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THE INFLUENCE OF ANISOTROPY AND DOMAIN STRUCTURAL FACTORS ON INITIAL PERMEABILITY OF PERMALLOYS

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Résumé

Des mesures ont été effectuées sur du permalloy contenant du molybdène (Ni : 80,2% , Mo : 4,7% , Mu : 0,5% , Fe : 14,6%) pour étudier les énergies d'anisotropie magnétocristalline et magnétostrictive, ainsi que l'influence de la taille des grains et de la teneur en impuretés sur la perméabilité initiale /IP/ et la force coercitive /CF/. La perméabilité initiale, mesurée à une valeur donnée de l'anisotropie, est influencée essentiellement par la teneur en S et O₂. Des conditions de fabrication et de recuit plus avantageuses ont également été déterminées.

Abstract

Examinations were carried out on Mo-permalloy (Ni: 80.2 wt%; Mo: 4.7 wt%; Mn: 0.5 wt%; Fe: R) to study the effects of the magnetocrystalline and magnetostrictive anisotropy energies, the grain size and impurity contents on the initial permeability /IP/ and coercive force /CF/, respectively. The IP was influenced mainly by the S and O₂ content at given anisotropy energies. More advantageous manufacturing and annealing conditions were also determined.

Introduction

This work intends to study the Mo-permalloy material in respect of essential physical effects which determine the IP and CF of the material so that the most advantageous manufacturing and annealing conditions could be determined. The magnetic anisotropy energy directly unfolds its effect on the IP and CF by means of the rotational process of magnetisation. An indirect effect, however, on the IP and CF can also be attributed to the anisotropy energies through the determination of the domain wall energy and the actual built-in of the domain structure in a given grain structure. The grain structure and impurity content also unfold a considerable influence on the domain wall motion, which appears in the technical parameters, as well. The complex appearance of these effects can only be studied by simultaneous examinations of anisotropy energies and domain structure.

In the work presented here we proposed to examine how the magnetocrystalline and magnetostrictive anisotropy energies, the grain size and impurity contents determine the IP and CF, respectively. The anisotropy parameters (K_1 ; λ_{100} ; λ_{111}) were derived from advanced approach to saturation measurements /1,2/ while the domain structure characteristics - such as average domain size (2a); domain wall restoring pressure (α); rotational part of the IP (μ_{ω}) - were derived from the fit of the complex IP vs. frequency spectra with proper theoretical models /3,4,5/. The average grain size (D) was determined by linear intercept method and the impurity content by the thermal conductivity analysis /O₂ content/ and infra-red analysing method /C and S contents/.

Experimental

Two parallel experiments were performed so as to reach our aim: at first, the

magnetocrystalline and magnetostrictive anisotropy energies were changed widely, while the average grain sizes and impurity content were restricted at nearly equal level /ExpI/. For second, very different grain sizes and impurity contents were generated at approximately equal anisotropy constants /ExpII/. Both experiments were performed on Mo-permalloy (Ni 80,2 wt%; Mo 4,7 wt%; Mn 0,5 wt%; R Fe) materials.

In ExpI the samples were prepared from sheets cold rolled to the required 0.2 mm thickness, a 50 % final reduction. Toroidical samples were punched for the IP and CF measurements and 50x2 mm strips were cut from the same part of the sheet for the anisotropy measurements.

Each sample was annealed for 4 hours at the 1250 °C temperature, then furnace cooled at 100 °C per hour to room temperature. This kind of heat treatment yielded more or less same average grain sizes - about 0.52 mm of diameter - and the same measure of the purification, which means C<10ppm; S<2ppm; O<25ppm, respectively.

To reach the proper anisotropy energies the samples were baked in vacuum for 2 hours at a temperature (T_q) ranging between 400-800 °C that was followed by rapid cooling to room temperature. This baking did not essentially influence the grain sizes and impurity contents established earlier. This kind of preparation of the samples made it possible for us to reach very different anisotropy energy values with constant grain sizes and impurity contents. The constants of the magnetocrystalline and magnetostrictive anisotropy energies - K₁; and λ₁₁₁ - were determined by means of approach to saturation measurements improved by us earlier for polycrystalline materials. In this method the common examination of the external magnetising field (H) and the external elastic stress (σ_e) dependence of the approach to saturation made it possible to experimentally eliminate the influence of the disturbing non-anisotropic effects. These measurements are suitable for the determination of very low value of K₁ (in order of 10 J/m³) and λ₁₁₁ (in order of 10⁻⁷) (see further details in ref.2)).

The toroidical and strip samples of the Exp II. were prepared from cold rolled sheets of different thickness and final reductions (see Table 1). The atmosphere of the final annealing was also varied but all the samples were exposed to the same annealing procedure: holding on 1250 °C for 4 hours then cooled in furnace at about 100 °C per hour to room temperature.

