ANISOTROPY OF COERCIVE FORCE OF SINGLE CRYSTALS AND SHEETS OF SILICON IRON WITH DIFFERENT TEXTURE

Natalia Shkatulyak[1], Elena Dragomeretskaya[1], Valentin Usov[2]

[1]Department of Physics, South Ukrainian National Pedagogical University named after K.D. Ushinsky, Odessa, Ukraine
[2]Department of technical disciplines, South Ukrainian National Pedagogical University named after K.D. Ushinsky, Odessa, Ukraine

Abstract

In this paper we investigate the regularities of anisotropy of coercive force both single crystals with the orientation of surfaces parallel (001) so and the polycrystalline sheets of alloy Fe - 3% Si with different texture. The mechanisms of anisotropy of coercive force are discussed.

Keywords

Anisotropy, Coercive Force, Non-destructive Method, Texture, Domain Structure

1.INTRODUCTION

Effect of anisotropy of the magnetic properties of ferromagnetic crystals, theoretically grounded by N.A. Akulov in the 1928 year [1], has practical application. A number of studies (e.g., [2-5]) show the possibility of estimation of the structural state of ferromagnetic structural materials, the level of accumulated in them fatigue damage, the internal stresses by measuring of the coercive force H_c, which is the main characteristic of the magnetic hysteresis loop and, by definition, does not depend on the geometric sizes of sample. Coercive force is equal to the demagnetizing field that should be applied to the magnetized ferromagnetic materials order to reduce it to zero magnetization. In general, the coercive force can be represented as the sum of components including the magnetocrystalline anisotropy, internal elastic stresses arising in the presence of lattice defects, as well the grinding of crystals and their elongation [1].

In [6] was found a linear correlation of coercive force H_c with pole density in rolling direction RD and transverse direction TD and corresponding broadening of the X-ray diffraction lines with increasing of strain ratio by internal hydraulic pressure of pipeline samples of steel.

Considerable number of papers is devoted to the study in alternating and rotating magnetic fields of magnetization anisotropy, magnetic losses, magnetostriction and coercive force in single crystals of silicon iron with different orientations and in sheets of electrical steel with Goss texture {011}<100> [7-11]. It is shown that, for example, anisotropy of the magnetization and coercive force is not always the same and depends not only on the crystal orientation or texture type and its sharpness, but also on other factors such as magnitude of external field, frequency of its changes, size of crystals, domain structure and its dynamics. Anisotropic characteristics in above mentioned papers usually were measured on samples cut in the appropriate directions, as well on disks.

At the present time are the standard coercimeters allow making measurement the coercive force of various products and structural elements, including in the field terms without cutting of samples. However, regularities of anisotropy H_c measured by non-destructive methods have been insufficiently studied.

The aim of this paper is to establish regularities of anisotropy of the coercive force measured by non destructive method in single crystals of silicon iron and in sheets of alloy Fe-3% Si with different texture.

2.METHODOLOGY

As starting materials used the strip of 0.2 mm of thick of alloy Fe - 3% Si with large single crystals of size ≈ (10×6) cm, and sheet of alloy Fe - 3% Si (by weight) 2.5 mm of thick with the equiaxed grains (average size ~ 22 μm) under conditions of factory shipment.

The X-ray method was used for study of texture of sheets. Texture analysis was carried out by means of direct and inverse pole figures (IPF) of normal direction (ND) to the plane of sheets, rolling direction (RD), as well of diagonal direction (DD, i.e. RD + 45º) and of transverse direction (TD). Composite samples were used for the record of inverse pole figures of RD, DD and TD.

Coercive force H_c was measured by means of non-destructive method using coercimeter KRM-Ts-MA. Device at first magnetizes the sample to saturation, then demagnetizes it to zero, further the full magnetic hysteresis loop subjected to electronic processing with evaluation of coercive force value. The maximum error does not exceed 2% [12]. In large single crystals H_c was measured in direction [100] (H_{c001}) and directions that are deviated from [100] at angles 45º (H_{c110}), as well 90º (H_{c010}) lying in the plane of single crystals (001). In sheet H_c was measured in the rolling direction (H_{cND}), diagonal direction (H_{cDD}, i.e. in RD+45º) and the transverse direction (H_{cTD}) of sheets.

