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Nd-Fe-B MELT - SPUN POWDER QUALITY ESTIMATION BASED ON STONER - WOHLFARTH MODEL

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Abstract. *The squareness factor γ, defined as the ratio ${}_BH_c/{}_iH_c$, plays very importance role. This factor reflects the magnetization process under a reversed magnetic field in magnet. So the factor γ can be used as the critical parameter for estimating the magnet quality. For the powder compaction of 6.48 g/cm^3 mass density, if γ is less than 0.48 then the preparation conditions were far from the optimal ones. The value higher than 0.48 of factor γ proves that in prepared powder the coupling between grains is enhanced. From the theory and the experiments it was proved that for full dense compaction of NdFeB Stoner-Wohlfarth particles the squareness factor γ and the maximum energy product $(BH)_{max}$ can not be lager than 0.53 and 10.7 MG. Oe, respectively.*

I. INTRODUCTION

The model of uniform rotation of magnetization, developed by Stoner and Wohlfarth [1], is the simplest classical model describing magnetization reversal and used for the case if the energy minimum of systems is caused only by the balance between the anisotropy and the magnetostatic energy terms.

The high-performance NdFeB bonded magnets based on the high-quality rapid quenched powders are developed very intensively during last decade, the growth rate reaches to 20% annually [2] and this tendency is reserved for the near future. These powders usually are produced by optimizing conditions of the melt-spun technology. The high-quality melt-spun powder grains consist of the homogenously dispersed micrograins, the sizes of which are larger than superparamagnetic limit and slightly smaller than the single-domain size. For NdFeB material these two limits equal to 2 and 100nm, respectively, hence the optimal size of micrograins inside powder obtained by the melt-spinning and the appropriate annealing processes is in the range 50 – 80 nm. To enhance the coercivity, these micrograins are separated themselves by non-magnetic phases. For this micrograin assembly the Stoner – Wohlfarth model seems to be suitable to describing its magnetic properties.

In technological sense, it is important to have a tool to evaluate quickly the powder quality. In the usual way the powder quality is estimated by measuring a set of magnetic characteristics of the magnet sample made from the powder, likely the magnet remanence B_r, the magnetization coercivity ${}_iH_c$, the induction coercivity ${}_BH_c$, the maximum energy product $(BH)_{max}$. The most of these parameters are dependent strongly on the magnet granular structure, particularly the magnet mass density, grain sizes, grain shapes etc... and hard used for characterizing the powder quality.

In spite the simplicity, Stoner – Wohlfarth model is usually used in modified sense to evaluate magnetic properties of complicated systems, which included either the intergrain exchange coupling [3], or the inhomogenous anisotropy distribution [4, 5]. Based on Stoner – Wohlfarth model the present paper evaluates the criterion allowing quick estimation of the quality of NdFeB melt-spun powders used for producing bonded magnets.

II. THEORETICAL

As usually defined, the squareness factor γ is a measure of how square the second quarter of the magnetization (M) hysteresis loop is and that is a dimensionless quantity between 0 and 1, defined by the ratio of the reverse field required to reduce M by 10% from the remanence magnetization M_r to the intrinsic coercivity field ${}_iH_c$. There are several other methods to quantify the squareness of the loop, such as the ratio of M_r to the saturation magnetization M_s. For high coercive magnetic materials, particularly for NdFeB, the squareness factor γ can be defined as the ratio of the two values of the reversal field, in which either the induction B vanishes (${}_BH_c$) or the magnetization vanishes (${}_iH_c$), $\gamma = {}_BH_c/{}_iH_c$.

Below we call the S-W magnets if they consist of Stoner-Wohlfarth particles, which are the single-domain non-interacting grains with uniaxial anisotropy and with the homogenous spontaneous magnetization M_s.

The free energy of Stoner-Wohlfarth particle consists only of the anisotropy and Zeeman contributions:

$$F = -K_u \cos^2(\theta - \theta_o) - HM_S \cos\theta \tag{1}$$

Here K_u is the anisotropy constant, θ_o and θ are the angles between the particle easy axis, the magnetization M and the field H, respectively.

