STUDY OF THE EFFECT OF THERMOMAGNETIC TREATMENT ON THE STABILITY OF THE MAGNETIC PROPERTIES OF AN Fe—Co ALLOY

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Results are presented from an experimental study of the stability of the magnetic properties of an iron—cobalt alloy with different regimes of thermomagnetic treatment.

Fe—Co alloys with different amounts of the basic components and V and Mn alloying additions have a high saturation and are used in instrument manufacture, radio electronics, computer technology, and the manufacture of the pole pieces of high-resolution NMR radio-frequency spectrometers [1, 2]. The magnetic properties of these alloys depend on their structure and method of treatment [3, 4]. Several difficulties caused by the stringent requirements for uniformity of the properties of the polar material have to be overcome in making high-resolution NMR RF spectrometers.

As already noted in [5, 6], one source of nonuniformity of the magnetic field, limiting the resolution of the instruments, is the macrocrystalline structure of the pole-piece material. To obtain a fine-grained structure in pole material made of iron—cobalt alloys [6], it was recommended in [6] that these alloys be further alloyed with other elements. It was shown that different treatment methods for Fe—Co alloys could improve their magnetic properties so as to ensure a uniform magnetic field and resolution in RF spectrometers. However, to further increase resolution, the field not only has to be uniform, but stable over time [7].

The present work studies the effect of thermomagnetic treatment on the stability of the magnetic properties of an Fe—Co alloy of the Permendur type with a 2% alloying addition of vanadium.

The stability of the magnetic properties was investigated on specimens of the Fe—Co—2V alloy (49% Fe, 49% Co, 2% V) subjected to various regimes of thermal and magnetic treatment. To ensure uniform magnetization over the entire volume, the specimens had the form of an ellipsoid of revolution with a semiaxis ratio equal to 10. It was shown in [4] that thermal and mechanical treatment affect the magnetic properties and microinhomogeneity of the Fe—Co—2V alloy. It was also indicated that minimum internal stresses — hence a minimum density of microscopic defects — are obtained in the deformed alloy with an annealing temperature close to 820°C. This is recommended as the optimum temperature for heat treatment of the alloy to improve its properties. The studies in [3] explained the effect of the magnetic field during heat treatment on the magnetic properties of the iron—cobalt alloy.

However, the above works did not study the stability of the properties of this alloy over time after different methods of its treatment. All types of effects on the alloy — mechanical, thermal, magnetic, etc. — lead to changes in its structure and, thus, its properties. The structure reaches a stable state after a certain period of time, rather than immediately. The character of the transition from a metastable thermodynamic state to a more equilibrium state is different under different conditions and depends on the heat-treatment temperature, the time the material is kept in a given temperature regime, the heating and cooling rate, prior strain, and other factors.

We studied the effect of annealing temperature and simultaneous thermal and magnetic treatments on the stability of the magnetic properties of specimens of the Fe—Co—2V alloy over time. The specimens were annealed in a vacuum, with almost no oxidation of the surface having occurred.

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The 600, 740, 820, and 980°C annealing temperatures used covered all characteristic regions of the phase diagram for an Fe—Co alloy [1, 2].

According to the diagram, the α-phase goes from an ordered state (α') to a disordered state (α). A magnetic transformation with a transition from the α-phase to the γ-phase occurs with further heating at 980°C.

Considering the results in [8], in our experiments the specimens were heated to the above annealing temperatures at a fixed rate $v = 200°C/h$, subsequently held one hour at the annealing temperature, and finally cooled with the furnace at a rate $v = 100°C/h$. This heat-treatment regime was maintained for all of the specimens and all of the investigated annealing temperatures.

Together with the conventional annealing at the above tempertures, we conducted a thermomagnetic anneal using a specicl unit illustrated in Fig. 1. The specimen 1 was placed in a quartz tube 3 in which a sufficient vacuum could be maintained during testing. The specimens were heated using a heating element 2 with a bifilar winding, which compensated for the magnetic field created by the current in the element.

A molybdenum shield 4 was installed to create high temperatures on the inside surface of the quartz tube. The specimen temperature was continuously recorded with a thermocouple 6 in contact with its ruface. A magnetic field was created during annealing by using the solenoid 5. The length of the solenoid was such that the magnetic field in which the specimen was located was nearly uniform. The temperature regime of the solenoid was maintained by cooling the latter with flowing water passed through a copper coil wound closely against the coil form. Special guides ensured the same vertical and coaxial location of the major axis of each specimen relative to both the vertical of the unit as a whole and the direction of the external magnetizing field.

Time changes in the magnetic properties of specimens annealed with and without a superimposed magnetic field were studied on a special magnetometric unit (MU) [9].

