Features of temperature-dependent internal friction at phase decomposition of Nb-Ti alloy

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Experimental dependence of internal friction $Q^{-1}(T)$ on heating temperature T of pre-strained Nb-(48.5±1,5)Ti (wt %) alloy is a superposition of two internal friction peaks with maxima at 250°C and 300°C. It is shown by calculation that both the peaks are due to nucleation and subsequent diffusion-controlled growth of α-Ti segregates in the walls of dislocation cells. The first peak is connected with Ti diffusion in the cell boundaries while the second one, in the volumes thereof.

Экспериментальная зависимость внутреннего трения $Q^{-1}(T)$ от температуры T нагрева предварительно деформированного сплава состава Nb-(48.5±1,5)Ti (wt.%) представляет собою суперпозицию из двух пиков внутреннего трения с температурами максимумов 250 °С и 300 °С. Расчётным путём показано, что оба этих пика обусловлены зарождением и последующим диффузионным ростом выделений α-Ti в стенках дислокационных ячеек. Первый пик связан с диффузией Ti в границах, а второй – в объёмах этих ячеек.

The Nb-Ti alloys are of considerable importance in applications of low-temperature superconductivity. The role thereof in the electric energy production and use (thermonuclear units, electrical energy accumulators and generators, magnetically suspended high-speed transport, etc. [1]) confirms enough that importance. The Nb-(48.5±1,5)Ti (wt. %) alloy is referred to in engineering as NT50. The current-carrying structure of wires and strips made of that alloy is formed in the course of its thermal/mechanical treatment. First, a strong plastic straining it the region of the single-phase β state with b.c.c. lattice results in formation of cellular dislocation structure in NT50. Then, under isothermal holding of the pre-strained alloy, e.g., for 400 h at 390°C [2], the supersaturated solid solution is decomposed under segregation of α-Ti particles with h.c.p. lattice mainly at the dislocation cell boundaries.

The kinetics of isothermal NT50 decomposition has been traced in [3] using the internal friction method. When considering the first order phase transitions, the observation time t of the phase decomposition under isothermal conditions [3-5]:

$$Q^{-1}(K,n,t) = \frac{cKnt^{n-1}}{T}\exp\left(-Kt^{n}\right) \tag{1}$$

Eq. (1) can be used both for forced and free vibrations [3]. Therein, T is the instantaneous temperature of the alloy; the parameter $n>0$ characterizes the formation and growth rate of the new phase nuclei. According to [4], the phases are segregated at $n = 1$ along the boundaries of the structure elements; at $n = 2$, at the edges of structure elements; when the phase nucleation rate decreases, $3 \leq n \leq 4$. The factor $c = c(t)$ includes the sample vibration frequency f connected with the material elasticity modulus: $G \sim f^2$. The changes in elastic moduli in time is not expressed so clear as compared to the internal friction, since the elastic properties are defined mainly by the binding forces between atoms in the lattice and to a lesser extent by the material structure. That is why $f(t) = $ const can be assumed in calculations using Eq. (1); then $c(t) = $ const. At certain numerical values of K
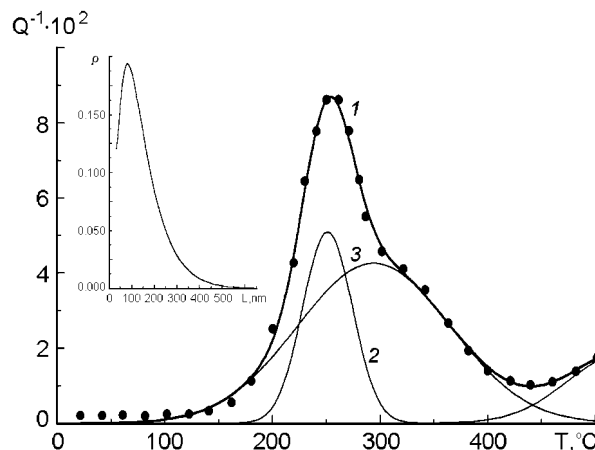

