QUESTION OF THE MECHANISM OF FORMATION OF THE DAMPING CONDITION OF HIGH-CHROMIUM FERRITIC STEELS

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The question of the influence of internal stresses of different origin on magnetomechanical damping in high-damping ferritic alloys was widely discussed in the International Conference on Questions of Internal Friction in Solids (ECIFUAS-6). The participants in the discussion presented the results of their investigations of influence of the degree of preliminary deformation, static tensile stresses, the number of cycles of vibrations, and other characteristics on the parameters of inelastic dispersion of energy. The investigation of the problem is continued in this article. Primary attention is devoted to discussion of the influence of crystal line lattice defects and stresses created by them on the damping capacity of Fe–Cr alloys.

High-chromium ferritic steels are of interest as the result of the good combination of mechanical properties and corrosion resistance and also the capacity to reach high levels of internal dispersion of energy [1]. The basic mechanism providing a high level of damping in high-chromium ferritic steels is magnetomechanical dispersion of energy [1-3]. At frequencies of vibrations negligibly small in comparison with the frequency of Brakhausen changes ($f \ll 10^6$ Hz) it is caused by magnetoelastic hysteresis, which is related to irreversible displacements of the boundaries of magnetic domains. The level of dispersion of energy is determined by the resistance of various crystalline structure defects to movement of domain boundaries. The structural defects are sources of internal stresses (σ_i) and local distortions of magnetic anisotropy.

In the majority of cases an increase in internal stresses in alloys reduces the losses to magnetoelastic hysteresis (δ_h) [2] as the result of the decrease in mobility of the domain boundaries. Normally the influence of σ_i is considered without their differentiation by types of sources [4], which, in addition to dislocations, may be interphase boundaries, point defects, accumulations of them, segregations, particles of a second phase, etc. In each case interaction with the domain boundaries occurs according to a certain mechanism taking into consideration the type, concentration, and topography of the defects and the energy of their bond with the domain boundaries.

The rule of totalling of the partial effects of internal friction [3] is normally used for totalling of losses of internal energy in deformation of alloys. For ferritic materials in which additional magnetostrictive deformation occurs together with the elastic, the experimental results may far from always be explained by the additive influence of various sources of losses (structural defects) to the level of hysteresis magnetomechanical damping. As examples data on the unsteady relationship of δ_h to aging time in decomposition of a supersaturated solid solution of carbon in a ferritic steel with 0.2% C [5], the annealing temperature in Fe–Cr system alloys [1], and the tempering temperature in interrupted decomposition in Fe–14.5% Mo-alloys [6] may be quoted.

The purpose of this work was investigation of the rules of influence of various types of crystalline structure defects on the effects of magnetoelastic hysteresis in high-chromium ferritic steels.

Kh16 (15.4% Cr, 0.01% C), Kh16M4 (15.6% Cr, 4.09% Mo, 0.009% C), and Kh25 (24.3% Cr, 0.08% Al, 0.013% C) steels were investigated.

The 10-kg ingots were melted in a Baltsers vacuum induction furnace with cooling in sand and forged at 1100°C into 8-mm rods. Then the rods were drawn according to the sequence $\phi\ 8 \to \phi\ 3 \to \phi\ 1.5 \to \phi\ 0.8$ mm with annealing at 750°C for 30 min, acid-alkaline pickling, and washing after each pass.

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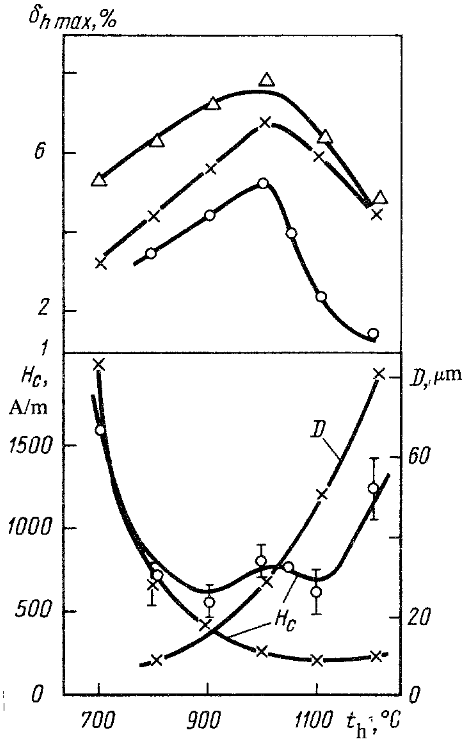

