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ELECTRICAL AND MAGNETIC PROPERTIES

Effect of Parameters of Heat Treatment on Magnetic Properties and Magnetization Distribution in Ribbons of Amorphous Soft Magnetic Iron-Based Alloys

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Abstract—The effect of the isothermal-holding temperature and cooling rate upon in-air heat treatment on the magnetic properties and magnetization distribution in ribbons of amorphous soft magnetic iron-based alloys with positive saturation magnetostriction has been investigated. The results of the investigation showed that the dependence of the maximum magnetic permeability on the isothermal-holding temperature correlates with the corresponding changes in the magnetization distribution in the ribbon and is determined by diffusion processes that occur upon in-air heat treatment at a specific isothermal-holding temperature. An increase in the cooling rate leads to an ambiguous effect on the level of magnetic properties. The increase favors an improvement in magnetic properties when, after in-air heat treatment, either a predominantly amorphous state of the surface or a state with the formed amorphous–crystalline surface layer with a nearly optimal thickness is obtained.

Keywords: magnetic permeability, magnetization, amorphous soft magnetic alloys, heat treatment, cooling rate, isothermal-holding temperature

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INTRODUCTION

It is known that the physical factors that affect the level of magnetic properties of ribbons of amorphous soft magnetic alloys due to in-air heat treatment include a reduction in the level of quenching-induced internal stresses, the interaction of the ribbon surface with atmospheric water vapor, and the formation of the amorphous–crystalline surface layer [1]. Under certain conditions, the last two factors result in the induction of plane pseudo-uniaxial stresses. All the processes occurring upon in-air heat treatment are diffusive. The their activity course is primarily connected with the isothermal-holding temperature [2, 3], and heating and cooling rates are very important parameters. Since the actions of the last two factors on the formation of the level of magnetic properties after in-air heat treatment were studied to a lesser extent and manifest thenselves most pronouncedly at a comparatively low level of internal stresses, in this work, we investigated the effect of the isothermal-holding temperature and cooling rate on the magnetic properties and magnetization distribution in ribbons of amorphous soft magnetic iron-based alloys. Since the activity of diffusion processes increases with a rise in temperature and they take place at all stages of heat treatment (upon heating, isothermal holding, and cooling), variations in the cooling rate favor the understanding of the role of a given mechanisms in the formation of the magnetization distribution and specific level of magnetic characteristics.

The investigations were performed based on the example of ribbons of soft magnetic rapid-quenched alloys $Fe_{77}Ni_1Si_9B_{13}$ and $Fe_{81}B_{13}Si_4C_2$ with positive saturation magnetostriction that possesses a nearly uniform level of magnetic properties and different values of the Curie and crystallization temperatures. This made it possible to vary the degree of activity of diffusion processes in a wider temperature range [3]. The studied samples were in the form of strips with dimensions of 100 × 10 × 0.022 mm. Samples of the amorphous $Fe_{77}Ni_1Si_9B_{13}$ alloy were heat-treated in air at 360–430°C with cooling rates of about 15 and 40 K/min. Since the level of magnetic properties after annealing depends on the magnetization distribution in the initial state of the ribbon [1, 3], for the investigations, we chose samples with a uniform distribution of magnetization in the initial (quenched) state. The magnetization distribution was determined according to the procedure we developed [4]. The magnetization curves and hysteresis loops were measured by the induction–pulse method in closed magnetic circuits. To close the circuits, we used a permeameter. Errors of measuring static magnetic characteristics and magnetization distribution were not higher than 3 and 5%, respectively.

