intrinsic coercivity decreases. It even results in a sharp decline at the beginning of the exaggerated grain growth [3].

3. Materials and Methods

3.1. Materials. For these investigations, strontium ferrite powders with different particle sizes were selected (type: S6, S9T, S14T, and S60T; manufacturer: Tridelta and type: CSP and SK-06; manufacturer: Tokyo Ferrite MFG. Co., Ltd.). The particle sizes, as indicated by the manufacturers, are summarized in Table 1.

The powders were mixed with a binder (type: Licomont EK583G; manufacturer: eMBe Products & Service GmbH), which can be extracted in a two-step process. The parts were first immersed in a water bath and then thermally processed in a furnace. The filling degree was 60 vol%.

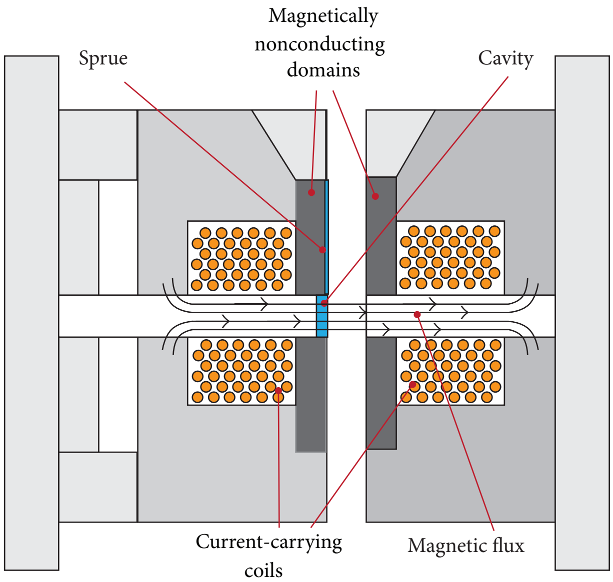
Table 1: Powder types and their particle sizes.

Powder type	Particle size
S6	3,0 μm (Fisher)
S14T	1,25 μm (Fisher)
Sk-06	3,0 μm (average)
S9T	2,6 μm (Fisher)
S60T	1,35 μm (Fisher)
CSP	1,06 μm (average)

3.2. Specimens. In order to detect the influencing factors acting upon the various magnetizable strontium ferrite powders in the process of MagnetPIM, a simple geometry of 20 mm × 20 mm × 2 mm with a bipolar magnetization was chosen.

3.3. Processing. For compounding of the feedstock, a twin-screw extruder was used at a working temperature of 140°C (type: ZSE 27 HP 40 D; manufacturer: Leistritz AG). Injection molding was performed on an injection molding machine (model: K110-2F; manufacturer: Ferromatik Milacron GmbH) at 160°C melt temperature, 55°C mold temperature, and 20 mm/s injection speed. More processing parameters are shown in Figure 2. An electric coil in the tool induces a magnetic field, which is applied to the cavity for the purpose of magnetizing and orienting the magnetic powder in the feedstock. The directional magnetic field was varied by the energy in the coils inside the mold. For the investigations, magnetic fields of 0, 100, 200, 400, 800, and 1600 mT were applied.

The binder was removed from the green compacts in two steps. First, the water-soluble binder component was



(a)

Material	Change over pressure (bar)	Holding pressure (bar)	Time for holding pressure (s)
S60T	900	300	1
S6	320	50	1
S9T	580	200	1
S14T	460	150	1
SK-06	560	100	1
CSP	620	100	1

(b)

Figure 2: (a) Mold (including illustration of magnetic field), (b) process parameters applied.

dissolved in a water bath at room temperature. Then, the other components evaporated in a furnace at temperatures up to 300°C. Sintering was carried out at temperatures of 1200°C, 1225°C, and 1250°C for a period of 6 minutes.

The magnetic properties (remanence and intrinsic coercivity) after sintering and impulse magnetization were characterized by using a permagraph (type: C-300; manufacturer: Magnet-Physik Dr. Steingroever GmbH). The size distribution was measured by the laser diffraction method and visualized via scanning electron microscope (SEM).