Fig. 1

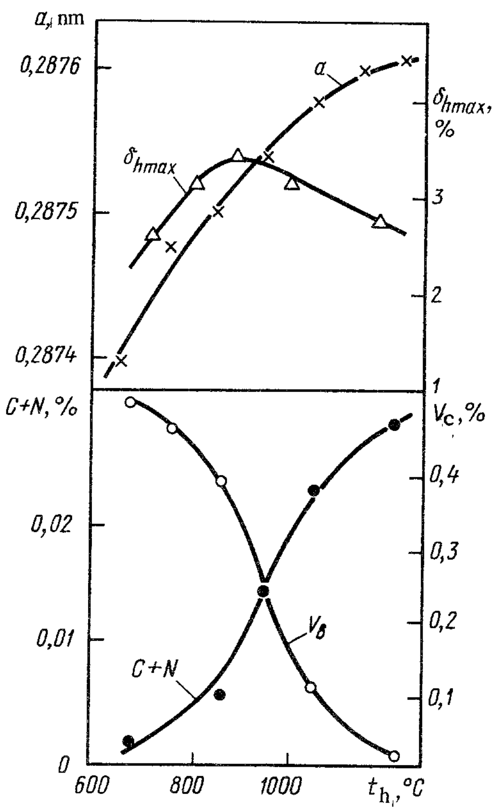

Fig. 2

Fig. 1. Relationship of the maximum logarithmic decrement of vibrations $\delta_{h\ max}$, average grain size D, and coercive force H_c of Kh16M4 steel to hold temperature in heat treatment: ×) annealed according to method 1; Δ) annealed at 1200°C + annealed according to method 1; O) water hardened.

Fig. 2. Relationship of the maximum logarithmic decrement of vibrations $\delta_{h\ max}$, C + N content in the solid solution, wt. % of precipitates of carbonitrides V_c, and crystalline lattice parameter a to hold temperature of Kh25 steel: Δ) in annealing; O, ×, ●) in hardening.

The amplitude relationship of internal friction was measured on a reverse torsional pendulum type relaxation oscillator on 0.8-mm diameter wire specimens with amplitudes of deformation of $\gamma = (1\text{-}140)\cdot 10^{-5}$ and a frequency of vibrations of $f \cong 1$ Hz. In determination of the dislocation component (δ_{disl}) the experiments were made without a magnetic field and in a field with a strength up to $H_s = 3.2\cdot 10^4$ A/m. The magnetomechanical component of damping (δ_h) was determined using the normal method according to the equation

$$\delta_h(\gamma) = \delta(\gamma) - \delta_{\text{дисл}}(\gamma),$$

where $\delta(\gamma)$ is the total damping and $\delta_{disl}(\gamma)$ is the damping as the result of dislocation mechanisms.

The residual internal stresses of the first order (σ_{iI}) were determined on a DRON-2.0 diffractometer from the displacements of the (220) line of the α-phase by the two-position plotting method. The internal stresses of the second order (mean-square microdeformation $<\varepsilon^2>^{1/2}$) were determined from the broadening of the $\beta_{(110)}$ x-ray diffraction lines and the relaxation of the stresses ($\Delta\sigma_{rel}$) after tests of wire specimens on an Instron machine with stresses of $0.8\sigma_{0.2}$ and a loading rate of 0.04 sec^{-1} for 15-20 min. The C and N contents in the high-chromium ferrite and the share of precipitated carbide phase V_c were determined from the height of the Snoek peak in tests of hardened specimens [7]. The coercive force H_c was measured on cylindrical specimens with a diameter of 3 and a height of 40 mm with an F190 microwebermeter. The Curie points of the alloys were determined from the temperature relationship of specific heat c_p. The structures of the steel were investigated with a Neophot light microscope and a JEM-200CX electron microscope with an accelerating voltage of 200 kV. The foils for electron microscopic analysis were prepared by electrolytic polishing in a solution of 860 g of orthophosphoric acid and 100 g of chromic anhydride.