Further the sheet was rolled into original factory rolling direction (the direct rolling) at room temperature with small reductions (~ 3 - 5 %) by a laboratory rolling mill with a rolls diameter of 180 mm up to thickness of 1.5 mm. Further the part of such rolled sheet was subject to subsequent rolling in cross direction (the transverse rolling) to a final thickness of 1.17 mm. The coercive force was measured in the above directions after direct and transverse rolling. A series of isochronous (of duration 1 hour) vacuum annealing through every 30 - 50ºC was carried out to determine the start recrystallization temperature ($T_{s.r.}$), which was determined by means of X-ray photo method by the appearance of first points on the diffraction rings of powder patterns. We are found that in our samples $T_{s.r.}$ is approximately 500ºC. Then, the sheets after rolling were subjected to annealing for 1 hour at 450º C to relieve stresses without recrystallization, in order to not change of texture. The coercive force was measured again after annealing in mentioned above directions of sheets.

The metallographic structure was studied in the reflection mode from the rolling plane of samples by means of metallographic microscope MIM-7 with the camera VEB-E-TREK DEM 200, which allowed displaying the image of microstructures on a computer screen. The investigated surfaces before study were mechanically polished and etched in a 5% solution of picric acid in ethanol.

3.RESULT & DISCUSSION

The measurement results of H_c in single crystals of Fe - 3% Si with the orientation (001)[110] that were averaged on data of five grains are shown in Table 1.

Table 1. Coercive force in different directions (001) single crystals of silicon iron Fe - 3% Si.

Coercive force, A/cm		
H_{c100}	H_{c110}	H_{c010}
0,45	0,55	0,4

It can be seen that the maximum value of H_c is observed in the direction of axis of middle magnetization, which coincides with the crystallographic direction [110] [1].

Figure 1 shows IPF of initial sheet of alloy Fe - 3% Si. The maximum of pole density 2.26 observed in the pole <011> of IPF ND. This means that a family of crystallographic planes {011} lie parallel to the plane of initial sheet.

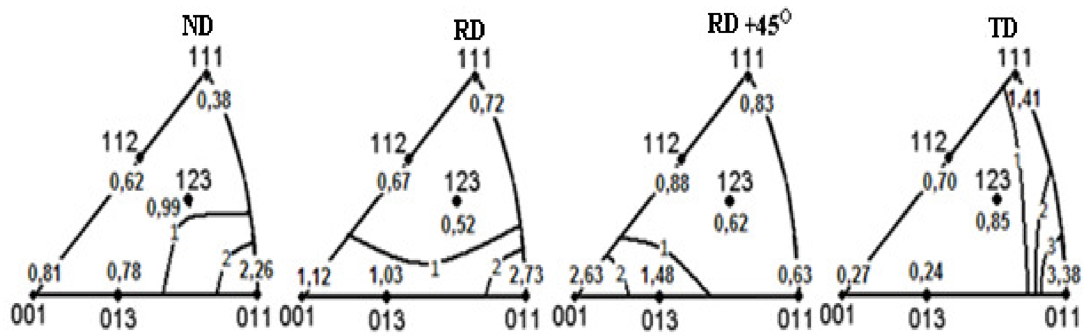

Figure 1. IPF of initial sheet of alloy Fe - 3% Si of 2.5 mm thickness

A continuous distribution of pole density along diagonal <001> – <011> of the stereographic triangle is observed at this on IPF RD and the maximum value of 2.73 of pole density corresponds to the pole <011>. On IPF RD+45º the maximal pole density is in the pole of <001>. At the same time on IPF TD is observed a continuous distribution of pole density along the diagonal stereographic triangle <011> - <111> with a maximum of 3.38 in the pole <011>. Based on the above-described distribution of orientations in Figure 1 one can be concluded that in initial sheet is formed the limited axial texture with the axis <110> parallel to the ND. This texture can be described by a combination of ideal orientations {011} <100> + {011} <533> with roughly the same content in volume.