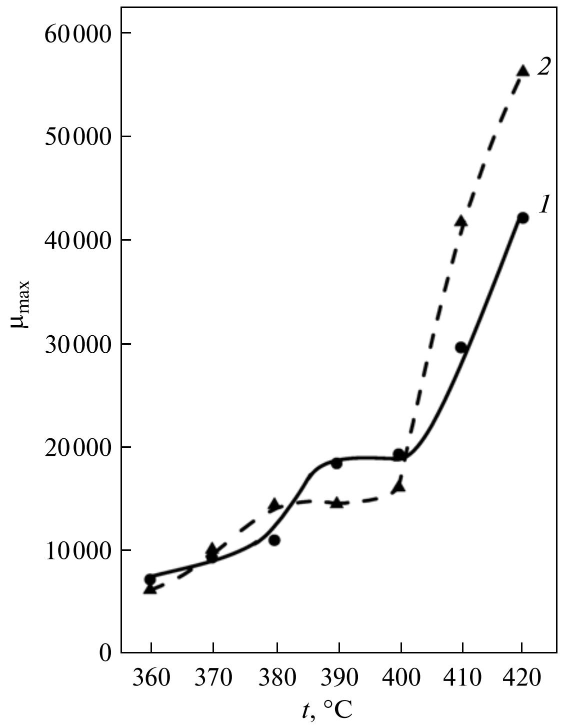

Fig. 1. Dependence of maximum magnetic permeability of samples of amorphous soft magnetic $Fe_{77}Ni_1Si_9B_{13}$ alloy on temperature of in-air heat treatment with an isochronous duration of isothermal holding $\tau = 5$ min and cooling rates 15 and 40 K/min (curves *1* and *2*, respectively).

Figure 1 displays the dependence of the maximum magnetic permeability of samples of the amorphous soft magnetic $Fe_{77}Ni_1Si_9B_{13}$ alloy on the temperature of in-air heat treatment with an isochronous duration of isothermal holding $\tau = 5$ min and cooling rates of 15 and 40 K/min. It can be seen that growth in the isothermal-holding temperature favors an enhancement in the maximum magnetic permeability. Nevertheless, in the dependence of μ_{max} on the isothermal-holding temperature, a plateau is observed in a temperature range of 380–400°C upon cooling with both rates of 15 and 40 K/min. In other words, in this temperature range, the maximum magnetic permeability is nearly independent of isothermal-holding temperature upon in-air heat treatment of ribbon samples of the amorphous soft magnetic alloy of the indicated composition.

Figures 2a, 2c, and 2d show the effect of isothermal-holding temperature during in-air heat treatment with an isochronous duration of isothermal holding $\tau = 5$ min on the magnetization distribution in samples of the amorphous $Fe_{77}Ni_1Si_9B_{13}$ alloy. It can be seen that, in this case, the characteristic violation of the monotony of the dependence of relative volumes of domains with orthogonal V_{ort} and planar magnetization oriented along (V_{180}) and transverse (V_{90}) the ribbon axis on the isothermal-holding temperature is observed. In this temperature range, the dependence of the degree of perfection of the magnetic texture in the ribbon plane characterized by the ratio V_{180}/V_{90} behaves analogously (Fig. 2b). Thus, the dependence of the maximum magnetic permeability on the isothermal-holding temperature correlates with the corresponding change in the magnetization distribution in the ribbon, which in turn is determined by the diffusion processes that occur in the ribbon upon in-air heat treatment at a specific isothermal-holding temperature. It can also be seen from Figs. 1 and 2 that the growth of the cooling rate in different temperature ranges leads to a varying character of the effect of heat treatment on the magnetization distribution and maximum magnetic permeability. For example, due to the enhancement in cooling rate, after the heat treatment at 360°C, the maximum magnetic permeability decreases, whereas after the heat treatment at 420°C μ_{max} increases. These results are within the concepts on the interaction of the ribbon surface with atmospheric water vapor during heat treatment [5]. The vapor treatment at 360°C, as well as that at room temperature, leads to a reduction in the maximum magnetic permeability and an increase in the volume of domains with the planar magnetization oriented transverse to the ribbon axis [5–7]. This takes place because, at 360°C, the resulting magnetization is oriented in the plane of the ribbon along its axis and an enhanced concentration of hydrogen and oxygen atoms embedded into the surface is formed transverse to the ribbon axis, which induces pseudo-uniaxial stress. In our case, at 360°C, a small enhancement in μ_{max} and a decrease in the relative volume of domains with orthogonal magnetization, as compared to the

Table 1. Magnetization distribution and maximum magnetic permeability of ribbon samples of the amorphous soft magnetic $Fe_{77}Ni_1Si_9B_{13}$ alloy in the as-quenched state and after heat treatment with a cooling rate of 15 K/min