TABLE 1

Heat treatment of Kh16M4 steel	$\delta_{h\,max}$, %	σ_{il}, N/mm²	$\Delta\sigma_{rel}$, N/mm²	σ_t, N/mm²	$\sigma_{0.2}$, N/mm²	$\Delta l/l$, %	$\langle\varepsilon^2\rangle^{1/2}$	$\rho\cdot10^{-9}$, cm^{-2}	H_c, A/m	$\chi\cdot10^{-6}$, T/A
Water hardened from 1000°C for 40 min	3–4	230–240	35	560	410	27	$4{,}2\cdot10^{-4}$	5/8	800	<50
Annealed at 1000°C for 40 min, furnace cooled to 600°C, and then in air	7–8	80–120	18	490	305	32	Taken as the standard	3/2	300	115

Explanation. The first figure is the specific dislocation density determined by the electron microscopic method and the second by the x-ray diffraction method.

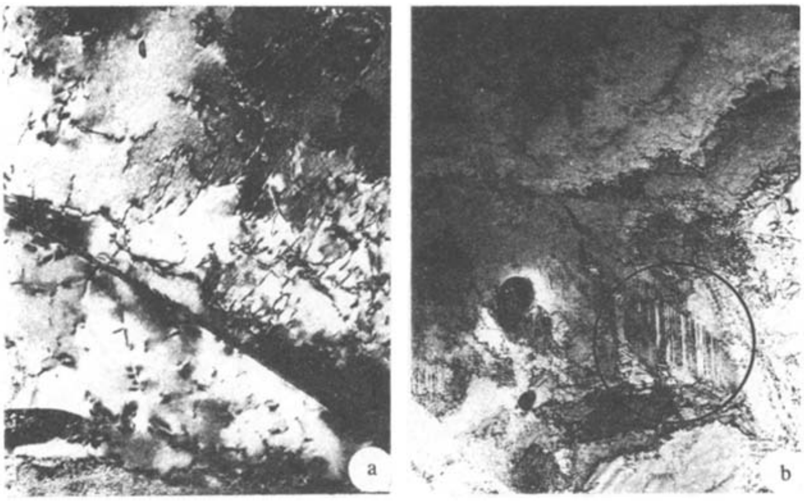

Fig. 3. Structure of Kh16M4 alloy after water hardening from 1000°C for 1 h: a) dislocations in the bodies of the grains and at the grain boundaries, 37,000×; b) lath martensite (in the circle), 10,000×.

The cold-drawn specimens were heat treated according to two methods: 1 — annealed at 700-1200°C for 40 min, furnace cooled to 600°C at a rate of 2°/min, and then air cooled; 2 — water hardened from 700-1200°C for 40 min. In addition a portion of the annealed specimens was also annealed at temperatures below the Curie point ($T_c \cong 680$ and 720°C for Kh16M4 and Kh16 steels, respectively).

It was established that an increase in hold temperature in annealing or hardening of specimens of Kh16, Kh16M4, and Kh25 steels in the 700-1200°C range has an ambiguous influence on the maximum level of the logarithmic decrement of vibrations $\delta_{h\,max}$, coercive force H_c, grain size D (Fig. 1), and magnetic susceptibility χ.

Both after annealing and after hardening there is a maximum on the curves of the $\delta_{h\,max}(t_h)$ relationships, at t_h = 1000°C for Kh16M4 steel (Fig. 1) and at t_h = 900°C for Kh25 steel (Fig. 2). An increase in t_h of the investigated steels leads to a steady growth in the average grain size (Figs. 1 and 3). With an increase in hardening temperature there is an increase in the crystalline lattice parameter a (Fig. 2) and in the height of the Snoek maximum, which is an indication of enrichment of the solid solution by carbon and nitrogen atoms as the result of solution of carbide phases (presumably type $Cr_{23}C_6$ carbides) (Fig. 2).