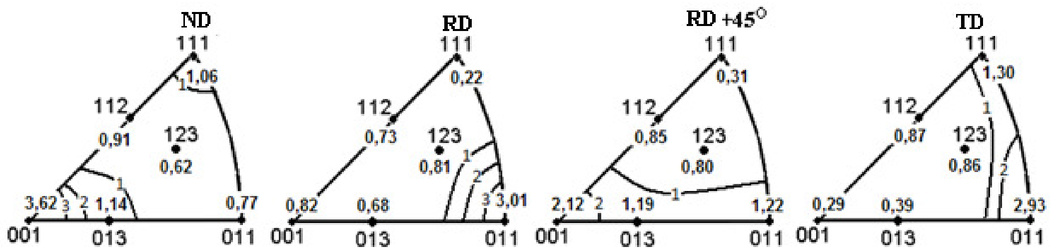

Figure 2. IPF of sheet of alloy Fe - 3% Si after direct rolling to a thickness of 1.5 mm

It can be seen that pole density maximum 3.62 corresponds to the pole <001> on IPF ND. The enhanced pole density of 1.06 is also observed in the pole <111>. This means that crystallographic planes {001} and {111} are arranged parallel to the rolling plane. At the same time takes place the pole density maximum 3.01 on IPF RD in the pole <110>. A continuous distribution of pole density along <011> - <111> diagonal of stereographic triangle is observed on IPF RD+45º. Above mentioned distribution of pole density on IPF of sheet after direct rolling is evidence on the development of limited axial texture that can be described as a combination of ideal orientations {001} <110> + {111} <110> with approximate content of 77 and 23% in volume, respectively.

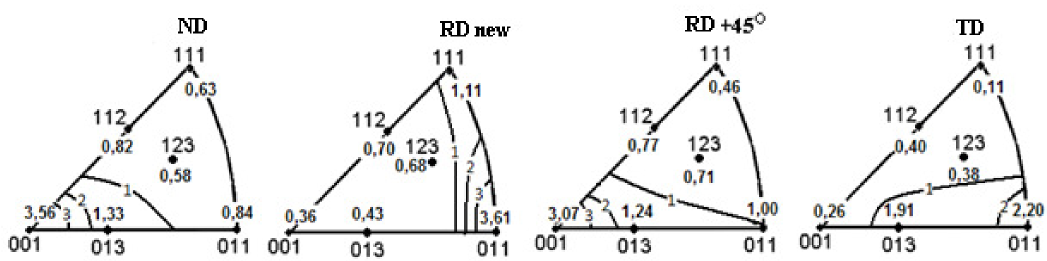

Figure 3. IPF of sheet of alloy Fe - 3% Si after the transverse rolling to a thickness of 1.17 mm

The Figure 3 shows that was formed texture of type of rotated cube {001} <110> after transverse rolling in the sheet.

The results of measuring the coercive force in the starting sheet are shown in Table 2.

Table 2. The coercive force in the initial sheet of alloy Fe – 3 % Si

Type of texture	Coercive force H_c, A/cm		
	H_{cRD}	H_{cDD}	H_{cTD}
{011}<100>+{011}<533>	2,04	2,31	2,40

The results of measuring the coercive force in the sheets after rolling and annealing are shown in Table 3.

Table 3. The coercive force in the rolled and annealed sheets of alloy Fe – 3 % Si

Type of texture	Coercive force H_c, A/cm					
	Before annealing H_{c_rolled}			After annealing $H_{c\_annealed}$		
	$H_{c\mathrm{RD}}$	$H_{c\mathrm{DD}}$	$H_{c\mathrm{TD}}$	$H_{c\mathrm{RD}}$	$H_{c\mathrm{DD}}$	$H_{c\mathrm{TD}}$
Direct rolling {001}<110>+ {111}<110>	4,5	4,7	4,9	2,4	2,6	2,7
Transverse rolling {001}<110>	4,6	5,2	5,7	2,4	2,6	2,9

The Table 2 show that takes place the anisotropy of coercive force in initial sheet. The minimum value of coercive force is observed in RD, maximal value correspond to the TD, and in the diagonal direction (i.e. in a direction at an angle of 45º to RD) the coercive force has an intermediate value.