The magnetization loop of the S-W particle in a cycling external magnetic field H is obtained by minimizing the energy F and has the following formula:

$$h = [-2m(1-m^2)^{1/2}\cos(2\theta_o) - (1-2m^2)\sin(2\theta_o)]/2(1-m^2)^{1/2} \tag{2}$$

where m=M/M_S - the reduced magnetization, h= H/H_S - the reduced field and H_s is related to the saturation magnetic field. For NdFeB rapid quenched powders, H_s is about 2.4 T [6, 7].

For characterizations, an amount of powders is compacted into pellets. This powder compaction is considered as an assembly of S-W particles. Based on the equation (2) the magnetization loop of this assembly is derived taking into account the angle θ_o-distribution of particles. For the case of the entire random orientation of the particle easy axes, the loop averaged over all the particles and $\langle h \rangle$ is expressed as follows:

$$\langle h \rangle = \frac{\int\limits_0^{\pi/2} h.\sin(\theta_o)d\theta_o}{\int\limits_0^{\pi/2} \sin(\theta_o)d\theta_o} \tag{3}$$

The equation (3) allows calculating the magnetization loop of a S-W magnet. The loop of the full dense S-W magnet (the entire volume of which consists of S-W particles) is presented in Fig. 1.

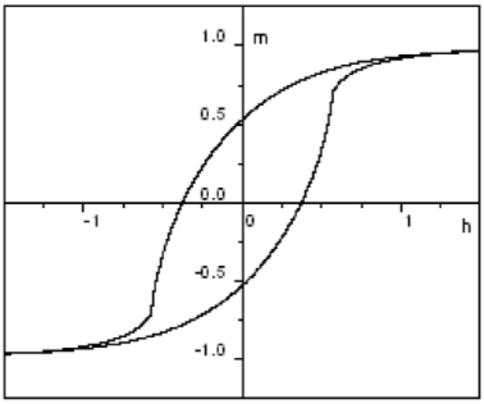

Fig. 1. The magnetization loop of the 100% dense S-W powder compaction

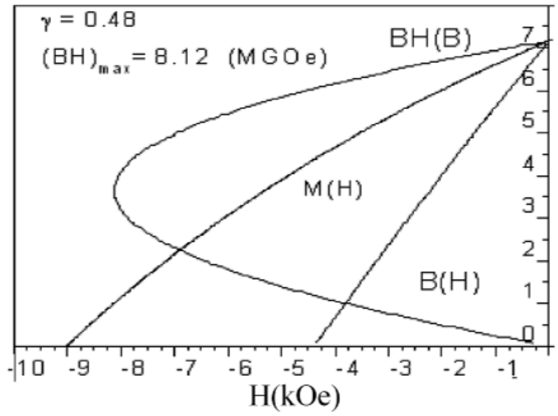

Fig. 2. The demagnetization curve of 6.4 g/cm^3 mass density NdFeB S-W powder compaction

In practice, NdFeB isotropic bonded magnets are produced by hot-compacting resin blended melt-spun flakes, their mass density is about 6.4 g/cm^3. In order to compare with the experimental data, in the same way the loop for NdFeB S-W magnet was calculated. The parameters used for this calculation were: M_s=1.61 T, H_s=24 kOe, the mass density $\rho = 6.4$g/cm^3. Fig. 2 shows the second quarter of this loop (so called the demagnetization curve). Together with the M versus H curve, the dependence B(H)=M(H)–H is also presented in this figure.

Two values 0.48 and 8.12 MG·Oe of the squareness γ and the maximum energy product $(BH)_{max}$respectively, obtained from Fig. 2 are the maximal values of these two parameters for the NdFeB S-W magnet of 6.4g/cm^3 mass density.

The calculation procedure was repeated for all the values of S-W magnet mass density ranged from 4 to 7.6 g/cm^3, and the results are plotted on the figures 3 and 4 for the squareness factor and the maximum energy product, respectively.

