Determination of the crystal alignment of $SrFe_{12}O_{19}$ permanent magnets using the (006) X-ray pole figure.

Gugliotti, C. F.[1*]; Lima, N. B.[1]; Oliveira, R. R.[1]; Silva, M. R. M.[1]; Silva, S. C.[1]

[1] Energy and Nuclear Research Institute, Av. Lineu Prestes 2242 – CEP: 05508-000 São Paulo – Brazil

* e-mail: caroline.gugliotti@usp.br

Resumo

O grau de alinhamento de ímãs permanentes de ferrita de estrôncio produzidos com diferentes tamanhos médios de partícula foi investigado utilizando análise de figura de pólo. Os ímãs foram produzidos a partir de pós sem moagem e moídos em moinho de bolas com tempos de moagem entre 60 e 360 minutos. O tamanho médio de partícula foi determinado utilizando Fisher Sub-sieve sizer e granulômetro CILAS 1064. A morfologia dos pós foram analisados utilizando imagens de MEV. Os pós foram alinhados a um campo magnético, prensados isostaticamente e sinterizados. Os ímãs foram caracterizados magneticamente utilizando um permeâmetro. O grau de alinhamento dos ímãs sinterizados foi calculado utilizando a reflexão (006) e a morfologia dos grãos do ímã foi analisada utilizando imagens de MEV. Os ímãs permanentes de ferrita de estrôncio produzidos com pós moídos apresentaram maior grau de alinhamento (0,82) quando comparados ao ímã produzido com pós não moídos (0,79).

Abstract

The crystallographic alignment of strontium ferrite permanent magnets produced with different average particle size powders was investigated by X-ray pole figure analysis. The magnets were produced with non-milled powders and powders ball-milled between 60 and 360 minutes. The average particle size was determined using Fisher Subsieve Sizer and CILAS 1064 granulometer. The morphology of the powders was analyzed using SEM images. The powders were aligned to a magnetic field, isostatically pressed and sintered. The magnets were magnetically characterized using a permeameter. The degree of alignment of the sintered magnets was calculated using the (006) reflection and morphology of the sintered magnet was analyzed using SEM images. The strontium ferrite permanent magnets produced with milled powders presented a higher degree of alignment (0.82) when compared to the magnet produced with non-milled powder (0.79).

Keywords: strontium ferrite, x-ray pole figure, magnet.

1. Introduction

Strontium ferrite, $SrFe_{12}O_{19}$, is a ferrimagnetic oxide used worldwide as a permanent magnet in automotive sector due to its features such as low cost, chemical stability, corrosion resistance and heat resistance. This material was introduced in 1950 and remains technically important and widely used on applications where large demagnetizing fields are required [1-3].

A crucial issue to obtain strontium ferrite permanent magnet with higher magnetic properties is to produce a dense and anisotropic microstructure and simultaneously grain size smaller than 1 µm.

The remanence is calculated using the following formula:

$$Br = \langle \cos\theta \rangle \, f \, P \, \mu_0 \, M_s \quad (1)$$

Where $\langle \cos\theta \rangle$ represents the degree of alignment, f represents the fraction of magnetic grains present in the magnet, P represents the ratio between the actual density and theoretical density of the magnet, μ_0 is the vacuum permeability (1.257×10^{-6} $H.m^{-1}$) and M_s represents the saturation magnetization [4].

In this study the crystal alignment of $SrFe_{12}O_{19}$ permanent magnets produced with powders milled during different milling time in function of the particle size was investigated using the (006) X-ray pole figure (XRPF) and X-ray diffraction pattern (XRDP).

2. Materials and methods

A commercial $SrFe_{12}O_{19}$ powder was used in this study. The powder was milled in a planetary ball mill Pulverissete Fritsch during 60 and 360 minutes using deionized water as milling medium. The resultant powders were dried during 24 hours. The average particle size of the dried powder was measured using Fisher Sub-Sieve sizer and a granulometer CILAS 1024. Morphology was studied using SEM images carried out in Philips XL 30 microscope.

After this process, the powders were aligned to 6 T magnetic field, isostatically pressed (200 MPa) and sintered in a tubular furnace in air at 1220ºC during 25 minutes. Magnetic measurements of the sintered magnets were performed in a permeameter LDJ BH-5000 after saturation in a pulsed field of 6 T.

The degree of crystallographic alignment (<cosΘ>) of sintered strontium ferrite permanent magnet was calculated by X-ray pole figures using (006) reflection and by the X-ray diffracted intensities of strontium ferrite hexagonal phase.