In [10] investigated the anisotropic magnetic properties of two grades of electrical steel with 3.25% Si of 0.27 mm thickness with Goss texture. Measurements using samples of Epstein size cut with interval of 10º between the RD and TD showed that variation of the magnetizing field H in the steel is directly proportional to internal energy W_k of magnetic anisotropy of a cubic crystal. The maximum of the both values as H, as well and W_k correspond to the direction of hard axis of magnetization [111], which lies at an angle of 55º to the RD in a sheet with a sharp Goss texture.

Anisotropy of the coercive force in sheets of isotropic, as well anisotropic electrical steel with the Goss texture was noted also in study [13] that was carried out on samples cut by of Epstein. A minimum of H_c, was found in the direction [100], which is located in the RD of grain oriented steel. The maximum of H_c was found in the direction [110] lying in TD, but not in direction [111] that is located under 55º to RD in sheets with Goss texture. The ratio of H_c90º > H_c55º in [13] is explained by results of the magnetization process. As an example, was indicated that value of magnetic induction B90º is less than the B55º at the intensity of applied field H_0 < 200 A/m [13] in the sheet of grain-oriented Fe - 3% Si steel M6 by classification AISI. Thus, the possible cause of the anisotropy of the coercive force, according to their data can be not only a sharp texture.

A similar behaviour of the coercive force in the transformer steel with 3.2% Si with Goss texture was observed in [14]. According to the measurement of Barkhausen noise [14] it was shown that the 180º domain walls with such texture oriented parallel to the RD. When the field is applied to the TD, i.e. perpendicular to the 180º domain walls, their motion is impossible. In this case, an irreversible rotation of 90º should to occur before any movement of the domain walls that means a lower initial permeability for 90º sample and a maximum of coercive force in TD of sheet.

In [11] investigated the magnetic field in the samples of size Epstein that were cut in the directions 0º, 15º, 30º, 45º, 60º and 90º from the RD to the TD of sheets isotropic electrical (NO i.e. Not Oriented) and anisotropic (GO i.e. with Grain Oriented) with 3.5% Si steel, having a Goss texture in a range of frequency variation from 5 to 200 Hz alternating magnetic fields with magnetic induction of 0.5, 1.02 and 1.5 T. It was found that at small induction of the external field (B = 0.5 – 1.02 T) in an anisotropic steel with Goss texture the easy magnetization axis lies in the RD, and the axis of hard magnetization is oriented in the TD. Change frequency of induction B of external magnetic field does not affect on the location of the easy and hard magnetization axes. The axis of hard magnetization lie at an angle of 60º to the RD and the east magnetization axis is oriented in RD in the case of high value of magnetic induction B. The influence of magnetic induction B on orientation of the axes of easy and hard magnetization was explained in [11] due to the interaction and movement of 90º and 180º domain walls during the magnetization. According to [11] axis of difficult magnetization is located at an angle of 60º to the RD (close to the direction [111] at the texture of Goss), but not in the TD at a small value in a GO steel, since the this case the orientation of grains are not so important as at high values of induction B. Similar behaviour is observed in the isotropic NO steel at high induction. Anisotropy is not as strong as in the case of Fe-Si GO steel [11]. It is noted that the position of the axis of anisotropy turned directly related to the specific energy loss. The lowest losses were observed in the rolling direction (GO and NO) and the highest value were found in the direction under 60° to the RD in the case of GO steel and, respectively, in the TD in the case of NO steel.

Anisotropy of hysteresis losses associated with the position of easy and hard axis of magnetization also was found in [9]. Losses in alternating and rotating magnetic fields were measured on small single-crystal disks of 4% silicon iron with the surface (110). The results were explained on the basis of changes in the domains structure observed in various conditions. The surface energy of the domain walls is released as heat when the two adjacent walls merge and disappear [9], so that the total energy of the walls, which are alternately appear and disappear during each cycle, can be transformed into hysteresis loss. If the external magnetic field is applied in the [100] direction in the (110) plane, are formed oppositely oriented 180º domains. Losses are minimal. Magnetization in the [110] direction under the influence of the alternating field is done by the formation of a finely ground 90º domain structure. Most of the hysteresis loss under such conditions can be attributed to the energy dissipation due to domain wall annihilation. When a field is applied in the direction [111] such domain structures are formed much rarer than in the previous case. Accordingly, the loss in the [111] direction are less than losses in the [110] direction.