It has been established (Fig. 1) that annealing of the steels at 700-900°C leads to a sharp drop in coercive force, which is sensitive to the volume share of precipitates and the level of internal stresses. After hardening from 700-900°C a significant quantity of fine (0.3-1.5 μm) carbides is observed in the structure. With an increase in hardening temperature the share of them decreases and a reduction in H_c is observed. After hardening from 1100-1150°C there are practically no carbides in the structure. It should be observed that H_c depends significantly upon the cooling rate after holding in the 1100-1150°C range (Fig. 1), which may be explained by structural changes in the steels. After annealing at 1000°C with furnace cooling a lower dislocation density ρ is observed in the structure of Kh16M4 steel than after hardening (Table 1). In the cold-drawn condition $\rho = 24\cdot10^9$ cm^{-2}.

After annealing the structure of the steels consisted only of ferritic grains with individual 0.3-0.5 μm rounded particles of type Fe_2(Mo, Cr)C phase (a = 1.6276 nm, b = 1.0034 nm, c = 1.1323 nm). The volume share of this phase in the annealed steel is 1.2-2 times more than in the hardened. X-ray diffraction investigations showed that in addition to the above structural constituents the hardened steels contained about 3% austenite. Individual areas of lath martensite were also

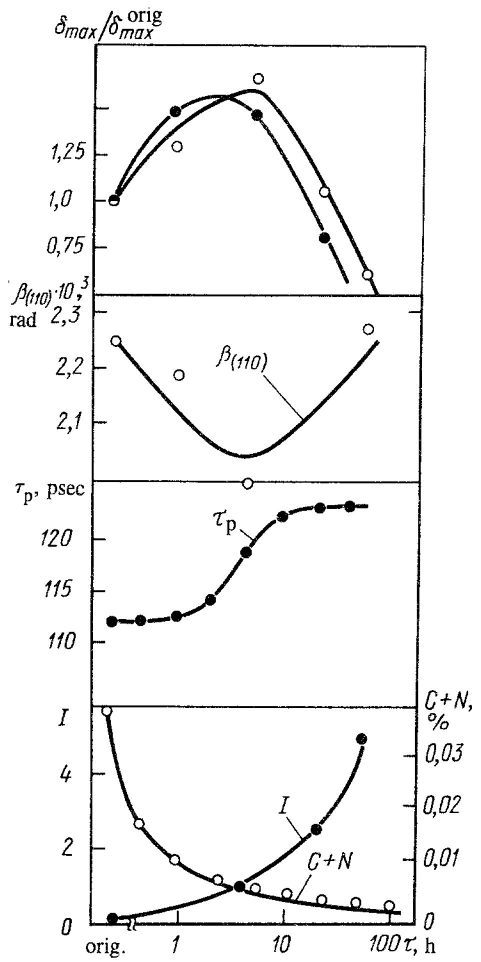

Fig. 4. Influence of annealing time at 475°C on the relative change in maximum values of the logarithmic decrement δ_{max}/-δ_{max}^{orig} of Kh16 (○) and Kh16M4 (●) steels, on the width of the $\beta_{(110)}$ x-ray line of Kh16M4 steel, on the carbon and nitrogen content C + N in high-chromium ferrite, on the intensity I of the low-angle dispersion of neutrons, and on the time of existence of positrons τ_p in Kh25 steel.

observed in the structure of the hardened steels (Fig. 3b). The martensite and residual austenite were primarily localized at the grain boundaries.

The data of Fig. 1 indicates that the curve of the $\delta_{h\ max}(t_h)$ relationship has a maximum and in testing of specimens with a grain size previously created at 1200°C, D ≅ 80 μm, that is, D = const. The presence of a maximum on the $\delta_{h\ max}$ (t_h) curves is observed both after hardening and after annealing (Fig. 1). However, in the case of annealing the absolute level of internal friction is higher than with hardening in the whole investigated heat treatment temperature range. The values of σ_{iI}, $<\varepsilon^2>^{1/2}$, $\Delta\sigma_{rel}$, σ_t, $\sigma_{0.2}$, ρ, H_c, and χ of the hardened and annealed specimens of Kh16M4 steel differ significantly (Table 1).

The maximum on the $\delta_{h\ max}(t_h)$ curve for annealing of the steels corresponds to the area of low values of H_c and to the minimum level of internal stresses (Table 1).