State of samples	μ	V_{ort}, %	V_{pl}, %	V_{90}, %	V_{180}, %	V_{180}/V_{90}
Quenched	4100	42	58	20	38	1.9
HT [heat treatment] 420°C, $\tau = 5$ min	42200	11.5	89	21	67	3.2
HT [heat treatment] 430°C, $\tau = 1$ min	41 000	10.1	90	26	64	2.5

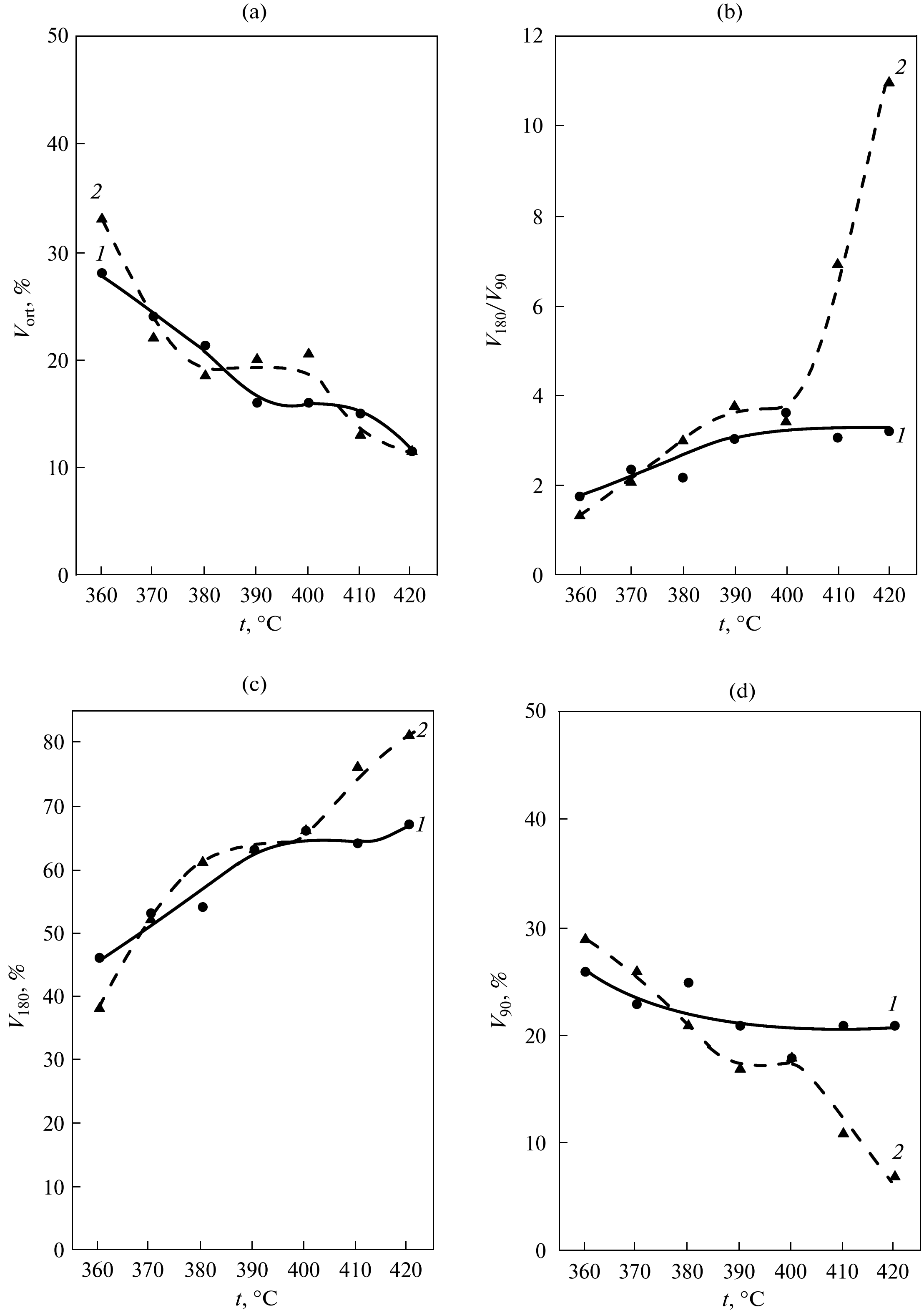

Fig. 2. Dependences of relative volumes of domains with (a) orthogonal V_{ort} and planar V_{pl} magnetization oriented (c) along (V_{180}) and (d) transverse (V_{90}) to the ribbon axis and of (b) the ratio V_{180}/V_{90} characterizing the degree of perfection of the magnetic texture on the temperature of in-air heat treatment with an isochronous duration of isothermal holding $\tau = 5$ min and cooling rates 15 and 40 K/min (curves *1* and *2*, respectively) for samples of amorphous soft magnetic $Fe_{77}Ni_1Si_9B_{13}$ alloy.

