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Determination of intrinsic magnetic parameters of $SmCo_5$ phase in sintered samples

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ABSTRACT

$SmCo_5$ magnets are usually produced by powder metallurgy route, including milling, compaction and orientation under magnetic field, sintering and heat treatment. The samples produced by powder metallurgy, with grain size around 10 μm, are ideal for determination of intrinsic parameters. The first step for determination of intrinsic magnetic parameters is obtaining images of domain structure in demagnetized samples. In the present study, the domain images were produced by means of Kerr effect, in a optical microscope. After the test of several etchings, Nital appears as the most appropriate for observation of magnetic domains by Kerr effect. Applying Stereology and Domain Theory, several intrinsic parameters of $SmCo_5$ phase were determined: domain wall energy 120 erg/cm^2, critical diameter for single domain particle size 2 μm and domain wall thickness 60 Å. In the case of $SmCo_5$, and also other phases with high magnetocrystalline anisotropy, Domain Theory presents several advantages when compared with Micromagnetics.

Introduction

Among the most fundamental parameters that describe the magnetic characteristics of a given ferromagnetic phase - besides the magnetization of saturation and the anisotropy field - are the domain wall energy, the critical diameter for single domain particle size and the domain wall thickness.

There are several methods for determination of those intrinsic properties [1-4] and, as consequence, the values reported in literature for the domain wall energy of $SmCo_5$, found using those different methods, are within an extensive range (36-154 ergs/cm^2 [1-3]). Thus, we decided carrying out our own determination, as well as presenting a discussion about the methods employed and their reliability.

Experimental Procedure

The experimental procedure comprises [5]: i) milling until 3-4 μm. ii) orientation and compaction under magnetic field – 1 T iii) sintering at 1150°C, during 30 min. under 1.0 atm of argon iv) a heat treatment that includes cooling at 2°C min from 1150 down to 850oC, plus 150 min at 850°C, the entire cycle under 1.0 atm of argon.

The different chemical etchings tested were: i) Nital. A solution HNO_3 and alcohol, with nitric acid at 2%. ii) Chromic acid. A solution with 8g CrO_3 and 2g Na_2SO_4 in 100 ml of H_2O. iii) No etching: sample only polished. Among these three different conditions, it was found that Nital is the only etch able to allow the domain observation by Kerr effect (under polarized light).

Results and Discussion

Magnetic Domains and Estimative of Intrinsic Parameters of $SmCo_5$

One of the greatest mysteries about the $SmCo_5$ magnets is the large increase of coercivity (from 1-5 kOe up to 50 kOe) after the heat treatment at 850°C, which takes place without any apparent effect in the microstructure [6,7]. In an attempt for elucidating this question, the domain structures before and after the heat treatment were studied (Fig. 1 and 2). But no differences were found when comparing these two different conditions (Fig. 1 and 2), thus the increase of coercivity is not associated with factors related to domain structure.