4. Results and Discussion

4.1. Magnetic Properties. It is essential for the process of MagnetPIM that the powder particles can be magnetized and oriented during the process of injection molding. Indicators of particle orientation are the magnetic properties of the green compacts that are due to the increase when stepping up the directional magnetic field in the mold, until the saturation limit is reached (at approximately 800 mT). Above 800 mT, there is no significant increase (Figure 3). While the differences in remanence are in the same order of magnitude with all green compacts, that is, in the range of 150 and 200 mT, when using a 1600 mT field strength, the intrinsic coercivity differs significantly between 150 and 400 kA/m, while S14T reaches the highest values and CSP and SK-06 reach the lowest values.

In order to achieve the desired results, the green compacts were debindered and sintered for six minutes at temperatures of 1200°C, 1225°C, and 1250°C. Figure 4 shows the results of the magnetic properties of the green compacts and the sintered devices, without the devices made of SK-06. Sintering of these parts resulted in the distortion of the specimen, making it impossible to measure them. It can be shown that remanence of the specimen is always higher after sintering of the green compacts. This increase in remanence can be explained by the fact that, after debinding and sintering, the polymer matrix is completely evaporated and can no longer reduce the magnetic properties of the devices. Additionally, while sintering ceramic parts, their densities increase, which leads to higher remanence of the part. While applying higher temperature in this processing step, the grain growth continues, and the density, as well as the remanence, increases. This can be shown for S60T (1200°C to 1225°C), S6, and CSP. The remanence of S9T and S14T decreases when applying higher sintering temperature. This effect is caused by the intrinsic coercivity of the material. Intrinsic coercivity of the sintered devices is lower than that of the green parts, and, additionally, at sintering temperature up to 1250°C, the materials lose their intrinsic coercivity until it is below 50 kA/m. Microscopic analyses reveal an exaggerated crystal growth. This implies that the selected temperature was too high for these powders. The intrinsic coercivity of S6 and CSP increases from green compacts to sintered parts because the polymer matrix is completely removed. While high intrinsic coercivity of the final parts requires small grain sizes, it decreases while sintering at higher temperature. The behavior of S60 can be classified between the described material types. The intrinsic coercivity is lower for sintered parts and decreases with higher temperature. Furthermore, the remanence at a sintering temperature of 1250°C is lower than that at 1225°C, but the behavior is not as pronounced as it was for S9 and S14T.

4.2. Particle Sizes. Figure 5 shows that the investigated strontium ferrite particles build very strong and big agglomerates