Measurements of (006) pole figure were carried out by the Schulz's reflection method with RINT 2000 diffractometer from Rigaku using CrKα radiation [5] The tilt angle (α) was varied from 0º to 75º in steps of 5º. The rotation angle, azimuth angle (β), was varied from 0º to 360º in steps of 5º. The crystal alignment (<cosθ>) was calculated using the following formula:

$$< cos\theta > = \frac{\int_0^{\pi/2} \exp\left(-\frac{\alpha^2}{2\sigma^2}\right) sin\alpha \; cos\alpha \; d\alpha}{\int_0^{\pi/2} \exp\left(-\frac{\alpha^2}{2\sigma^2}\right) sin\alpha \; d\alpha} \qquad (2)$$

Additionally, grain size of the sintered magnets was studied using SEM images.

3. Results and discussion

The average particle size (APS) from powder as received and ball-milled between 60 and 360 minutes are shown on figure 1 with granulometer measurements. Powders milled during 360 minutes presented average particle size equal to 1.3 µm according to the Fisher subsieve sizer technique and 11.04 µm according to granulometer measurements.

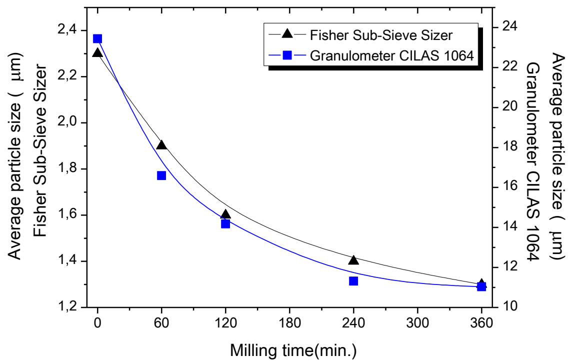

Figure 1: Average particle size of the powders in function of different ball milling times.

SEM images of the powders are shown on figure 2. It can be seen that even with 360 minutes of milling there are particles with nearly the same size as the non-milled powder.

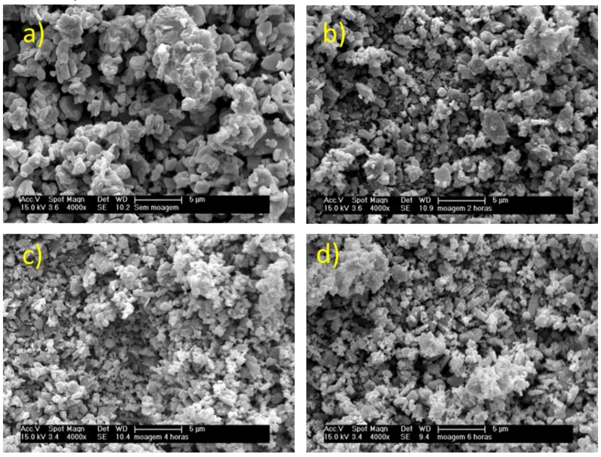

Figure 2: SEM images of the powders a) non-milled, b) milled during 60 minutes, c) milled during 120 minutes, d) milled during 240 minutes and e) milled during 360 minutes.

In the micrographs is observed that particles have a flake shape and agglomerates. In addition, heterogeneity powders was noticed with all milling time. These results are related with the Fisher Sub-Sieve Sizer measurement, showing that particles tend to agglomerate in liquid media.

Demagnetization curves, presented on figure 3, show that the sample produced with non-milled powder presents the highest magnetic properties. According to the SEM images (fig. 2) the reason of low magnetic properties values is, most probably, due to the presence of hard agglomerates. These agglomerates interfere on the quality of green bodies at particle alignment step before pressing in a way that particles cannot be well aligned once that agglomerates present many particles with different orientations and sizes.

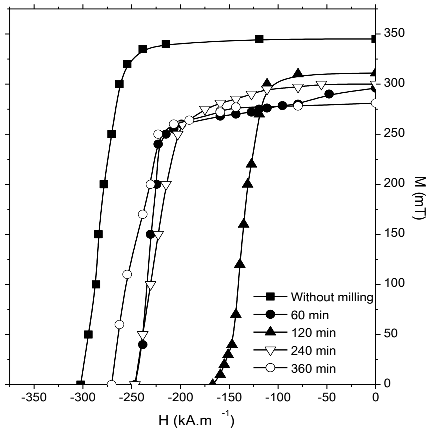

Figure 3: Demagnetization curves of permanent strontium ferrite magnets.

Normalized intensities for the (006) reflections in function of the α angle for the sintered magnets produced with non-milled powder and powders milled with different milling time are shown in figure 4. Figure 5 presents 2D and 3D representations of (006) pole figure from strontium ferrite permanent magnet produced with 120 minutes milled powder. The calculated crystallographic alignment was 0.82.