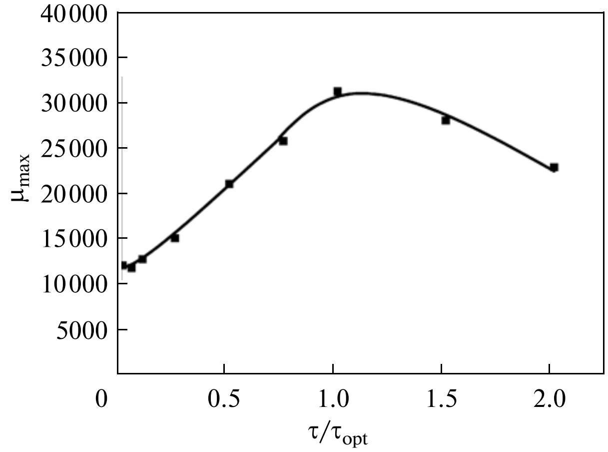

Fig. 3. Dependence of maximum magnetic permeability of samples of amorphous soft magnetic $Fe_{77}Ni_1Si_9B_{13}$ alloy on relative duration of isothermal holding during in-air heat treatment at 410°C with $\tau = 5$ min and a cooling rate of 15 K/min.

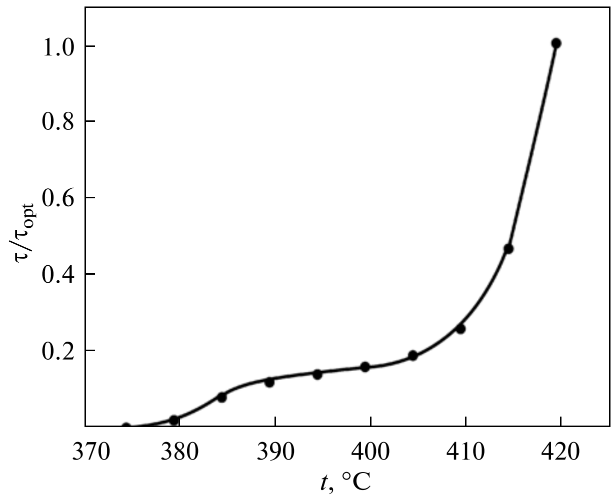

Fig. 4. Dependence of relative duration of isothermal holding at $\tau = 5$ min on temperature of in-air heat treatment for samples of amorphous soft magnetic $Fe_{77}Ni_1Si_9B_{13}$ alloy.

initial state (Table 1), is connected with some reduction in the level of internal quench stresses. Nevertheless, the volume of domains with the planar magnetization oriented transverse to the ribbon axis increases. Because, in this case, a predominantly amorphous state of the ribbon surface is retained, an increase in V_{90} is caused by the pseudo-uniaxial tension transverse to the axis due to the interaction of the surface with atmospheric water vapor.

The enhancement in the cooling rate limits the occurrence of diffusion processes at this stage. Therefore, after heat treatment at 360°C with a cooling rate of 40 K/min, internal quench stresses relax to a lesser extent with the result that higher values of the relative volume of domains with the orthogonal magnetization take place (Fig. 2). The magnetization distribution in the ribbon plane is characterized by a large volume of domains with the planar magnetization oriented transverse to the ribbon axis, a smaller volume of domains whose magnetization is oriented along the axis, and a relatively lesser degree of perfection of the magnetic texture. This can be connected with the retention (transverse to the ribbon axis) of a higher concentration of atoms embedded into the surface due to limitation of the occurrence of diffusion processes at the stage of cooling [5, 8]. The combination of these factors brings about a decrease in μ_{max} with increasing cooling rate.