A similar impact of the contribution of interaction 180º and 90º domains in coercivity was presented previously in [7], where was obtained a quantitative relation between the value of H_c with the energy per unit volume of cross-domain and their relative volume. The results obtained by means of proposed ratio, were close to data of their experiments.

Not excluding the impact of the above factors on the observed anisotropy of the coercive force (Table 2) let us estimate the energy of magnetic anisotropy in initial sheet of alloy Fe - 3% Si. Internal energy in the investigated ferromagnetic material obeys to the criterion of minimizing [6]. At a homogeneous internal structure of the material and the absence of an external applied voltage can be assumed that the coercive force is due only to the magnetic anisotropy energy. Anisotropy energy W_c of material for a cubic system has the form [5]:

$$W_k = K_0 + K_1\left(\alpha_1^2\alpha_2^2 + \alpha_2^2\alpha_3^2 + \alpha_1^2\alpha_3^2\right) + K_2\left(\alpha_1\alpha_2\alpha_3\right), \qquad (1)$$

where α_1, α_2, and α_3 α3 are direction cosines of the magnetization relative to the axes of the cube; K_0, K_1 and K_2 are anisotropy constants.

The constant K_0 is not dependent on the angle, and K_2 is small and can be neglected. Thus, the energy of the magnetocrystalline anisotropy in the first approximation can be expressed as

$$W_k \approx K_1\left(\alpha_1^2\alpha_2^2 + \alpha_2^2\alpha_3^2 + \alpha_1^2\alpha_3^2\right). \tag{2}$$

We call the expression

$$\psi = \left(\alpha_1^2\alpha_2^2 + \alpha_2^2\alpha_3^2 + \alpha_1^2\alpha_3^2\right) \tag{3}$$

magnetocrystalline anisotropy energy function.

Values 0.173; 0.312; 0,315 for RD, DD and TD, respectively, were obtained by calculating the function Ψ for ideal orientations given in Table 2. Correlation analysis carried out by us showed the close linear correlation between values of the function Ψ and H_c. The reliability coefficient of approximation $R^2 = 0.95$.

Thus, the anisotropy of coercive force that is observed by us in the initial sheet of alloy Fe - 3% Si may be related mainly to the influence of the magnetocrystalline anisotropy energy.

The Table 3 shows that after rolling (but before annealing), the value of the coercive force significantly increased in comparison with its value in the initial sheet. Likely increase in the coercive force of alloy sheets was caused by internal stresses appeared during the rolling. The anisotropy of H_c also takes place. After annealing, the value H_{c_annealed} decreased almost halved compared to H_{c_rolled} after rolling. We detected the close linear correlation with the between values of the coercive force before and after annealing. Reliability coefficients of approximation R^2 in this case are not less than 0.96. This means that influence of the stresses due to the rolling on coercivity of sheets was isotropic.

Annealed sheets after rolling are showing the anisotropy of coercivity similar to that of the source sheet, although the textures are different from of the original texture (Figures 1-3). As shown in Table 3, the minimum of H_c after direct rolling is observed in RD, maximal value H_c takes place in TD and in direction at 45° from the RD (i.e. in DD) H_c has intermediate value. In this case the observed anisotropy of coercivity cannot be explained by the influence of the magnetocrystalline anisotropy. Anisotropy function ψ, which has been calculated by (3) using the corresponding volume content of these orientations, shows a maximum in the RD and TD, and at 45° to the RD has minimum. Similar results are obtained for the sheet after transverse rolling. This is not consistent with experimental data in Table 3.

