INFLUENCE OF THE ANNEALING TEMPERATURE AND HOLDING TIME ON THE STABILITY OF MAGNETIC PROPERTIES OF A Fe-Co ALLOY

P. P. Galenko

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Experimental investigations are presented for the stability of the magnetic properties of an iron-cobalt alloy for different annealing temperatures and holding times.

To obtain highly homogeneous magnetic fields in gaps of high-resolution NMR radio spectrometer electromagnetics, high requirements are imposed on the pole shoe material properties [1]. These materials should possess high saturation induction and stability of the magnetic properties during exploitation.

Iron-cobalt alloys of the permendure type possess high saturation induction and are applied extensively in individual areas of instrumentation for the fabrication of magnetic lenses for electron microscopes, radio electronics, computer engineering, as well as for the fabrication of high-resolution NMR radio spectrometer pole shoes [1, 2]. The magnetic properties of an Fe-Co alloy with equal quantities of the components with the alloying admixtures V, Mn depend on the structure and treatment methods [3-6]. It is shown in [7, 8] that the source of magnetic field inhomogeneity that limits the resolution of the devices is the coarse-crystalline structure of the material. To obtain a fine-grain structure in iron-cobalt alloys it is recommended that alloying admixtures be inserted [8].

Investigations [3-7] showed the influence of plastic deformation, thermal, thermomagnetic, and mechanical treatment conditions on the magnetic properties and microinhomogeneity of the Fe-Co alloy. It is clarified [4, 5] that a minimal value of the internal stresses and, therefore, of the microdefect density, is achieved in a deformed Fe-Co alloy at an annealing temperature close to 820°C. This temperature is indeed recommended as optimal in heat treatment to improve the magnetic properties of the alloys mentioned. The possibility is also shown for the production of axial texture for specific deformation schemes and the application of textured shoes in NMR radio spectrometers.

It is shown in the papers cited that by using different methods of treating Fe-Co alloy poles, their magnetic properties can be improved and the homogeneity of the magnetic field and the resolution in radio spectrometers can therefore be raised. Despite all efforts, for a further rise in the resolution, the principal obstacle was not so much the field inhomogeneity as its limited stability in time [9]. Hence, a study of the stability of the magnetic properties of Fe-Co alloys in time for different mechanic, thermal, and thermomagnetic treatment methods acquires practical and scientific interest. Results on the stability of the magnetic properties of Fe-Co alloy specimens in time presented in [11] for different annealing temperatures in which all the characteristic phase equilibrium diagram domains are included. These data show that as the annealing temperature rises in the alloy specimens, irreversible processes associated with aging, diffusion, and other phenomena are accelerated because of the diminution in the internal stresses, the dislocation density, and other microdefects, and therefore, the time to stabilize the magnetic properties diminishes.

It is established in [10] by investigations of the influence of thermomagnetic treatment on the stability of the magnetic properties of a Fe-Co alloy that as the annealing temperature rises, artificial aging is accelerated for a simultaneous superposition of the magnetic field during the heat treatment, which contributes to the passage of the crystalline ferromagnet structure from the metastable into a more equilibrium state. The time of magnetic property stabilization for Fe-Co alloy specimens under the effect of a magnetic field during heat treatment is diminished 30% on the average relative to its value for annealing without a field.

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Fig. 1. Curves of the change in the magnetic properties of Fe-Co-2V specimens in time for the annealing temperatures T = 600 (A) and T = 820°C (B): a)  $t_t^1 =$ 1 h; b)  $t_t^2 = 5$  h; c)  $t_t^3 = 10$  h.

This paper is devoted to an investigation of the stability of the magnetic properties of a Fe-Co alloy of permendure type with a 2% alloying admixture of vanadium as a function of different annealing regimes and holding time durations at a given temperature. The stability of the magnetic properties was studied in Fe-Co-2V alloy specimens (Fe - 49%, Co -49%, V = 2%) in the shape of ellipsoids of revolution with a semiaxis ratio of 10. Such a shape assured homogeneous magnetization over the whole specimen. The specimens were annealed by the method developed in [10, 11]. The annealing temperature range 600, 740, 820, 980°C included all the characteristic domains of the phase equilibrium diagram ( $\alpha$ '-phase of the ordered state,  $\alpha$  - the disordered state, and the  $\gamma$ -phase) of the Fe-Co alloy system [1, 2]. Taking account of the data in [10-12], the specimens in our tests were heated to the annealing temperatures mentioned at a given rate  $v_h$  = 200°C/h and then cooled with the furnace at the rate  $v_c = 100^{\circ}C/h$ . The holding duration time for a given annealing temperature was 1 h for all the specimens investigated in [10, 11]. To clarify the influence of the holding time (tt) at a given annealing temperature on the stability of the magnetic properties of the specimens, we took  $t_t^i = 1$ ,  $t_t^2 = 5$ , and  $t_t^3 = 10$  h in the tests. One heating and cooling mode was here conserved strictly for all the specimens and all the annealing temperatures under consideration.