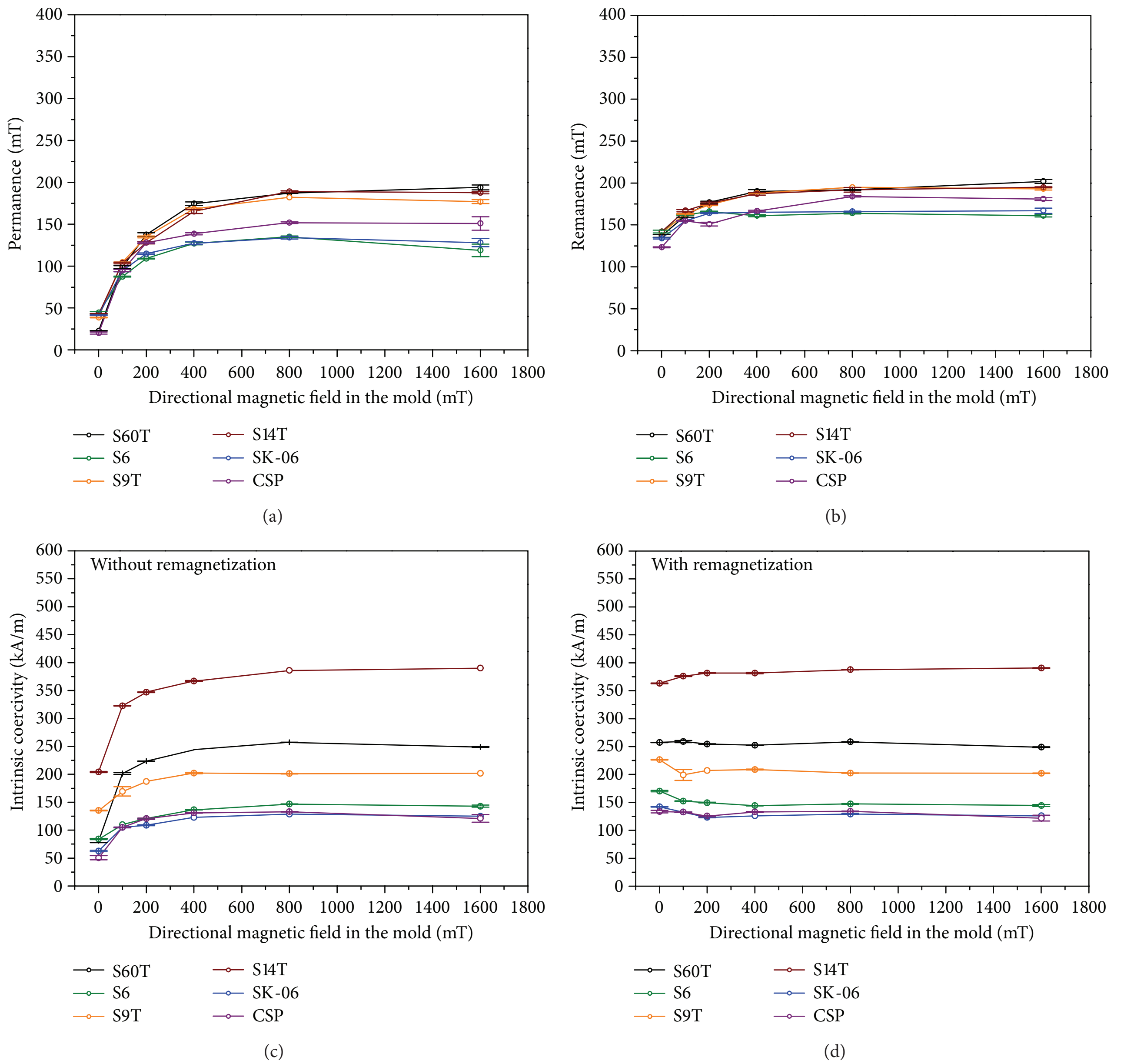


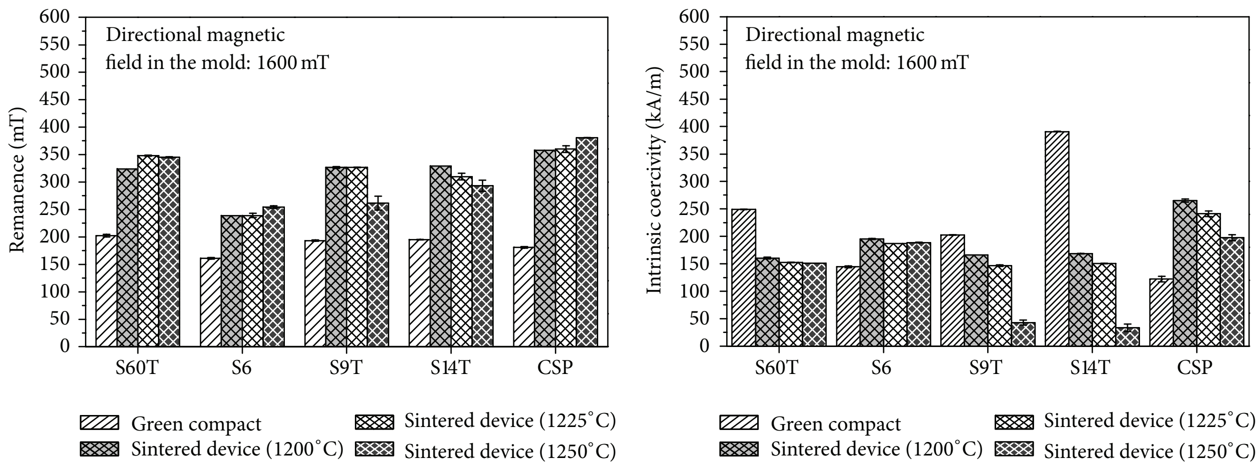
Figure 3: Magnetic properties of green compacts with different SrFeO powders, as a function of the directional magnetic field in the mold. (a) Permanence, (b) remanence, (c) intrinsic coercivity (without remagnetization), and (d) intrinsic coercivity (with remagnetization).

that could not get cracked completely by standard measuring via laser diffraction method. The powders CSP and S60T show smaller agglomerates than the other powders.

Because of this result, the microscopy images show the strontium ferrite powders in two different magnifying steps to get an impression of the size distribution. In the images on the left side, the agglomerates can be seen, but it is impossible to quantify them. This is due to the method of preparation for the SEM. The finest powder is CSP. S9T and S14T appear to have very uniform particle shapes. S6, S60T, and Sk-06 powders combine large particles to finely fractured particles (Figure 6).