Because, upon heating, the energy of anisotropy of the shape drops faster than the internal quench stresses relax, at comparatively high temperatures, the resulting magnetization in the ribbon plane is reoriented in the direction of quenching-induced tensile stresses, i.e., transverse to the ribbon axis. This favors the formation of an enhanced concentration of hydrogen and oxygen atoms embedded in the surface along the ribbon axis. As a result, in this direction, after heat treatment, the additional tension is induced, which in turn causes an increase in the degree of perfection of the magnetic texture in the ribbon plane and is the factor responsible for improving the magnetic properties (Figs. 1, 2; Table 1). In this case, the increase in cooling rate makes it possible to retain a higher concentration of atoms embedded in the surface of the ribbon along its axis and, hence, to enhance the level of tensile stresses in this direction, which provides a means of additionally improving magnetic properties, e.g., at 420°C.

An analysis of the behavior of curve *1* presented in Fig. 1 shows that, in a temperature range of 360–380°C, the maximum magnetic permeability grows relatively weakly with a rise in temperature. Based on Fig. 3, which displays a dependence of the maximum magnetic permeability of samples of the amorphous soft magnetic $Fe_{77}Ni_1Si_9B_{13}$ alloy on the relative duration of isothermal holding τ/τ_{opt} during in-air heat treatment at 410°C with $\tau = 5$ min and a cooling rate of 15 K/min, it can be seen that μ_{max} depends slightly on the relative duration of isothermal holding down to $\frac{\tau}{\tau_{опт}} \approx 0.1$. Figure 4 depicts the dependence of the relative duration of isothermal holding at $\tau = 5$ min on the temperature of in-air heat treatment for samples of the amorphous soft magnetic $Fe_{77}Ni_1Si_9B_{13}$ alloy calculated using the empirical formulas

$$\alpha/\alpha_0 = 1290.2x^3 - 2903x^2 + 2177.7x - 544.51$$
$$\text{and} \quad \tau_{opt} = (V_{ort}/\alpha)^3,$$

where $x = t_{HT}/t_{cryst}$, V_{ort} are the values of the relative volume of domains with the orthogonal magnetization in the quenched state, α is the coefficient determining the degree of activity of diffusion processes at a given temperature, and α_0 is the coefficient introduced for adjusting dimensions and is equal to 1 $min^{-1/3}$ (this value of α is obtained at $x = 0.836$) [3]. It can be seen that, in the temperature range of 360–380°C, at $\alpha < 0.07$, the upper boundary of the range corresponds to a ratio of the temperature of heat treatment to the crystallization temperature ~0.72 and the ratio τ/τ_{opt} is less than 0.1 (Fig. 5). Consequently, it can be considered that, after heat treatment in this temperature range with a duration of isothermal holding $\tau = 5$ min, a predominantly amorphous state of the ribbon surface is retained.

It can also be seen from Fig. 1 that, in a temperature range of 370–380°C, growth in the cooling rate leads to an enhancement rather than a decrease in the maximum magnetic permeability. In this case, some reduction is observed in the volume of domains with orthogonal magnetization. This can be a consequence of the action of plain tensile stresses caused by the entry of hydrogen and oxygen into the surface layer of the ribbon [8, 9] against a background of a higher degree of relaxation of internal quench stresses, since the limitation of diffusion processes at the stage of cooling favors the retention of an enhanced concentration of atoms embedded into the surface. In addition, the increased cooling rate allows one to retain a larger difference between concentrations of atoms embedded into the ribbon surface along and transverse to the axis. In this instance, along the ribbon axis, plain pseudo-uniaxial tensile stresses of a higher level are induced. As a result, the relative volume of domains with the planar magnetization oriented transverse to the ribbon axis becomes smaller, the volume of domains with magnetization directed along the axis increases, and the degree of perfection of the magnetic texture in the ribbon plane grows (Fig. 2). Thus, based on the results presented in Figs. 1 and 2, it follows that the temperature at which the process of magnetization reorientation begins during heating is ~370°C when the ratio of the temperature of heat treatment to the Curie temperature is ~0.9 and the spontaneous magnetization is $I = 0.5I_0$.