The time changes in the magnetic properties of specimens subjected to different annealing modes were determined continuously by using a special magnetometer apparatus [10, 11]. The results of the experimental investigations characterizing the change in the magnetic properties of Fe-Co-2V alloy specimens in time as a function of the annealing temperature and the holding time are represented as the curves I = f(t) in figures A and B. It is seen from an analysis of these curves that a more intensive change in the magnetic properties of the specimens proceeds in the initial stage of process stabilization in the first time intervals, and its velocity later diminishes after a certain time and the change in their magnetic properties becomes practically negligible. Such a nature of the stabilization process was observed for all annealing temperatures, and as has been shown in [10], the experimental curves can be described by the equation

$$I = I_0 e^{-\varkappa t} + I_\infty (1 - e^{-\varkappa t}), \tag{1}$$

where  $I_{\infty}$  is the steady value of specimen magnetization after the passage of its crystal structure from the metastable into the more equilibrium state.

Experience shows that the time of stabilization onset for the specimen magnetic properties is distinct for different annealing temperatures and, moreover, depends on the holding duration during heat treatment. For the specimens under investigation which have been annealed at a 600°C temperature with subsequent 1, 5, and 10 h holdings, the process stabilization time takes the respective values  $t_{st600}^1 = 125$ ,  $t_{st600}^5 = 90$ ,  $t_{st600}^2 = 75$  h (see Fig. A). These results show that as the annealing duration increases for a given temperature the aging process accelerates and therefore, the stabilization time diminishes for the specimen magnetic properties. Experimental results for the 820°C annealing temperature also confirm the diminution in the stabilization time for the specimen magnetic properties as the annealing duration increases:  $t_{st620}^2 = 75-80$ ,  $t_{st620}^2 = 60$ ,  $t_{st620}^2 = 40-45$  h (see Fig. B).

An analogous behavior for the curves of the change in magnetic properties is obtained for stabilization of the structure of Fe-Co-2V alloy specimens for both temperatures above 820°C and for their intermediate values with a different annealing duration.

A general regularity for all the specimens under investigation in the diminution in structure stabilization time during aging for both a rise in the annealing temperature and an increase in annealing duration. For holdings from 1 to 10 h at a given annealing temperature, the structure stabilization process is accelerated, and the time of passage from the metastable to the equilibrium states diminishes 45-50% on the average. The longevity of the annealing duration at the temperature under investigation, exactly the same as the imposition of the magnetic field during treatment [10], contributes to the progress of irreversible processes in the crystalline lattice for Fe-CO-2V alloy specimens, resulting in stabilization. This passage of the ferromagnet crystalline structure from the metastable thermodynamic state to a more equilibrium state is due to the irreversible nature of the time instability of its magnetic properties, and as a rule, changes occurring in the chemical constitution, the degree of phase dispersion, and their redistribution, the dislocation density, the values of the internal stresses and other microdefects can here by observed. As x-ray [14] and electron microscope [15] investigations of an iron-cobalt alloy with a vanadium admixture showed, vanadium redistribution at the sites of the dislocations and other defects, resulting in the formation of vanadium clusters, occurs in predeformed specimens at 400-600°C annealing temperatures. Consequently, the vanadium concentration in the defect-free zone changes and the alloy lattice parameter diminishes. Hence, the change in the alloy physical properties during stabilization of the crystalline structure can be explained, for the temperature range mentioned, by its ordering which is accompanied by the formation of vanadium clusters and their equilibrium redistribution with the slow relaxation processes associated with aging and elastic aftereffect in individual microvolumes.

At the 820°C annealing temperature corresponding to the disordered  $\alpha$ -phase domain, a more intensive diminution of the internal stresses, dislocation density and other microdefect density occurs in specimens of the alloy under investigation, which affects the magnitude of the time for stabilization of their magnetic properties. The magnitude of the stabilization time for the magnetic properties can diminish as a function of the rise in the annealing temperature and its duration. A further rise in the annealing temperature for both the disordered  $\alpha$ -phase domain and the transitional  $\alpha \neq \gamma$ -phase and  $\gamma$ -phase will result in a diminution in the time for stabilization of the magnetic properties. This tendency to diminish holds even as a function of the duration of the annealing. Therefore, a rise in the annealing temperature and its duration will accelerate natural aging and contribute to the transition of the ferromagnet crystalline structure from the metastable to the more equilibrium state.

The results of the investigation obtained are of practical interest in connection with the broad application of these alloys in different areas of instrumentation as well as theoretically from the viewpoint of investigating magnetic relaxation phenomena and revrse magnetization processes in ferromagnets.

## NOTATION

t, time; T, temperature; I, magnetization;  $\varkappa$ , a parameter dependent on the annealing temperature and the properties of the ferromagnet; v, heating and cooling rate; and  $\alpha$ ,  $\alpha'$ ,  $\gamma$ , phase notations in the phase diagram.

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