The squareness factor γ of NdFeB permanent magnets plays very important role. First, this factor reflects the magnetization process occurred under a reversed magnetic field in magnets, its value strongly depends on the magnet granular microstructure and the magnetic properties of every grain. Secondly, for high coercive hard magnetic materials as isotropic NdFeB , the linearity of B versus H curve is the one of the requirements of high quality of samples. Finally, this factor γ defined as the ratio${}_BH_c/{}_iH_c$ is a parameter easily and precisely to be measured. So the factor γ can be used as the critical parameter for estimating the magnet quality.

The rapid quenched powders, either the melt-spun or atomized ones, after the manufacturing process are compacted into samples of the mass density of the range 4 - 7.6 g/cm^3. Typical compactions of non-binder powders has a mass density of 5 -5.8 g/cm^3, the maximum density of the cold-compressed binder-blended compaction is 6.4 g/cm^3. The hot compaction of non-binder powders can raise the mass density up to 7.6 g/cm^3. For the sample of the given mass density ρ, the squareness value γ defined by using $_B\mathrm{H}_c$ and $_i\mathrm{H}_c$ measured on a BH-graph indicates the powder quality.

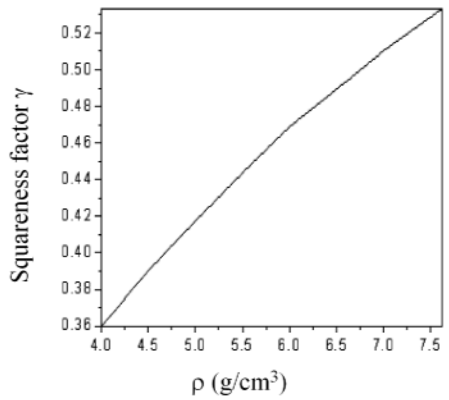

Fig. 3. The squareness factor g of S-W magnets of different mass densities

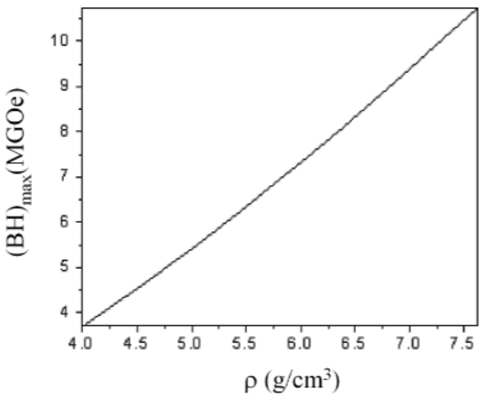

Fig. 4. The maximum energy product of S-W magnets of different mass densities

The low-quality powder has the factor γ below and far from the S-W limit curve sketched in Fig. 3, the grain size of these powders whether is bigger than single domain size or the phase composition is deviated from $Nd_2Fe_{14}B$.

The value of γ factor ranged in the narrow region of the S-W limit curve indicates that the powders are of S-W particles. It is worthy to note that for this case the squareness can not higher than 0.48 for the powder compaction of 6.4 g/cm^3 and even in the case of the full dense compaction 7.6 g/cm^3 – not higher than 0.53 (see Fig.3). Correspondingly, the maximum energy products of 8.1 and 10.7 MG·Oe are the S-W limits for these two compactions.

One observed that there exist NdFeB bonded magnets compacted from the melt-spun, HDDR or strip-casted powders which have the squareness and therefore the maximum energy product higher than the S-W limits presented in Fig. 3. For these case, the magnets are whether anisotropic or exchange-coupling types, so there is strong coupling interaction between grains which enhanced the factor γ in comparison with the case of S-W particles.