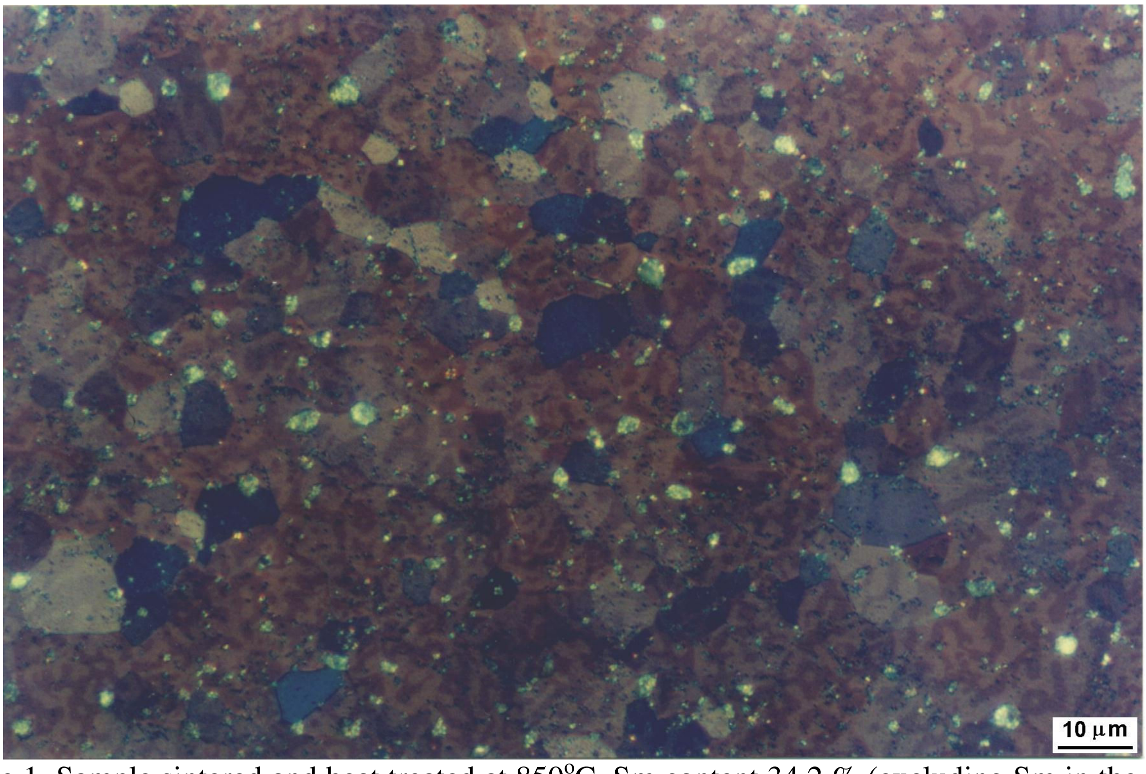

Figure 1. Sample sintered and heat treated at 850°C. Sm content 34,2 % (excluding Sm in the oxide Sm_2O_3 and carbide $SmCoC_2$) [5] $_iH_c$ >25 kOe. Nital etch. Domains revealed by Kerr Effect. The bright particles are oxides under polarized light. Transverse section to the easy axis of the magnet.

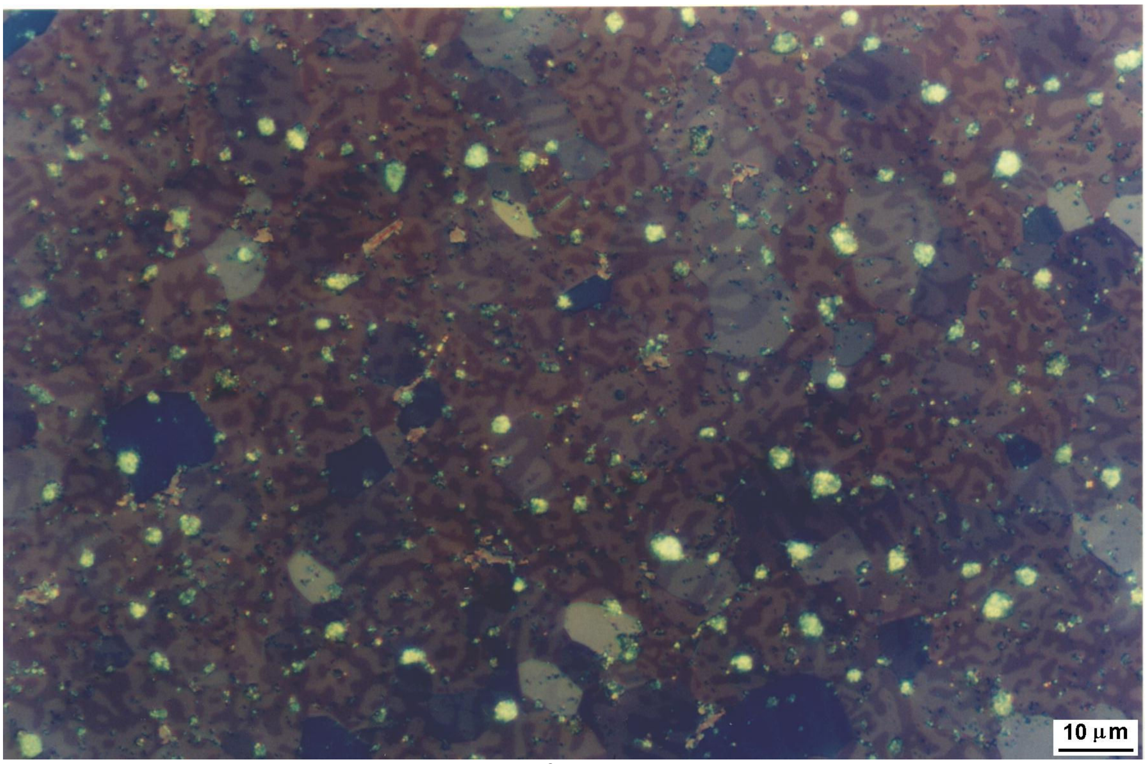

Figure 2. Sample “re-sintered” ½ hour at 1150°C. Sm content 34,2 % (excluding Sm in the oxide Sm_2O_3 and carbide $SmCoC_2$) [5] ${}_iH_c$ = 2.3 kOe. Nital etch. Domains revealed by Kerr Effect. The bright particles are oxides under polarized light. Transverse section to the easy axis of the magnet.