Fig.1. Experimental temperature dependence $Q^{-1}(T)$ of internal friction for pre-strained NT50 alloy at the sample heating rate 2 deg/min (*1*) and its components (*2, 3*). Inset: distribution function of dislocation cell linear dimensions in the NT50 sample under study [2].

and n, Eq. (1) "forecasts" an exponential drop of internal friction in NT50 during an initial time period and appearance of temporal $Q^{-1}(t)$ maxima later. Those internal friction $Q^{-1}(t)$ features were found for NT50 before in experiments [3]. Processing of experimental data on $Q^{-1}(t)$ using Eq. (1) had resulted in the following values of Avrami parameters: $n = 1$ and $n = 4.4 \pm 0.5$ for strained NT50 annealed at 300°C; $n = 1$ and $n = 2.60 \pm 0.75$ for cast NT50 annealed at 375°C. The phase decomposition kinetics may influence the character of temperature dependences of internal friction in NT50. Therefore, the purpose of this work is to reveal and consider numerically the features of temperature-dependent internal friction in NT50 reflecting the phase decomposition kinetics of the alloy.

An NT50 ingot was prepared by vacuum arc melting. To provide the cellular structure, the ingot was plastically strained by pressing and dragging at 600°C [2]. To measure the internal friction, samples of 2 mm diameter and 20 mm working element length were cut out of the workpiece so obtained. In a dark-field electron microscopic image of NT50 alloy subjected to a similar thermal and mechanical treatment, α-Ti accumulations of about 100 nm average size are seen at the cell boundaries. The distance L_{gb} between surfaces of two neighboring accumulations along the cell boundary was 50 to 100 nm [6]. The internal friction was measured at a constant heating rate $\dot{T} = 2$ deg/min by damping torsional vibrations method at an amplitude of the order of 10^{-4} and frequency $f \approx 3$ Hz using a unit described in [7].

In an experiment with NT50 alloy, a complex temperature dependence of internal friction $Q^{-1}(T)$ is recorded (Fig. 1) consisting of two peaks at two rather different temperatures 250°C and 300°C. To establish the mechanisms providing that feature of internal friction, let the $Q^{-1}(T)$ curve be simulated. To that end, let the change interval of heating temperature ΔT during the internal friction measurement time $t = \Delta T / \dot{T}$ be introduced into Eq. (1), provided that the sample heating rate is not too high. At a slow (quasi-equilibrium) heating, the temperature lag in the sample inner regions against its outer layers can be neglected. Since the NT50 decomposition is of diffusion nature, let the Avrami parameter K be presented as $K = (1/\tau)^n$ by introducing the characteristic diffusion time τ. This time is a function of diffusion coefficient D of dissolved atoms and the diffusion path (distance) that those have to pass to each growing particle. At the initial growth stage of just a formed α-Ti nucleus, its growth rate will be controlled by the boundary diffusion. Then, however, it is volume diffusion that will be predominant [8]. When the formation work of a precipitate nucleus is low as compared to the activation energy U of the volume diffusion, and there is no energy barrier for such diffusion at the phase interface, then the following relationships are valid:

$$L \approx (D\tau)^{1/2}, \text{or} \tag{2}$$

$$\tau \approx \frac{L^2}{D} = \frac{L^2}{D_0} \exp\left(\frac{U}{kT}\right),$$

where D_0 is the frequency factor of the volume diffusion; k, the Boltzmann constant. In the presence of volume diffusion, Eq. (1) takes the form

$$\frac{Q^{-1}(n,L,T)}{c} = \frac{1}{T}\left(\frac{D_0}{L^2}\right)^n \left(\frac{\Delta T}{\dot{T}}\right)^{n-1} \left[\exp\left(-\frac{nU}{kT}\right)\right]\left\{\exp\left[-\left(\frac{D_0}{L^2}\right)^n \left(\frac{\Delta T}{\dot{T}}\right)^n \exp\left(-\frac{nU}{kT}\right)\right]\right\}. \tag{3}$$