Experimental curves $I = f(t)$ characterizing the change in the magnetic properties of the Fe—Co—2V specimens over time as a function of different heat-treatment regimes are shown in Fig. 2. The properties change more rapidly in the initial time intervals, during the initial stage of stabilization of the process. The rate of change then decreases and the properties become nearly constant. Analysis of these results also shows that stabilization during aging begins at different times for different annealing temperatures and, furthermore, depends on the application of a magnetic field during the heat treatment. These times were roughly as follows for the investigated annealing temperatures of 600, 740, and 820°C without the magnetic field (Fig. 2a, b, c-I): $t_{600} \approx 120$ h, $t_{740} \approx 90$ h, $t_{820} \approx 75$ h. The stabilization times for these temperatures with a magnetic field with $H = 3980$ A/m (Fig. 2a, b, c-II) were $t_{600} \approx 90$ h, $t_{740} \approx 60$, $t_{820} \approx 50$ h. These tests showed that the time of stabilization of the process decreases with an increase in annealing temperature. This decrease is also seen in the case of thermomagnetic annealing, in which the magnetic field accelerates the stabilization process at the prescribed temperature. Stabilization time is reduced by 30% with thermomagnetic annealing compared to annealing without a magnetic field.

After elapse of the above stabilization periods, no appreciable change in magnetic properties is seen even after a long observation (more than 700 h).

Similar curves of magnetic property change during aging were obtained for specimens of the Fe—Co—2V alloy for temperatures above 820°C and for intermediate temperatures. The general law for all of the specimens is a reduction in stabilization time during aging with an increase in annealing temperature. Annealing with a superimposed magnetic field speeds up the stabilization process, shortening the time of transition from the metastable state to the equilibrium state.

Let us examine possible physical explanations for the experimental results. Instability of the magnetic properties of a ferromagnet over time are due to a change in its domain or crystal structure. The changes attributable to the domain structure are reversible in nature, while restructurings of the crystalline lattice are irreversible. The irreversible character of the instability of the magnetic properties of an electromagnet is due to the transition of the crystalline structure from a metastable thermodynamic state to a more equilibrium state. As a rule, this transition may also be accompanied by changes in chemical composition, the degree of dispersion of the phases and their distribution, and the density of dislocations and microdefects. X-ray [10] and electron microscope [11] studies of an iron—cobalt alloy

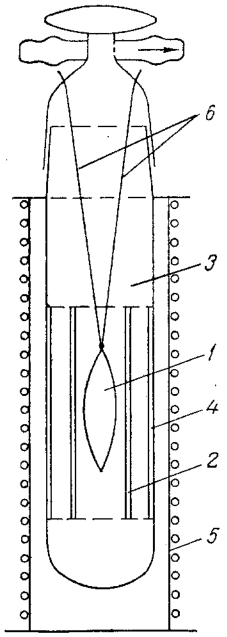

Fig. 1. Experimental unit.

with a vanadium addition have shown that a predeformed specimen annealed at 400-600°C experiences a redistribution of its vanadium at sites of dislocations and other defects, leading to the formation of vanadium clusters. This in turn leads to a reduction in the lattice parameter of the alloy and a change in the concentration of vanadium in the defect-free region. The change in the physical properties of the alloy in this case should be attributed to its ordering, accompanied by the formation of vanadium clusters. Annealing at 600°C, corresponding to the region of the ordered α'-phase, lowers internal stresses in deformed specimens along with the density of dislocations and microdefects, which in turn ensures a certain level of stabilization of the crystalline structure. However, complete stabilization of the crystalline structure for this annealing temperature begins after a certain period of time. It is apparent from Fig. 2, curve *a*-I, that the time for stabilization of the magnetic properties of the specimens is more than 120 h. Irreversible processes, causing the time change in magnetic properties, continue to occur in the crystalline structure during this time. This instability is connected with the formation of vanadium clusters and their equilibrium redistribution, accompanied by slow relaxation processes associated with the aging and elastic lag in individual microvolumes. Thermomagnetic annealing at this temperature with the superposition of a magnetic field with H = 3980 A/m speeds up the transition from the metastable state to the stable equilibrium state, shortening the stabilization time from 120 to 90 h. The magnetic field causes all of the irreversible processes which occur in the crystalline lattice and lead to its stabilization to take place more quickly.

An increase in annealing temperature (at 740°C — region of disordered α-phase) is accompanied by a more rapid reduction in internal stresses and dislocation and microdefect density, which in turn affects stabilization time. It can be seen from Fig. 2, b-I, that the stabilization time for an annealing temperature of 740°C, without a magnetic field, is less than the stabilization time for an annealing temperature of 600°C (Fig. 2, *a*-I). The times here are 90 and 120 h, respectively. With the application of a magnetic field at these annealing temperatures (Fig. 2, *a*-II, b-II), stabilization time also tends to decrease and amounts to 90 and 60 h, respectively. A further increase in annealing temperature leads to a reduction in stabilization time for both the disordered α-phase and for the transitional $\alpha \rightleftarrows \gamma$ phase and γ-phase (Fig. 2, c-I and c-II).