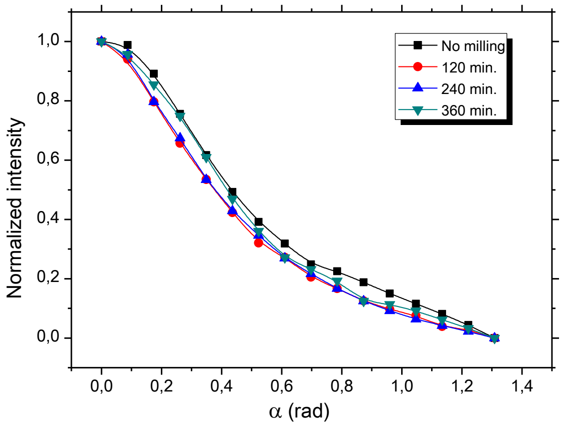

Figure 4: Normalized intensities in function of α angle for the (006) reflections of permanent magnets sintered using different average particle size strontium ferrite powders.

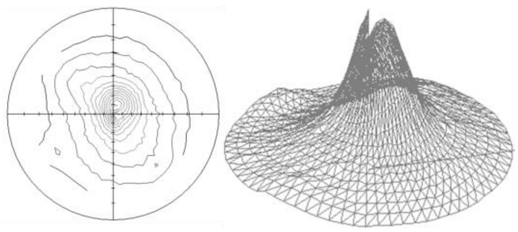

Figure 5: Representation of (006) pole figure and tridimensional representation from crystallographic plane from strontium ferrite magnet produced with non-milled powders.

Figure 6 presents the crystallographic alignment calculated using XRDP from all samples. The calculated crystallographic alignment with this methodology was 0.84.

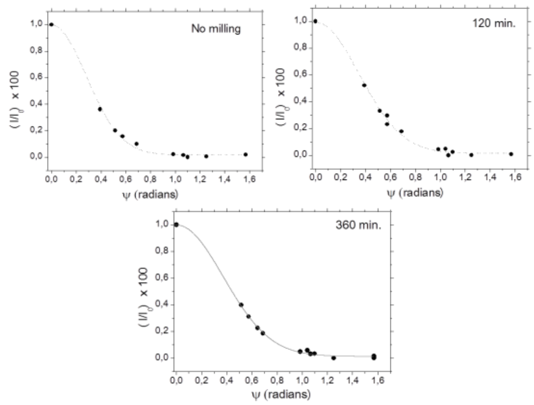

Figure 6: Ratio of the experimental normalized intensity of an oriented sample to the relative intensity of a sample with no texture as function of the angle *ψ*.

SEM micrographs from strontium ferrite permanent magnets produced with powders ball-milled during 120 and 360 minutes are shown on figure 7. It is observed that a pulsed 6 T magnetic field was not efficient to align all strontium ferrite powders due to the inhomogeneity of the milling step and agglomeration of the powders.

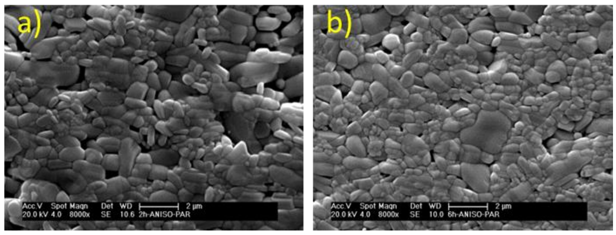

Figure 7: SEM micrographs of permanent strontium ferrite magnets sintered with powders milled in a planetary ball mill: a) during 120 minutes and b) during 360 minutes.

4. Conclusions

The magnets produced in this work using milled powders presented crystallographic alignment equal to 0.82.

The best magnetic properties were found in the strontium ferrite magnet prepared with non-milled powders, this is most probably due the homogeneous particle size and free agglomerate powder.

5. Acknowledgements

The authors are thankful to CNPq and IPEN-CNEN for their funding of this investigation and for providing a financial grant and to R. M. Leal Neto for laboratory experiments - grant #2007/55119-1 and #2001/03961-4, São Paulo Research Foundation (FAPESP).

6. References

[1] COCHARDT, A. J. Appl. Phys., v. 37, p. 1112-1115, 1966.

[2] KOOLS, F. *et al.* J. of Magnetism and Magnetic Materials, v. 242-245, p. 1270-1276, 2002.

[3] PULLAR, R. C. Progress in materials Science, v. 57, p. 1191-1334, 2012.

[4] CULLITY, J. R., RETHWISCH, D. G. Introduction to magnetic materials. IEEE Press, 2009.

[5] HERBST, J. F., TRACY, J. C. J. Appl. Phys., v. 50 (6), p. 4283, 1979.