The base heat treatment cycle for Kh16 and Kh16M4 steels was annealled at 900-1000°C (depending upon composition) for 40 min with subsequent furnace cooling to 600°C and then in air, which provides high damping properties of the investigated steels.

Additional annealing of Kh16 and Kh16M4 steels at a temperature of $t_{a.a}$ < T_c (1-h hold) after heat treatment using the base cycle leads to an increase in $\delta_{h\ max}$. The increase in $\delta_{h\ max}$ depends upon $t_{a.a}$ and it is small with $t_{a.a}$ < $t_{c.i}$ ($t_{c.i}$ is the temperature of condensation of interstitial impurities at dislocations [7, 8]). A more significant increase in $\delta_{h\ max}$ is observed after annealing in the $t_{c.i}$-T_c temperature range, which is related to the additional reduction in internal stresses. In the 450-500°C range there is some disturbance of the steady $\delta_{h\ max} = f(t_{a.a})$ relationship. The relationship of $\delta_{h\ max}$ to hold time at 475°C has a maximum, the position of which differs for different steels (Fig. 4). The maximum on the curve of the $\delta_{h\ max}(\tau)$ relationship corresponds to the minimum level of internal stresses in the material, which was determined from the width of the $\beta_{(110)}$ x-ray lines. With a further increase in annealing time at 475°C an additional maximum appears on the curves of low-angle dispersion of neutrons, indicating the process of stratification and formation in the solid solution of concentration nonuniformities, zones enriched with chromium [7]. The intensity of stratification in high-chromium ferrite increases with an increase in annealing time τ (Fig. 4).

With an increase in annealing time in the 300-525°C range a reduction in the height of the Snoek maximum indicating a decrease in carbon content in the solid solution of the steels (Fig. 4) and a decrease in the angle of slope of the amplitude relationship of internal friction (in a magnetic saturation field $H_s = 24 \cdot 10^3$ A/m) related to pinning dislocations by impurities are observed.

In the area of the maximum on the $\delta_{h\ max}(\tau)$ relationship there is observed an increase in time until annihilation of positrons (Fig. 4), which depends upon the elastic distortions occurring in the lattice and the type of junction of the zones enriched with chromium with the matrix (the average time of existence of a positron τ_p was determined from the area of the difference spectrum of the specimen and the standard with an error not exceeding ± 3 psec [9]).

The steady relationships of interstitial impurity content, volume share of precipitates, and average grain size to temperature and time of isothermal annealing does not make it possible to explain the nonsteady temperature $\delta_h(t_h)$ and time $\delta_h(\tau)$ relationships of the damping capacity of the investigated steels. The necessity arises of an analysis of both the separate and the combined influence of crystalline lattice defects of various types on the magnetic constituent of damping.

The unsteady reaction of a ferromagnetic solid body from the point of view of its capacity for internal dispersion of energy with a change in annealing temperature or time is caused by the simultaneous action of a series of micromechanisms of magnetoelastic hysteresis, each of which may be characterized by the coefficient of absorption ψ_{h_i} or by the decrement δ_{h_i}: $\delta_{h_{s.s}}$ is retarding of movement of domain boundaries as the result of strengthening of the solid solution by interstitial impurities (accumulations and segregations of point defects); δ_{h_d} is interaction of the domain boundaries with dislocations; $\delta_{h_{prec}}$ is with precipitates (areas of a second phase); δ_{h_g} is with grain and subgrain boundaries. For the investigated steels most typical is the situation when $\delta_h >> \delta_{disl}$ and the amplitude of external stresses σ does not exceed the level of average internal stresses σ_i.

To determine δ_{h_i} let us use the fact that in steels with a ferritic structure relatively low strength properties and a low level of internal stresses correspond to a high level of damping [10]. For determination of δ_{h_i} this makes it possible to use known concepts of the contribution of various mechanisms to strengthening of the class of materials considered [11, 12]: solid solution $\Delta\sigma_{s.s} = \sum_{i=1}^{n} k_i C_i$; dislocation $\Delta\sigma_d \sim \rho^{1/2}$; dispersion as the result of coherent precipitates $\Delta\sigma_{coh} \sim V$, partially coherent $\Delta\sigma_{p.c} \sim V^{1/2} t / D_0^{1/2}$, and noncoherent $\Delta\sigma_{nc} \sim V^{1/2}/d$; grain boundary $\Delta\sigma_g \sim D^{-1/2}$, where k_i is the hardening coefficient of the material by the i-th form of impurity with a concentration in the solid solution of C_i, d is the average size of noncoherent precipitates, t and D_0 are the thickness and diameter of partially coherent precipitates (zones of stratification) of disk shape, and V is the volume share of precipitates.