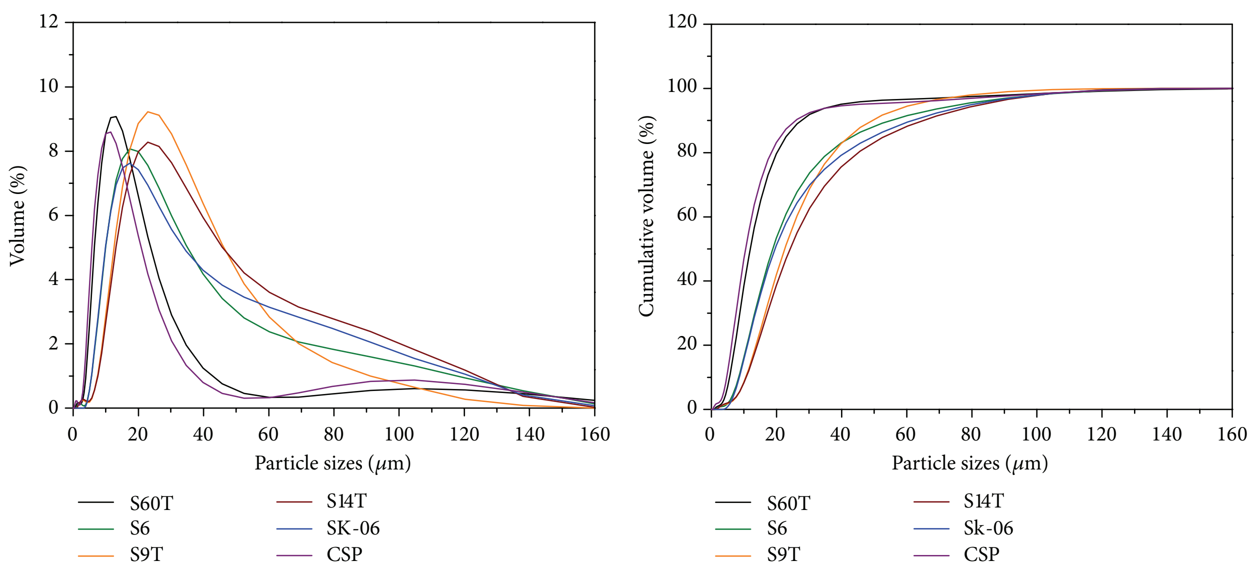
With SEM-microscopy, particle sizes can be measured only in 2D or 2.5D. Measuring is impossible if the particles are rather anisotropic, for example, flakes, or isotropic and spherical. During the injection molding process, the particles were magnetized and oriented. If the geometry of the particles is anisotropic and the particles are oriented, the shrinkage in width and thickness of the part is also anisotropic (Figure 7).

Figure 8 shows the results of anisotropic shrinkage in thickness and width for S60T, S9T, S14T, and CSP. The parts made of material S6 show almost isotropic shrinkage. It can be assumed that the particle geometry of this powder is nearly isotropic.



(a) (b)

Figure 4: Magnetic properties of green compacts and sintered devices.



(a) (b)

Figure 5: Size distribution of strontium ferrite powders.

The shrinkage resulting from the different sintering temperatures can be seen in Figure 9. S6 shows a sharp increase in shrinkage as the sintering temperature is increased.

5. Conclusions

The investigations showed that all SrFeO powders can be processed by MagnetPIM. The magnetic properties of green compacts vary significantly depending on the powder applied but cannot be used as an indicator of good magnetic properties after sintering. The S14T material has very good magnetic properties as a green compact. However, after sintering, it loses its good magnetic properties. The CSP material has very small particle size and poor magnetic properties as a green compact. After sintering, though, remanence and intrinsic coercivity increase, thus resulting in the best properties of all the specimens investigated. The particle orientation during molding and sintering can be inferred from the isotropic or anisotropic shrinkage, depending on whether the powder is geometrically anisotropic (e.g., platelet-shaped) or isotropic (spherical). Anisotropic geometry results in anisotropic shrinkage. This study shows that the production