In the range of 380–400°C, the maximum magnetic permeability hardly depends on the isothermal-holding temperature. This is connected with a weak temperature change in the degree of activity of diffusion processes (coefficient $\alpha = 0.10$–0.13 $min^{-1/3}$) at these temperatures [3]. In addition, the increased cooling rate brings about a reduction in μ_{max} and is mainly caused by the increase in the relative volume of domains with the orthogonal magnetization as the strongest factor that stabilizes the walls of domains with the planar magnetization. In this case, the magnetization distribution in the ribbon plane is characterized by a smaller volume of domains with magnetization oriented transverse to the ribbon axis and a somewhat higher degree of perfection of the magnetic texture. This change in the maximum magnetic permeability and magnetization distribution in the ribbon plane due to heat treatment in the range of isothermal-holding temperatures 380–400°C is connected with the action of the following factors. In this instance, it appears that, on the surface of the ribbon, a continuous amorphous–crystalline layer is formed with a thickness that is smaller than optimum. Nevertheless, plain tensile stresses induced by this layer in the amorphous matrix result in an additional decrease in the volume of domains with the orthogonal magnetization. In this case, as the limitation of the occurrence of diffusion processes at the stage of cooling reduces the thickness of the amorphous–crystalline layer and, hence, induced plain tensile stresses, a state with comparatively high values of V_{ort} is observed. Due to the formation of an enhanced concentration of hydrogen and oxygen atoms embedded into the surface along the ribbon axis, the rate of crystallization in this direction slows down. As a result, the amorphous–crystalline surface layer induces plain pseudo-uniaxial tensile stresses transverse to the ribbon axis that counteract the pseudo-uniaxial stress caused by the enhanced concentration of atoms embedded in the surface along the axis and reduce the anisotropy of stresses in the ribbon plane. The increase in the cooling rate slows down the decrease in the concentration of atoms embedded into the surface of the ribbon along its axis. In this instance, a state is formed with a higher level of tensile stresses in this direction, which favors a reduction in the volume of domains with the planar magnetization oriented transverse to the ribbon axis and an enhancement in the degree of perfection of the magnetic texture in the ribbon plane.

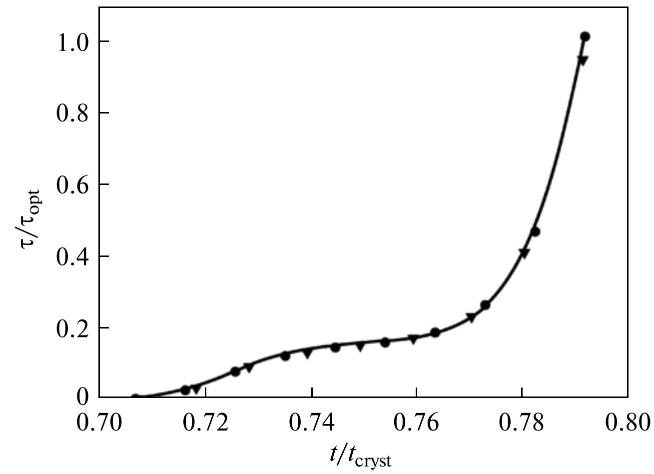

Fig. 5. Dependence of relative duration of isothermal holding at $\tau = 5$ min on the ratio of the temperature of in-air heat treatment to the crystallization temperature for samples of the amorphous soft magnetic $Fe_{77}Ni_1Si_9B_{13}$ and $Fe_{81}B_{13}Si_4C_2$ alloys (designated by points and triangles, respectively).

When the isothermal-holding temperature increases above 400°C, a rather strong activation of diffusion processes takes place. The duration of isothermal holding $\tau = 5$ min at 420°C becomes optimal,

Table 2. Effect of cooling rate upon in-air heat treatment on the magnetization distribution in samples of the amorphous soft magnetic $Fe_{77}Ni_1Si_9B_{13}$ and $Fe_{81}B_{13}Si_4C_2$ alloys