The hysteresis loss in the magnetic field during rotation of the single crystal silicon iron with a silicon content of about 4% with the surface oriented in the (001) plane were investigated in earlier work [8]. Anisotropy of hysteresis losses was practically absent until about 0.71 magnetization from saturation. When the magnetization is higher than of above mentioned, then difference in level of hysteresis losses along the [100] and [110] was approximately 8% from the mean value of the magnetization due to the influence of the demagnetizing field in the sample. When the magnetization is equal approximately to 0.95 from the level of saturation and more, then hysteresis loss are decreased sharply to almost zero. This behaviour of hysteresis losses was explained by irreversible movement of magnetic domain walls and their annihilation at the magnetization level higher than 0.71 from saturation that was confirmed by observations of the dynamics of the domain structure, calculations and measurements. Our measurements of the coercive force have been made, as mentioned above, by means of non destructive method using coercimeter KRM-Ts-KA, which previously magnetizes the sample to saturation. Consequently during the magnetization of samples with texture when crystallographic planes {001} lie parallel to the plane of the sheet may be similar changes of the domain structure.

Therefore, considering the above, it can be assumed that anisotropy of coercivity in sheets with texture, when in the plane of the sheet is located family of crystallographic planes {001}, is caused not by texture, not by dynamics of domain walls or internal stresses after annealing.

Let us recall the influence of shape and grain size on the coercive force. Dependence of the coercivity on the grain size d_g generally takes the form [16]

$$H_c = \frac{A}{d_ç} + B, \tag{4}$$

where A, B are some numerical coefficients. Similar relationships were found in the later works [17-19].

Let us estimate the influence of the shape anisotropy and grain size on the coercive force in our sheets after rolling and annealing. Plastic deformation by the rolling was conducted with small reduction per pass. The total deformation in thickness after direct rolling was about 40% and 53% after transverse rolling. It is about 0.5 and 0.76 for the direct and transverse rolling, respectively.

Deformation can be regarded as homogeneous in this case. By applying the principle of Taylor - Polanyi [20], we assume that each grain deforms as entire sample as whole. In this case the initial average size equiaxed grains of 22 µm (Figure 4) after direct rolling have taken the form of ellipsoids, elongated in RD and flattened in ND. The average length of grains in RD becomes approximately equal to 55 microns after direct rolling with 0.5 of true relative logarithmic strain.

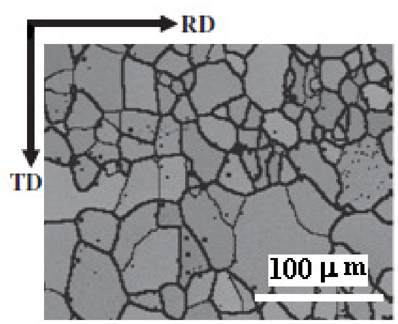

Figure 4. The microstructure of initial sample of alloy Fe – 3 % Si

The average grain size becomes approximately equal 29 µm in direction at 45º to the RD, and average grain size has not changed in TD, i.e. remained equal to 22 µm (Figure 5).

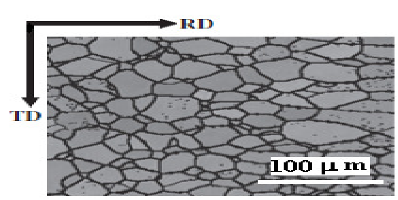

Figure 5. The microstructure of sample of alloy Fe – 3 % Si after direct rolling and subsequent annealing

Grains become even more flattened in the ND after transverse rolling. The average length of grain in a new RD becomes 27 µm, in old RD (i.e. in new TD) becomes approximately 55 µm, and 34 µm in an angle of 45º to the RD (Figure 6).