In the Maxwell distribution function of internal stresses the absorption factor, according to [4], at the maximum of its amplitude relationship ($\sigma = \sigma_m$) is determined from the equation

$$\psi_{max} = 0{,}34(2K\lambda_s E/\sigma_i). \tag{1}$$

In the area of small amplitudes of stresses

$$\psi_h = (8K\lambda_s E/3\sigma_i)x, \tag{2}$$

where $x = \sigma/\sigma_i$, E is Young's modulus, λ_s is the magnetostriction constant, and $K \cong 1$ is a dimensionless coefficient. With a constant amplitude of external stresses σ the relationship of δ_h (or ψ_h) to σ_i has the form

$$\delta_h \sim 1/\sigma_i^m \tag{3}$$

where m = 2 (with $\sigma << \sigma_i$) or m = 1 (with $\sigma \cong \sigma_m$).

The values of K, λ_s, and E are weakly structure-sensitive in comparison with δ_h. By substitution of the appropriate expressions for $\Delta\sigma_i$ in Eq. (3) we obtain:

$$\delta_{h_{s.s}} \sim 1/\left(\sum_{i=1}^{h} k_i C_i\right)^{2n},\ \delta_{h_d} \sim 1/\rho^n;\ \delta_{h_{coh}} \sim 1/V^{2n}; \tag{4}$$

$$\delta_{h_g} \sim D^n;\ \delta_{h_{nc}} \sim (d^2/V)^n,\ \delta_{h_{p.c}} \sim (D_0/Vt^2)^n, \tag{5}$$

where n = 1/2 (with $\sigma \cong \sigma_m$) or n = 1 (with $\sigma \ll \sigma_i$). The relationships given are confirmed by experimental data from which it follows that an increase in the steels in the concentration of sources of internal stresses (grain boundaries, dislocations, precipitates, impurity atoms), which are points of retarding of domain boundaries, promotes a reduction in the losses to magnetoeleastic hysteresis [2, 5, 13-16].

The basic factors in the increase in δ_h with an increase in annealing temperature are solution of carbonitride phases, the decrease in the volume share of precipitates (Fig. 2), an increase in the average grain size (Fig. 1), and annealing of internal stresses occurring in rolling of the specimens. Each of these factors steadily increase the corresponding constituent of δ_{h_i}. As the total result this group of factors causes a steady tendency toward growth of δ_h.

The following processes decrease δ_h: enrichment of the solid solution with interstitial impurities; internal stresses of hardening origin, which increase with an increase in heat treatment temperature and cause an increase in dislocation density; in individual cases the appearance and increase in the volume share of martensitic phase leading to an increase in internal stresses or of nonmagnetic austenitic phase. The latter factor apparently causes a change in the character of the curve of the $H_c(t_h)$ relationship in the area of high temperatures in furnace cooling and hardening (Fig. 1). This group of factors in combination causes a tendency toward a steady decrease in δ_h.

The combined influence on δ_h of two opposite factors (toward an increase and toward a decrease) leading to obtaining experimental relationships in the form of curves with a maximum (Fig. 1) cannot be explained with the use of simple totalling of the individual contributions of δ_{h_i} assuming that

$$\psi_{h_\Sigma} = \Delta W_\Sigma / W = \Delta W_1 / W + \Delta W_2 / W \tag{6}$$

where W is the energy of vibrations corresponding to the peak values of σ or ε; ΔW_1 and ΔW_2 are the energies dispersed during a cycle of vibrations as the result of the first and second groups of factors. The inapplicability of Eq. (6) is related to features of behavior of the ferromagnetic steels considered as a nonlinear medium, which is the result of the occurrence of additional (in relation to elastic) mechanostrictive deformation, the ΔG-effect, which is sensitive to internal stresses, and the presence of a relationship of the energy of vibrations of the crystalline lattice in local areas close to defects to their effectiveness as obstacles to 90°-type domain boundaries. In addition to the energy of elastic deformation the full energy of deformation W in such materials will include the energy of magnetic anisotropy and the domain boundaries and the magnetoelastic and magnetostatic energy.