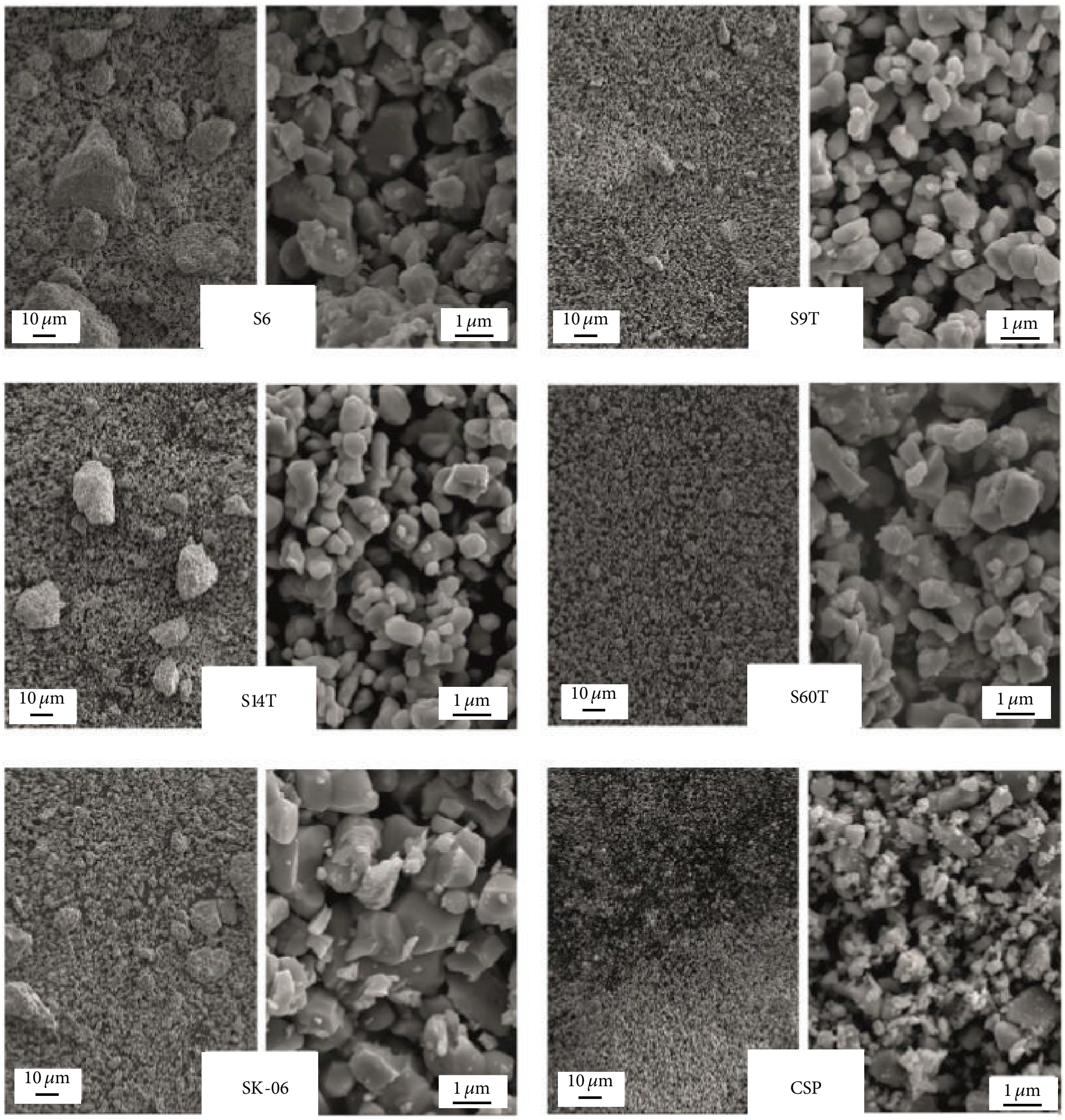


FIGURE 6: SEM-microscopy images of strontium ferrite powders (different magnifications).