Using expression (1) [8] the domain wall energy γ, can be found:

$$D = \frac{1}{M_s}\sqrt{\frac{\gamma \cdot L}{1.7}} \tag{1}$$

where L is the grain size, Ms is the magnetization of saturation, D is the domain width. From the figures 1 and 2, it is determined $\overline{D} \approx 3$ µm. It was previously found [5] that L ≈ 10 µm. Using the value 4πMs=11,2 kG or Ms=890 emu [9], the Eq. (1) results: $\gamma \approx 120$ erg/cm^2.

Expression (1) has validity only for small grain size. According Livingston and McConnell [1] that followed Kaczer [10], if $L \geq 32\ \gamma / Ms^2$ then, as consequence of formation of surface domain structures for reducing overall magnetostatic energy, domain spikes and other domain structures that result in domain refinement tend to appear in the surface. But this problem is avoided in our study because the limiting grain size is 48.5 µm, well above the grain size of our samples.

The critical diameter D_c for monodomain particle can be found by expression (2) [8]: $D_c \approx 2$ µm.

$$D_c = \frac{9}{2\pi}\frac{\gamma}{M_s{}^2} \tag{2}$$

Using the values $K_1 = 1.4\ 10^7$ ergs/cm^3, $K_2 = 0.5\ 10^7$ ergs/cm^3 [6], the domain wall thickness δ can be found with expression (3): δ ≈ 60 Å.

$$\delta = \frac{\pi \cdot \gamma}{4 \cdot K_1} \frac{1}{\sqrt{1 + K_2 / K_1}} \quad (3)$$

The values determined for γ , D_c e δ are in excellent agreement with others reported in literature for $SmCo_5$ [1,4].

It should be pointed out that the equation $\gamma=4(AK_1)^{1/2}$, where A is the exchange Constant, does not allow reliable results for the case of $SmCo_5$ [1], because when the domain wall is very thin (the case of phases with very high K_1), the estimation of A by the equation A=k Tc/ 8 d is not realistic and this leads to errors [1]. k is the Boltzmann constant, Tc is the Curie temperature and d is the minimum distance between spins parallels in the direction normal to the domain wall and, in the case of $SmCo_5$ it is adopted d = ½ a [1], where a is the lattice parameter, see for example ref. [6].

When the domain wall is very thin, a consequence of very high anisotropy field, the approximations (it is supposed that the exchange forces act only between nearest-neighbor atoms and that for a sufficiently large number of atoms inside the wall θ is small and that $\cos\theta \cong 1 - ½\,\theta^2$) used by Bloch [8] to find the expression $\gamma=4(AK_1)^{1/2}$ (as consequence of solving the exchange energy expression given by Eq. 4) are no longer valid.

$$E_{EX} \cong A\left(\frac{d\theta}{dx}\right)^2 \quad (4)$$

where θ is the angle between the magnetization of a given atom inside the wall and the bulk and x is a discrete space inside the wall.

As it was pointed out by Zijlstra and van den Broek [11,12], the necessity of taking into account the discreteness of the atoms in the lattice makes the approximation $\theta \cong 0$, used by Bloch, unrealistic. The theory named "Micromagnetics" always use expression (4), the so-called [1] "standard continuum model of a domain wall", that contains the mentioned approximations, so we conclude that it should be avoided the application of Micromagnetics for high anisotropy phases (as $SmCo_5$, $Nd_2Fe_{14}B$). However, the Domain Theory [8] allows directly the calculation of domain wall energy by means of equation (1), avoiding the use of the above mentioned approximations and, thus, is more appropriate for high anisotropy phases.

The intrinsic parameters reported in ref. [2,3] were calculated using equation A=k Tc/ 8 d, and according the several reasons above presented, should be considered less reliable that the values reported in ref. [1,4].

It should be added that the values γ, D_c e δ are necessary parameters in models for the estimation of variation of coercive force as function of the grain size, fact that increases the practical relevance of this determination.

Conclusions

Nital appears as the most appropriate etching for observation of magnetic domains by Kerr effect.

Some intrinsic parameters of $SmCo_5$ phase were determined: domain wall energy $\gamma \cong 120$ erg/cm^2, critical diameter for single domain particle size $D_c \cong 2$ μm and domain wall thickness $\delta \cong 60$ Å.

In the case of $SmCo_5$, and also other phases with high magnetocrystalline anisotropy, Domain Theory presents several advantages when compared with Micromagnetics.

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