To estimate the volume diffusion coefficient of Ti in NT50 alloy at moderate temperatures up to 600°C, let the universal relationship [9, 10] be used that is valid for any self-diffusion and hetero-diffusion processes:

$$D = \alpha \frac{a^2}{\tau_0} \exp\left(-\frac{U}{kT}\right),$$

where a is the alloy crystal lattice period; $1/\tau_0$, the maximum (Debye) vibration frequency of atoms in the lattice ($1/\tau_0 \sim 10^{13}$ s⁻¹). In binary substitution alloys, the atomic diffusion over vacancies predominates. For the vacancy mechanism of diffusion in a b.c.c. lattice, the geometry parameter $\alpha = 1/8$ [9]. The b.c.c. lattice period of NT50 solid solution is essentially unchanged within the 20 to 600°C temperature range of one-hour annealings and is $a = 3.2832$ Å [11]. Then the frequency factor of the volume diffusion in NT50 is $D_0 = \alpha a^2/\tau_0 \approx 0.14$ mm²/s. At temperatures near 1000°C, the volume diffusion activation energy for Ti impurity in **Nb** is $U = 0.9$ eV [12] and that for **Nb** impurity in β-Ti, $U = 1.5$ eV [13]. In the case under consideration when Ti diffuses in NT50 at temperatures up to 600°C, the activation energy seems to be of the same order, $U \approx 1$ eV.

When α-Ti accumulations grow due to Ti volume diffusion, the diffusion path length L is equal to the linear dimension of dislocation cell. That dimension is not fixed in strained NT50 alloys. It may ba distributed in various manners depending on the thermal and mechanical treatment modes. The inset in Fig. 1 presents the non-normalized distribution function $\rho(L)$ for linear cell dimensions in the NT50 sample under study [2].

When the NT50 samples are heated, the azimuthal disorientation of dislocation cells increases due to polygonization and the cells may transform themselves into subgrains [14]. That phenomenon will not be considered in what follows. Let the sample heating rate to be $\dot{T} = 2$ deg/min and the rounded values of Avrami parameters for NT50 to be $n = 1$, $n = 2$ and $n = 4$. For each individual n value, let the internal friction be calculated using the formula

$$Q_V^{-1}(n,T) = \int_{L_{min}}^{L_{max}} Q^{-1}(n,L,T)\rho'(L)\mathrm{d}L, \tag{4}$$

where $\rho(L)$ is the normalized distribution function $\rho(L)$. Let the partial temperature dependences (4) to be added together to get the total temperature dependence

$$Q_V^{-1}(T) = Q_V^{-1}(1;T) + Q_V^{-1}(2;T) + Q_V^{-1}(4;T)$$

of internal friction caused by nucleation and growth of a new phase due to Ti volume diffusion (Fig. 2, curve *1*).

Now let the contribution from the Ti boundary diffusion to the internal friction to be taken into account as follows. From the Fisher equation [10] for the spatial concentration distribution of the diffusing substance in the presence of boundary diffusion, it follows that the diffusion along a cell

boundary should be accompanied necessarily by diffusion in the volume thereof. This correlation manifests itself in the fact that the diffusion path along the cell boundary L_{gb} depends on the ratio $D_{gb}/D \gg 1$ between the boundary and volume diffusion coefficients, respectively:

$$L_{gb} \approx (\delta D_{gb}/D)^{1/2}(D\tau)^{1/4}, \tag{5}$$

where δ is the cell boundary width that is assumed usually to be $\delta \approx 0.5$ nm [8]. In the case of boundary diffusion, the diffusion path length is in proportion to forth root of the time (5) rather than to square root (2) as in the case of volume diffusion. According to D. Turnbull [8], the decomposition of alloys is controlled by the boundary diffusion if $D_{gb}/D = I_{gb}/\delta$. Then we get from (5) that

$$L_{gb} \approx (D\tau)^{1/2}, \text{ or}$$

$$\tau \approx \frac{(L_{gb})^2}{D} = \frac{(L_{gb})^2}{D_0}\exp\left(\frac{U}{kT}\right). \tag{6}$$