III. EXPERIMENTAL

In order to prove the above statements of using the squareness factor γ for determining the powder quality, different types of NdFeB melt-spun binder-blended powders were compacted into the pellets of cylindrical form 10x10mm. The dense bonded magnet samples of the mass density ρ around 6.4 g/cm^3 were compacted by using the hot pressing at 200^{o}C, slight higher than the melt point 180^{o}C of the binder, the pressure was about 6 Tons/cm^2. The demagnetization curve of samples was measured by using the BH –

graph and is shown in Fig. 5 for demonstration. Before measurements, the samples were magnetized in the pulsed 4 Tesla magnetic field.

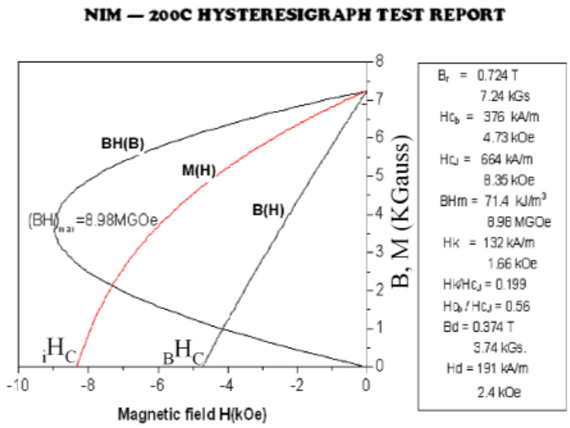

Fig. 5. The closed magnetic cuircuit BH-graph measurement of the hot mould pressed NdFeB isotropic magnet of 5.8 g/cm^3 mass density

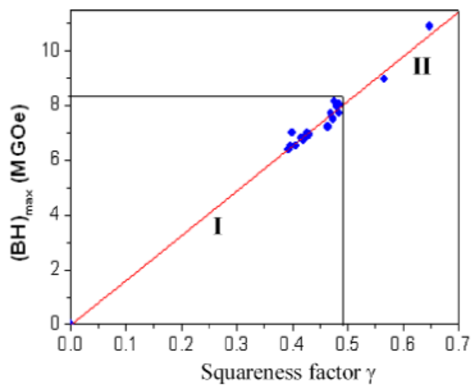

Fig. 6. The experimental data (dots) of the maximum energy product (BH)$_{max}$ versus the squareness factor of NdFeB magnets of various mass densities

Fig. 6 summarizes the measured data of the maximum energy product (BH)$_{max}$ and the squareness factor γ of all the samples. One has two regions on this figure. The first region corresponds to the S-W powders and the second – to the powder with enhanced coupling between grains. The boundary between these two regions corresponds to the two values γ=0.48 and (BH)$_{max}$=8.12 MG·Oe calculated above. Particularly, the powders with magnetic properties within the first region were produced by the conditions far from the optimal ones. The powders within the second region were produced from the flakes melt-spun on the Cu-wheel rotated with the optimal velocity v=31m/s, and the averaged size of micrograins inside flakes estimated by TEM is in the range 60 – 80 nm.

IV. CONCLUSIONS

It is proven, in the framework of Stoner-Wohlfarth model, that the squareness factor γ defined as the ratio between two values $_B$H$_c$ and $_i$H$_c$ of coercivity can be served as the critical parameter of estimating rapid quenched powders. For the powder compaction of 6.4 g/cm^3 mass density the measured value γ=0.48 serves the evidence of the Stoner-Wohlfarth behaviour of the powder grains and the preparation conditions are optimal for producing Stoner-Wohlfarth particles. In contrary, if γ less than 0.48 then the preparation conditions were far from the optimal ones. The value higher than 0.48 of the factor γ proves that in the prepared powder the coupling between grains are enhanced. Since the factor γ is very easily and precisely measured, so one can use this criterion for quick tool of evaluation of manufactured NdFeB rapid quenched powders. For the powder compaction of given mass densities the calibration curve of the squareness factor γ is presented in Fig. 3. One notes also that for the ideal, full dense compaction of NdFeB Stoner-Wohlfarth particle the squareness factor γ and the maximum energy product $(BH)_{max}$ can not be larger than 0.53 and 10.7 MG·Oe, respectively.

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