As the result of additional annealing in the $t_{a.a} < T_c$ range the increase in $\delta_{h\ max}$ occurs primarily as the result of the decrease in interstitial impurity concentration in the solid solution of the steels (Fig. 4). Even during a short anneal at 475°C decomposition of the solid interstitial solution is observed (in 1 h about 70% of the C and N atoms located in it are precipitated from the solid solution). This process leads to pinning of dislocations by Cottrell impurity atmospheres (kinetic coefficient of precipitation according to Wert–Zener is 0.5-0.7) and in the initial stages of annealing is accompanied by a reduction in internal stresses in the ferrite (Fig. 4). In combination these processes cause a tendency toward an increase in $\delta_{h\ max}$.

The following structural changes act in the opposite direction. With an increase in the isothermal annealing time at 475°C beyond 3 h (n = 0.1-0.3) the processes of decomposition of the solid interstitial solution in steels occur by growth of the earlier formed precipitates. Simultaneously as the result of thermodynamic instability of the Fe–Cr substitutional solid solution in this concentration temperature area there are observed processes of stratification occurring in high-chromium ferrite and leading to formation of zones enriched with Cr and the interstitial impurities N and C. As the result a structure modulated with respect to chromium, which is characterized by a high level of internal stresses occurring around zones enriched with Cr and causing the appearance of 475°-brittleness, is formed. with sufficiently long holds (100 h) at 475°C the structure is characterized by the following parameters (according to the data of the change in low-angle dispersion of neutrons for Kh25 steel [17, 18]): volume share of zones enriched with Cr, V ≅ 2%; density of the zones 10^{16}-10^{20} cm^{-3} (depending upon the original condition); radius of the zones (assuming as an approximation spherical precipitates) from 1 to 10 nm; fields of elastic distortions up to 15 nm; chromium content in the zones approximately 50% [12, 19]. Formation in the solid solution of zones enriched with chromium causes structural and magnetic inhomogeneity of the steel, which reduces the mobility of domain boundaries and consequently δ_h.

Therefore, the factors in the reduction in δ_h are caused primarily by accumulation in the material of zones possessing a low magnetic moment of unity of the volume and creating significant elastic distortions, which retard the shift in domain boundaries as the result of an increase in the Rayleigh potential. This is indicated by broadening of the $\beta_{(110)}$ x-ray line after annealing at 475°C for $\tau > 8$ h and the increase in time of annihilation of positrons (Fig. 4). As in the case of high-temperature annealing, the presence of oppositely acting mechanisms leads to formation of a maximum on the curve of the $\delta_{max}(\tau)$ relationship of the steels (Fig. 4).

Conclusions. Damping in high-chromium ferritic steels is formed primarily as the result of magnetoelastic dispersion of energy, the level of which is determined by the character of interaction of the magnetic domain boundaries moving in the field of applied cyclic stresses with some subsystems of stoppers, defects of the crystalline structure. The maximum in damping capacity of Kh16, Kh16M4, and Kh25 steels is observed after annealing at 900-1000°C and is the result of superposition of individual oppositely acting on the magnetic constituent of damping contributions during evolution of structural defects under different temperature–time actions. An analysis of the contribution of different crystalline lattice defects makes it possible to predict the path of formation of the controlled level of vibration absorbing properties of high-chromium steels. The high-damping condition is characterized by a low level of internal stresses, moderate values of coercive force, and non-linearity of the elastic properties under the action of external stresses. The damping capacity after annealing in the temperature area of stratification of high-chromium ferrite and appearance of 475°-brittleness is determined by the action of two primary tendencies, the reduction of internal stresses as the result of ecomposition of the supersaturated solid solution of interstitial impurities and the subsequent increase in them as the result of stratification of high-chromium ferrite and formation of zones enriched with chromium.

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