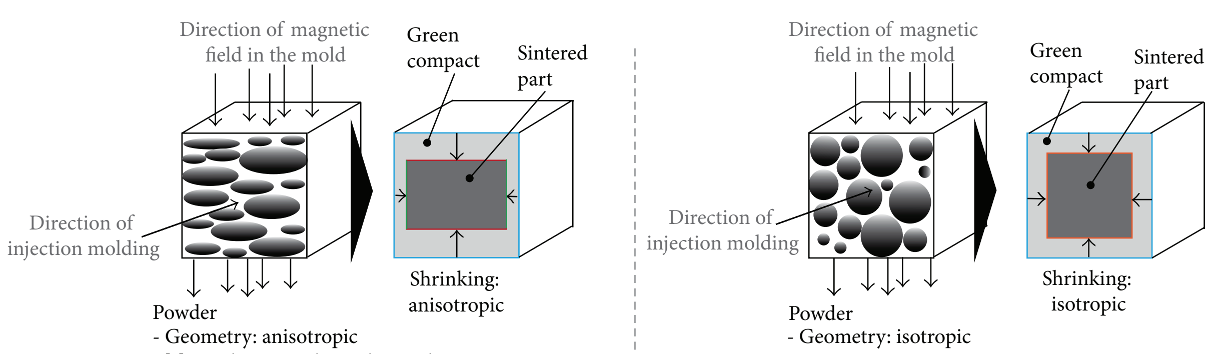


FIGURE 7: Isotropic and anisotropic shrinkage as a function of particle geometry.

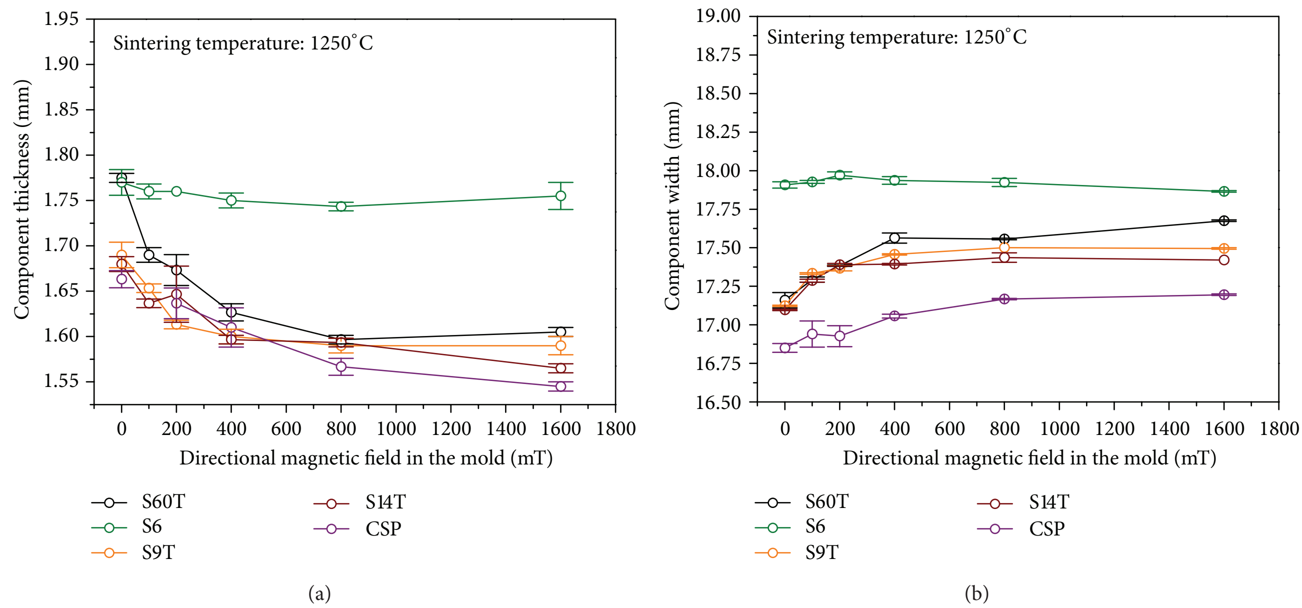


FIGURE 8: Sizes of sintered devices as a function of the directional magnetic field in the mold.