Alloy	t_{an}, °C	τ, min	V_{cool}, K/min	V_{ort}, %	V_{pl}, %	V_{90}, %	V_{180}, %	α	t/t_{cryst}	τ/τ_{opt}
$Fe_{81}B_{13}Si_4C_2$	360	2	15	20.0	80	28.1	52	0.13	0.75	0.07
			40	20.5	80	19.2	60			
$Fe_{81}B_{13}Si_4C_2$	350	5	15	20.0	80	23.2	57	0.11	0.73	0.10
			40	20.0	80	19.0	61			
$Fe_{77}Ni_1Si_9B_{13}$	380	5	15	21.3	79	25.0	54	0.07	0.72	0.08
			40	18.5	82	21.0	61			

which corresponds to the formation of the amorphous–crystalline surface layer of the optimum thickness. In the temperature range 400–420°C, the growth of the maximum magnetic permeability after the in-air heat treatment with a cooling rate of 15 K/min is practically caused by a reduction in the degree of stabilization of walls of domains with the planar magnetization produced by domains with the orthogonal magnetization due to the corresponding decrease in V_{ort}, which results from the induction of plain tensile stresses due to the interaction of the ribbon surface with atmospheric water vapor and the surface crystallization of the ribbon. Nevertheless, the magnetization distribution in the ribbon plane after the heat treatment with isothermal holding in this temperature range is nearly uniform and is connected with the manifestation of two competitive factors. The pseudo-uniaxial stress along the ribbon axis caused by the formation of an enhanced concentration of hydrogen and oxygen atoms is compensated for by the corresponding tension in the transverse direction due to an anisotropic partial crystallization of the ribbon surface. In addition, the concentration of atoms embedded along the ribbon axis drops at the stage of cooling, which likewise entails some reduction in the level of tensile stresses in this direction. The enhancement in the cooling rate brings about an increase in the maximum magnetic permeability due to the magnetization redistribution in the ribbon plane, since the limitation of the occurrence of diffusion processes retards the reduction in the concentration of atoms embedded along the ribbon axis and favors the formation of a state with a higher level of tensile stresses in this direction. As a result, a decrease in the volume of domains with the planar magnetization oriented transverse to the ribbon axis, an enhancement in the volume of domains whose magnetization is directed along the axis, and an increase in the degree of perfection of the magnetic texture in the ribbon plane are observed.

For comparison, Table 1 contains the results on the effect of heat treatment on the maximum magnetic permeability and magnetization distribution in $Fe_{77}Ni_1Si_9B_{13}$ samples after the in-air heat treatment at 430°C with an optimum duration of isothermal holding 1 min and a cooling rate of 15 K/min. Comparison with the heat treatment at 420°C with an optimum duration of isothermal holding 5 min shows, that in this case, despite the obtained state with close values of maximum magnetic permeability, the magnetization distribution in the samples is markedly different. Despite the lower values of the relative volume of domains with the orthogonal magnetization, the state of the ribbon is not distinguished by higher values of maximum magnetic permeability. The reason for this is that, in the ribbon plane, the magnetization distribution is characterized by higher values of the volume of domains with the planar magnetization oriented transverse to the ribbon axis. Since in this instance heat treatment was performed at the isothermal-holding temperature above the Curie point and a high degree of activity of diffusion processes ($t/t_{cryst} = 0.81$ and $\alpha = 0.45$ min$^{-1/3}$), at 430°C, no considerable anisotropy of the concentration of hydrogen and oxygen atoms embedded into the ribbon surface is formed, which leads to the induction of predominantly plain tensile stresses. In addition, during cooling below the Curie point, the energy of anisotropy of the shape favors the formation of a state with the resulting magnetization oriented along the ribbon axis. For this reason, the concentration of atoms embedded into the ribbon surface may arise transverse to the ribbon axis, which results in a pseudo-uniaxial stress in this direction and growth of the volume of domains with planar magnetization oriented transverse to this axis.

Figure 6 and Table 2 contain comparative data on the effect of the cooling rate upon the in-air heat treatment on the maximum magnetic permeability and magnetization distribution in samples of the $Fe_{81}B_{13}Si_4C_2$ alloy, which are identical in properties for