This kind of preparation of samples resulted in very different grain sizes and purifications during the holding, while the fairly similar cooling process yielded close values of the anisotropy and magnetostrictive constants /showed also in Table 1).

Table 1: The data of the samples prepared for Exp II.

Sample	:	1	2	3	4	5	6	7	8
Sample thick./mm/	:	0.2	0.2	0.2	0.2	0.2	0.15	0.3	0.4
Annealing atmos.	:	H ₂	V	H ₂	H ₂	H ₂	H ₂	H ₂	H ₂
Final reduct./%/	:	50	80	80	50	50	50	50	50
K ₁ /J/m ³ :	:	-9.1	-12.2	-8.6	-3.2	-4.5	-8.7	-4.2	-10.4
(λ ₁₀₀ -λ ₁₁₁) 10 ⁻⁶ :	:	1.6	1.7	1.6	1.2	1.2	1.6	1.3	1.7
IP	:	42530	30710	49120	71550	57850	42210	53850	36070

The anisotropy measurements were carried out on strip samples, while the IP, CF and complex IP measurements on toroidical samples.

The complex IP measurements were performed on Maxwell bridge in very low fields between 0.016-0.04 A/m in the 80 Hz-26 kHz frequency range. Linear extrapolation for H→0 was applied to determine the field-independent complex IP values for each selected frequency point. All of the samples were measured after a 50 Hz demagnetisation, where the starting field applied was 40000 A/m, respectively (see further details in ref.6).

The three applied theoretical models to fit the measured spectra were derived from the equation of motion of the 180° Bloch walls by K.L.M. Polivanov /3/ and J.E.L. Bishop /4,5/.

All of the models have some common assumptions related to the domain structure such as : mainly 180° Bloch wall form a bar-shape domain structure ; the

Figure 1. The effect of K_1 and λ_{111} on the IP and CF as a function of baking temperature (T_q).

Figure 2. The effect of the average domain size and impurity content on the domain structural and technical magnetic properties. The examined parameters - the μ_0 and H_c ; the impurity contents (C, S, O₂); the restoring pressure (α); the rotational ratio of the IP (μ_0/μ_0); and average number of domains in a grain ($D/2a$) - are presented as a function of the average grain size (D).

walls are perpendicular to the sheet surfaces and don't interact with each other; the magnetization of domains has no normal component to the sheet surfaces. The differences of the used models are in the assumptions related to the domain wall motion: the Polivanov model /3/ supposes ideal bowing motion of the walls, which means that the inertia of the walls (m), the spin relaxation damping (β), the restoring forces (α) and the finite wall energy (γ) were not taken into account. Only the average domain wall spacing ($2a$) determine the actual value of the complex IP (P.model). Bishop improved the Polivanov model in two ways so expanding the validity of the model on higher frequencies. His first approximation /4/ discussed a more complex way of motion where α, γ, β were also not zero (BI. model). In his other solution /5/ only the rotational magnetization process was considered in addition to the ideal bowing motion of walls (BII.model) - which was neglected in two solutions mentioned above.

Results

The results of these experiments are presented in fig.1 and 2.

Fig. 1. shows the effect of λ_{111} and K_1 on the technical magnetic properties (IP and CF). It can be seen that zero crossing of K_1 and λ_{111} show increasing in IP and decreasing in CF and K_1 has a greater effect than λ_{111} . This examination made it possible to determine an ideal T_q temperature for the rapid cooling following the heat treatment. That was 500 °C for this composition.

The results of Exp II can be seen in Fig.2. Two results are striking at the study of this figure: every parameter strongly correlates to the IP, except the (D/2a) ratio that shows similar behaviour to that of CF.

Let us consider the case of IP. When the domain wall motion was hindered by the impurities, the restoring pressure rose onto higher values and the rotational ratio in the magnetization process was also considerable - all that appears in a lower IP value. In this point of view the carbon content carried no importance. All these results are in good agreement with the observations of many other workers /8,9/.

The changes of the CF were rather monotonous, which was expected in this consideration. Only the (D/2a) ratio correlated closely to the CF, which is in good agreement with our earlier results: we had found that - in the materials where K_1 is near to zero - the (D/2a) ratio is basically determined by the average grain size and the sheet thickness /7/.

This experiment also proved some result in technological respect: the best IP and CF values were reached at 50 % final reduction and 0.2 mm sheet thickness annealing the samples in H₂ atmosphere.

Conclusion

On Mo-permalloy material we performed examinations to determine the effects of the magnetocrystalline and magnetostrictive anisotropy energies and the effects of the grain and impurity structures on the IP and CF, respectively. The effect of the K_1 was more decisive than that of the λ_{111} and at given anisotropy energies the IP was basically governed by the impurity content while the CF was mainly determined by the grain and statical domain structure.

A more advantageous manufacturing and annealing condition was also found for this material.

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