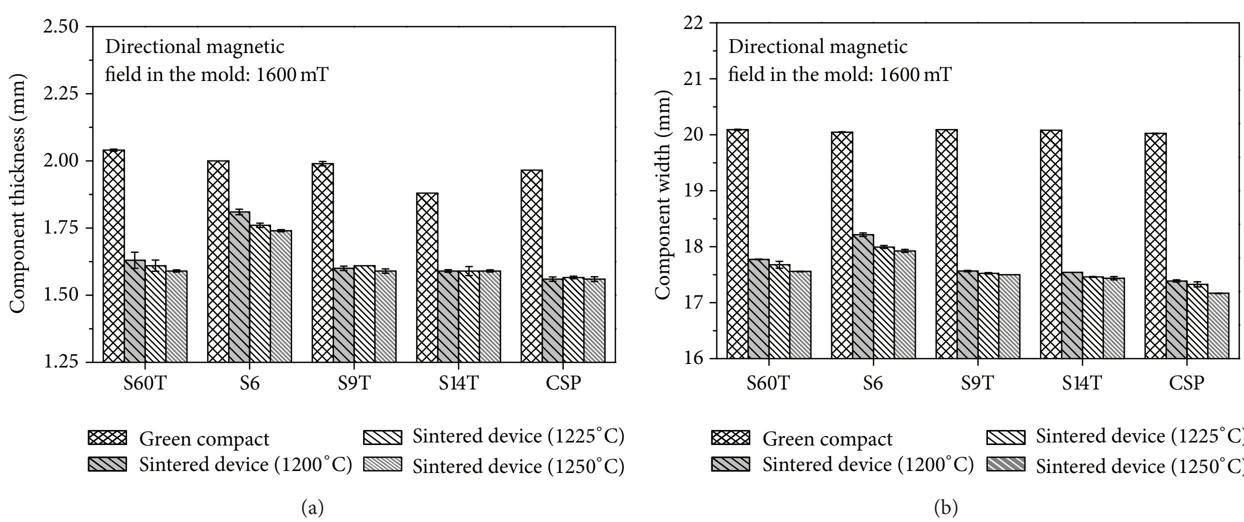


FIGURE 9: Shrinkage of sintered devices resulting from different sintering temperatures and powders.

of sintered magnets in the powder injection molding process is possible and yields promising results.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgments

The authors thank Mr. Wanner for the support and Dowa Holdings Co., Ltd., for providing the materials CSP and SK-06 and the Institute of Particle Technology (Friedrich-Alexander-Universität Erlangen-Nürnberg) for the measurement of the size distribution with the laser scanning method. Furthermore, they acknowledge the support by Deutsche Forschungsgemeinschaft and Friedrich-Alexander-Universität of Erlangen-Nürnberg within the funding programme Open Access Publishing.

References

[1] R. M. German and A. Bose, *Injection Molding of Metals and Ceramics*, Metal Powder Industries Federation, 1997.

[2] N. Murillo, J. González, C. Guraya, M. Gutiérrez, and F. J. Seco, "Structural and magnetic properties of sintered Sr-ferrites

fabricated by powder injection molding," *Journal of Magnetism and Magnetic Materials*, vol. 203, no. 1–3, pp. 165–168, 1999.

[3] S. H. Lee and W. Y. Jeung, "Anisotropic injection molding of strontium ferrite powder using a PP/PEG binder system," *Journal of Magnetism and Magnetic Materials*, vol. 226–230, pp. 1400–1402, 2001.

[4] B. S. Zlatkov, M. V. Nikolic, V. Zeljkovic, N. Obradovic, V. B. Pavlović, and O. Aleksić, "Analysis and modeling of sintering of Sr-hexaferrite produced by PIM technology," *Science of Sintering*, vol. 43, no. 1, pp. 9–20, 2011.

[5] D. Drummer and S. Messingschlager, "Sintermagnete aus einem Guss," in *Kunststoffe*, vol. 3, 2014.