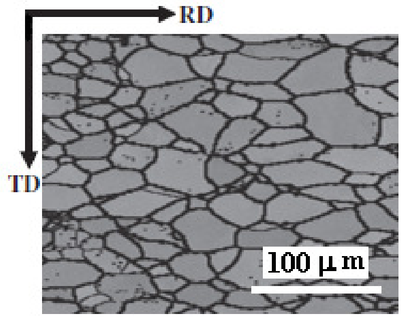

Figure 6. The microstructure of sample of alloy Fe – 3 % Si after transverse rolling and subsequent annealing

The carried out by us correlation analysis for the respective directions in sheets has showed the presence of close linear relationship between the values of H_c and $1/d_g$ in the annealed sheets. Corresponding regression equations have the form

$$H_c = 11{,}09\frac{1}{d_g} + 2{,}20 \text{ For the annealed sheet after direct rolling;} \quad (5)$$

$$H_c = -26{,}53\frac{1}{d_g} + 3{,}38 \text{ For the annealed sheet after cross rolling} \quad (6)$$

Reliability coefficients of approximation R^2 have amounted about 0.99.

Thus, the anisotropy of coercive force can be attributed mainly to the influence of shape and size of grains due to nature of the dynamics of domain structure during the magnetisation in different directions of rolled sheet of alloy Fe - 3% Si with a texture, when the crystallographic planes {001} lie parallel to the rolling plane.

4.CONCLUSIONS

The anisotropy of coercivity was investigated by using of non destructive method both in single crystals with a planes (001) parallel to the surface, so in sheets of alloy Fe-3% Si with different texture.

In single crystals with a surface (001), the maximum coercive force 0.55 A/cm was found in the crystallographic direction [110], which is an axis of middle magnetization. Minimum of coercivity 0.4 A/cm and 0.45 A/cm was observed in the directions [100] and [010], respectively, in accordance with the magnetocrystalline anisotropy.

In the initial sheet of alloy Fe-3% Si, 2.5 mm of thick with texture {011} <100> + {011} <533> the minimum value of the coercive force 2.05 A/cm is observed in the rolling direction.

Maximum of coercivity 2.40 A/cm was found in the transverse direction. Intermediate of its value 2.31 A/cm was found in a direction at an angle of 45º to the rolling direction. Such dependence of the coercive force from the direction in the sheet can be explained by the influence of magnetocrystalline anisotropy. This is confirmed by the presence of a linear correlation with a correlation coefficient of 0.95 between the value of the coercive force and magnetocrystalline anisotropy energy function. Although the influence of the dynamics of domain structure during magnetization in different direction on the character of anisotropy of coercivity not excluded. Texture {001} <110> + {111} <110> formed in the sheet semi finished of alloy Fe – 3 % Si after direct cold rolling to 1.5 mm of thick.

Texture of {001} <110> was formed in a sheet after further cold rolling in a cross direction (the transverse rolling) to a thickness of 1.17 mm.

Anisotropy of coercive force was found in the sheet of alloy Fe – 3 % Si after cold direct and transverse rolling. Character of anisotropy is similar to him in the initial sheet and does not depend on variation textures in the investigated material.

The value of the coercive force decreased about 2 times as a result of annealing for stress relief at 450ºC for 1 hour. Character anisotropy of coercivity has not changed after above mentioned annealing.

The value of the coercive force after cold rolling increased approximately 2 times compared with its value in the original sheet due to the influence of internal stresses due to hardening by rolling. The anisotropy of coercive force maybe attributed to the effect of sizes grain and their shape due to the nature of the dynamics of the domain structure in sheets alloy Fe - 3% Si with the texture, when the crystallographic planes {001} lie parallel to plane of sheets after rolling and annealing.

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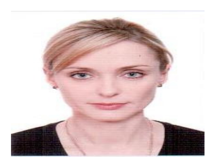
Authors

Shkatulyak Natalia was born 17.05.1956, private- professor of physics of the South Ukrainian National Pedagogical University named after K.D. Ushinsky, Odessa. She holds a PhD in the field of solid state physics, a research interest is new substances and materials, texture, fractal nature of the anisotropy of physical properties of crystalline solids.

Dragomeretskaya Elena was born 12.09.1981, Masters Degree, graduate student of Department of Physics of South Ukrainian National Pedagogical University named after K.D. Ushinsky, Odessa.



Usov Valentin was born 14.01.1950, Dr., Prof., head of the department of technical disciplines of the South Ukrainian National Pedagogical University named after K.D. Ushinsky, Odessa, the author of over 150 publications, a research interest is new substances and materials, texture, fractal nature of the anisotropy of physical properties of crystalline solids.