Thus, it follows from the results obtained that stabilization of the crystalline structure and, thus, the magnetic properties of the Fe—Co—2V alloy specimens depends on the regimes of thermal and thermomagnetic treatment. An increase in annealing temperature and the application of a magnetic field during annealing both accelerate artificial aging and promote the transition of the crystalline structure of a ferromagnet from the metastable state to a more equilibrium state. The time required for stabilization of the magnetic properties of specimens of the Fe—Co—2V alloy is shortened by 30% by the application of a magnetic field

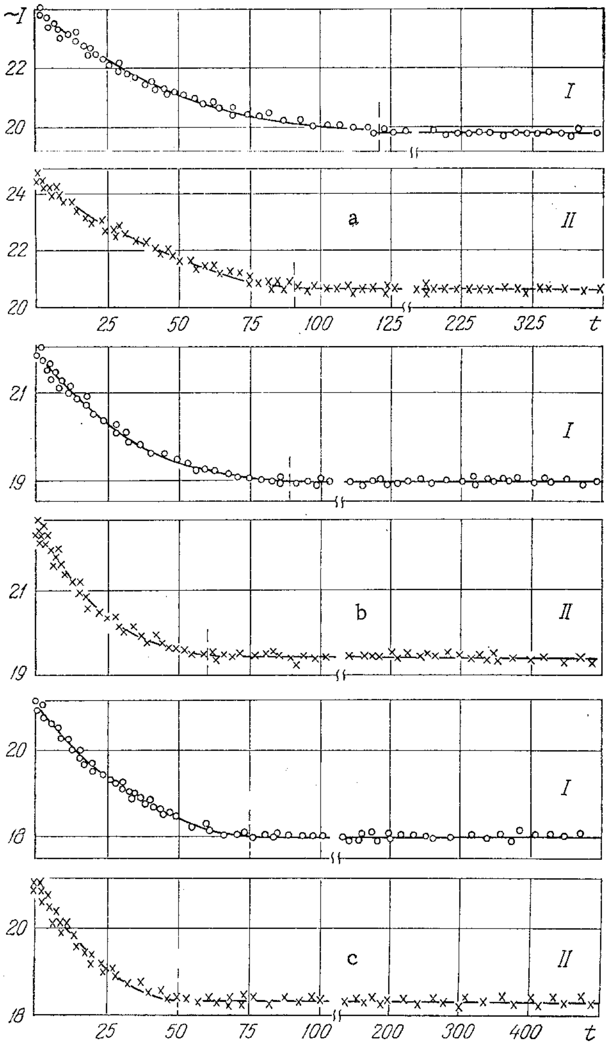

Fig. 2. Curves of change in magnetic properties of specimens of the Fe—Co—2V alloy over time for annealing temperatures T = 600 (a), 740 (b), and 820°C (c): I) without magnetic field; II) with superposition of magnetic field with H = 3980 A/m. t, h.

during annealing compared to the time required without the field. The results obtained will be not only of theoretical interest, from the point of view of studying magnetic relaxation phenomena in ferromagnets, but also of practical interest — in connection with the broad use of these alloys in different areas of technology.

NOTATION

t, time; T, temperature; I, magnetization; H, strength of magnetizing field; α, α', γ, notations for phases on phase diagram; v, heating and cooling rates.

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STUDYING THE THERMOPHYSICAL CHARACTERISTICS OF NONMETALLIC COATINGS

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A method is described for experimental determination of the thermophysical characteristics of nonmetallic coatings. Experimental data for one such coating is presented.

The wide use of nonmetallic coatings in different areas of technology to protect metal from corrosion, intensify radiant heat transfer, and provide thermal insulation confronts researchers with the task of measuring the thermophysical properties of these coatings. The methodological aspects of this problem are traceable to two basic difficulties. First, obtaining measurements within the broad range of working pressures and temperatures of the coatings is fairly complex in itself. Second, most well-known methods generally require the measurement of temperature at two points. Of course, such measurements cannot be obtained in thin coatings, since the thermal resistance of the coating in this case is comparable with the thermal resistance of the contact of the temperature transducer. This produces a substantial measurement error [1].

In connection with this, we believe nonsteady methods are promising. Such methods make it possible to obtain all necessary data with regard to temperature measurement at a single point on the specimen (the temperature of the metallic substrate).

We propose a variant of the method in [2] that will more accurately determine the thermal conductivity, diffusivity, and volume specific heat of thin coatings applied to a metal substrate with a known volume specific heat.

In the heating of the surface of a coating and the thermally insulated surface of a metal substrate by a constant heat flux with the condition $\lambda_m > \lambda$, the change in substrate temperature is described in dimensionless form by the following relation [3]

$$\frac{\theta_m(\mathrm{Fo})}{\mathrm{Ki}} = \frac{K\,\mathrm{Fo}}{1+K} - \frac{K(3+K)}{6(1+K)^2} - 2\sum_{n=1}^{\infty}\frac{(\mu_n^2+K^2)\cos\mu_n\exp(-\mu_n^2\mathrm{Fo})}{\mu_n^2(\mu_n^2+K^2+K)}, \tag{1}$$

where μ_n are the roots of the characteristic equation $\tan\mu_n = -\mu_n/K$.

When $\mathrm{Fo} > 1.5$, quasisteady heating begins and the series in Eq. (1) can be ignored [3]. In this case, Eq. (1) is written in dimensionless form as

$$\vartheta_m(\tau) = \frac{q\tau}{c\rho l + c\rho l_m} - \frac{qlK(3+K)}{6\lambda(1+K)^2}, \tag{2}$$

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