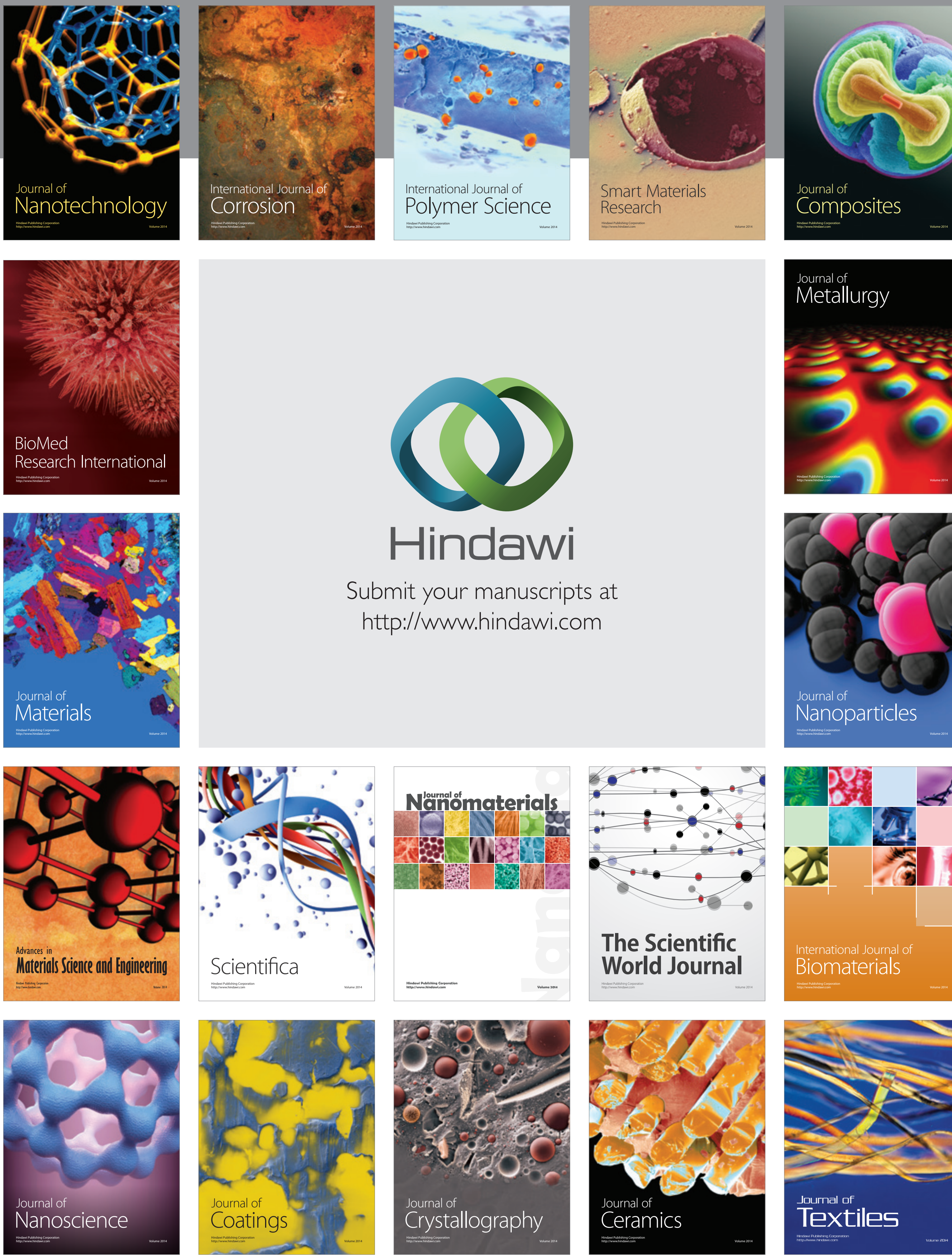
[6] B. S. Zlatkov, "The processing of advanced magnetic components by PIM: current status and future opportunities," *Powder Injection Moulding International*, vol. 3, no. 3, p. 41, 2009.

[7] "PIM successfully used for produce hard ferrite magnets," *PIM International*, vol. 4, 2010.

[8] S. H. Lee, W. Lee, W. Y. Jeung, and T. J. Moon, "Properties of anisotropic Nd(Fe,Co)B type sintered magnets produced by powder injection moulding," *Powder Metallurgy*, vol. 41, no. 3, pp. 185–188, 1998.

[9] S. H. Kim, S. J. Kim, S. J. Park, J. H. Mun, T. G. Kang, and J. M. Park, "Rheological behavior of magnetic powder mixtures for magnetic PIM," *Korea Australia Rheology Journal*, vol. 24, no. 2, pp. 121–127, 2012.

[10] F. Kools, "Reaction-induced grain growth impediment during sintering of strontium hexaferrite with silica addition," *Solid State Ionics*, vol. 16, pp. 251–259, 1985.


Journal of Nanotechnology
International Journal of Corrosion
International Journal of Polymer Science
Smart Materials Research
Journal of Composites
BioMed Research International
Hindawi
Submit your manuscripts at
http://www.hindawi.com
Journal of Metallurgy
Journal of Materials
Journal of Nanoparticles
Advances in Materials Science and Engineering
Scientifica
Journal of Nanomaterials
The Scientific World Journal
International Journal of Biomaterials
Journal of Nanoscience
Journal of Coatings
Journal of Crystallography
Journal of Ceramics
Journal of Textiles