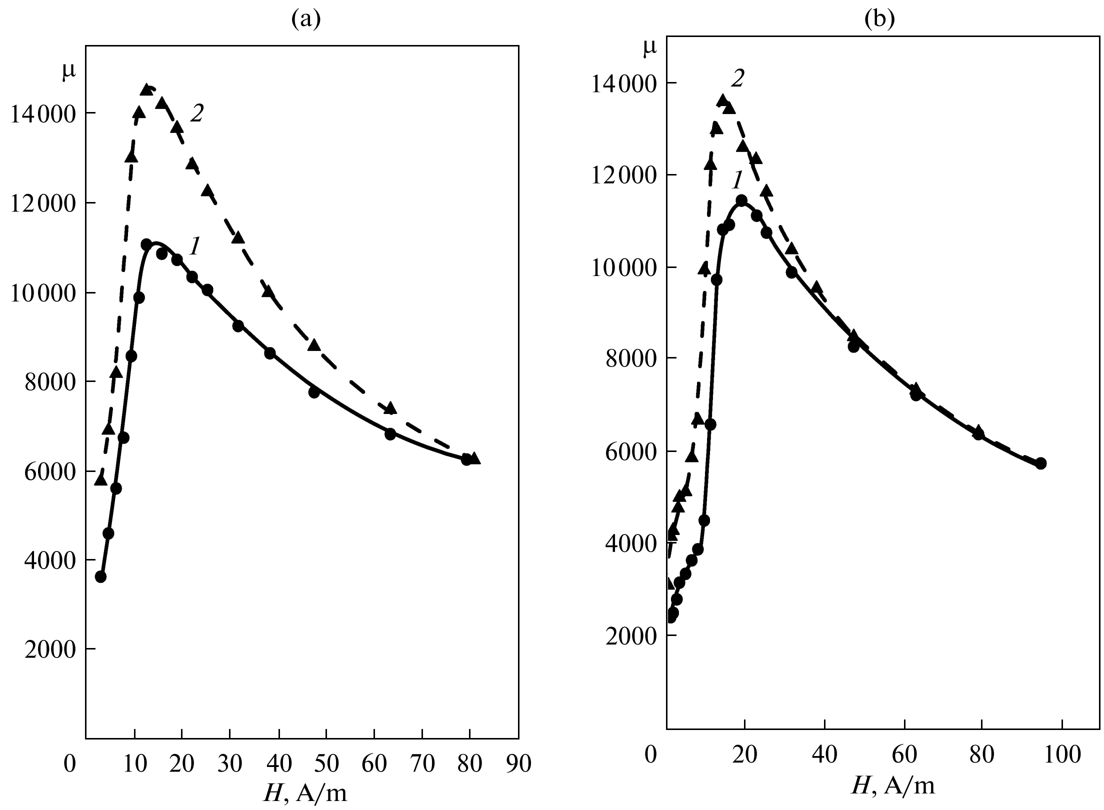

Fig. 6. Field dependences of magnetic permeability for samples of the amorphous soft magnetic $Fe_{77}Ni_1Si_9B_{13}$ and $Fe_{81}B_{13}Si_4C_2$ alloys after in-air heat treatment at (a) 380 and (b) 350°C with an isochronous duration of isothermal holding $\tau = 5$ min and cooling rates 15 and 40 K/min (curves *1* and *2*, respectively).

which the correlation dependence between τ/τ_{opt} and t/t_{cryst} is described by the same curve (Fig. 5). It can be seen that, under almost identical conditions of annealing (in which the predominantly amorphous state of the ribbon surface is obtained) and at a relatively low degree of activity of diffusion processes ($\alpha =$ 0.07–0.13 min$^{-1/3}$) when t/t_{cryst} = 0.72–0.75 and $\tau/\tau_{opt} < 0.1$), the increased cooling rate leads to an increase in the relative volume of domains with magnetization oriented along the ribbon axis due to decreasing values of V_{90}. This causes the growth of the maximum magnetic permeability.

CONCLUSIONS

The investigation of the effect of the cooling rate and isothermal-holding temperature during the in-air heat treatment of ribbons of amorphous soft magnetic alloys yielded the following conclusions:

(1) The dependence of the maximum magnetic permeability on the isothermal-holding temperature correlates with the corresponding change in magnetization distribution in the ribbon, which is in turn determined by diffusion processes that occur upon the in-air heat treatment with a specific isothermal-holding temperature.

(2) The enhancement in cooling rate results in an ambiguous effect on the level of magnetic properties and magnetization distribution in ribbons of amorphous soft magnetic iron-based alloys and favors the improvement of magnetic properties in the case when, after in-air heat treatment, either the predominantly amorphous state of the ribbon surface or a state with the formed amorphous–crystalline surface layer is obtained, the thickness of which is close to the optimum.

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