The comparison of (2), (3), and (6) shows that the contribution from Ti boundary diffusion to the internal friction can be taken into account by substituting the ratio $D_0/(L_{gb})^2$ in (3) for D_0/L^2. The accurate temperature dependence of L_{gb} is unknown. However, the new phase nucleation in the dislocation cell walls are fast over, and the diffusion-controlled growth of those nuclei in aging alloys (including NT50) proceeds very slowly [8]. So, using the X-ray small-angle scattering, it has been shown that the α-Ti inclusion radius increases only by a factor of 3, and the reduced spacing of those inclusions remains essentially unchanged during holding of cold-strained NT50 wires for 10^2 to 10^5 min at 380°C [15]. Thus, the diffusion path length L_{gb} can be believed to be constant during the measurement time of internal friction (300 min). Let $L_{gb} = 50$ nm and $T = 2$ deg/min. Then, for $n = 1$ and $n = 2$ parameter values (concerning, according to R. Cahn, only α-Ti precipitation in the cell walls), let the partial contributions $Q_{gb}^{-1}(1;T)$ and $Q_{gb}^{-1}(2;T)$ the total one

$$Q_{gb}^{-1}(T) = Q_{gb}^{-1}(1;T) + Q_{gb}^{-1}(2;T)$$

from the boundary diffusion to the sought internal friction be calculated using formula (3). The results are shown in Fig. 2 (curve *2*). The resulting curve of internal friction

$$Q^{-1}(T) = Q_{gb}^{-1}(T) + Q_V^{-1}(T)$$

in the presence of both boundary and volume diffusion is shown as curve *3* in Fig. 2.

The experimental data (Fig. 1) and calculated ones (Fig. 2) are seen to agree qualitatively rather well. This means that the temperature dependence of internal friction in NT50 is due to Ti diffusion both along the dislocation cell walls and within the cell volumes. It follows from the proposed calculation algorithm for $Q^{-1}(T)$ that the temperature position of the internal friction peak in NT50 associated with Ti diffusion within the dislocation cell volumes depends on the distribution function of the cell linear dimensions.
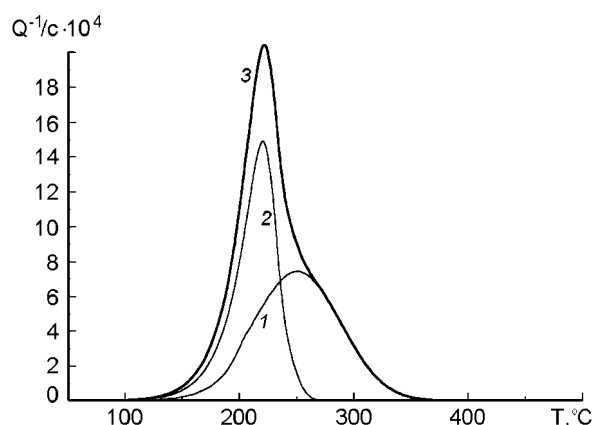

Fig.2. Calculated temperature dependences $Q^{-1}(T)$ of internal friction for pre-strained NT50 alloy at the sample heating rate 2 deg/min. Contributions from Ti volume diffusion (*1*) and boundary diffusion (*2*) to the internal friction and the resulting internal friction curve (*3*).

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Особливості температурно-залежного внутрішнього тертя прифазовому розпаді сплаву НТ50

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Експериментальна залежність внутрішнього тертя Q-1(T) від температури T нагрівання попередньо деформованого сплаву складу Nb—(48,5±1,5)Ti (масс.%) являє собою суперпозицію з двох піків внутрішнього тертя з температурами максимумів 250 °С і 300 °С. Розрахунковим шляхом показано, що обідва ці піки обумовлені зародженням і наступним дифузійним ростом виділень α-Ti у стінках дислокаційних комірок. Перший пік пов'язанний з дифузією Ti на границях, а другий - в об'ємах цих комірок.