

RESEARCH
OF THE POLYMORPHIC TRANSFORMATION
IN TITANIUM
WITH VARIOUS THERMAL MODES

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Introduction

At the study of phase transformations in the solid state a question arises inevitably as to whether the observable parameters of the structure change depend on the intensity of the external influence initiating the transition. With reference to titanium, chosen as an object in the present research, such a phase transformation is the reorganization of the crystal structure. At the normal pressure during the heating of the metal its hexagonal closed packed lattice (α -phase) is reconstructed into a body centered cube (β -phase) in the vicinity of 1155 K [1].

The scattering of the results of measurements of the transition temperature can be caused by the amount of impurity in a sample, by the way of the fixing of the moments in the beginning and the end of phase reorganization, at last, by the intensity and orientation of a thermal influence on metal. On Fig.1 published data on the temperature of structural reorganization in titanium are given depending on the rate of heating. The conclusion from the submitted picture is obvious – the rate of heating utilized in experiments, as well as its orientation, influence fixed parameters of phase transition.

The possible way of the registration of the transition temperature is formed by the temperature dependence of the metal physical characteristic, which measurement allows to fix the moments of a beginning and termination of the transition. The structural reorganization in the solid titanium is a phase transition of the first order, for which the latent heat of the transformation takes place. Therefore the basic way of the registration of the transition is the study of thermogram anomalies on conditions of the entered thermal energy constancy (or by the weak monotonous change).

In the present work just this way is chosen for supervision over the phase transformation. For its realization the volumetric heating of a specimen by the electrical current pulse is applied. The temperature of the specimen and electrical parameters are measured during the specimen heating allowing to determine the entered energy and the electrical resistance.

Experiment

The experimental set-up used in work, was described in [2]. Let's note here concrete parameters and conditions of described experiments. Tubular specimens of technical titanium VT1-0 (weight %: 0.09 Fe, 0.03 Si, 0.03 C, 0.03 O, 0.006 H, 0.02 N) with outside and internal diameters 14.02 and 13.55 mm accordingly were used.

Length of a sample was equal 55 mm. The ends of the specimens were densely fixed in the cold electrodes. A graphite cone bearing inserts provided good electrical contact of an outside cylindrical surface of trailer zones of the specimen to the surface of the current contact jaw.

For measurement of the voltage on the central zone of a sample were used two tungsten needles entered by the edges into apertures by a diameter 0.4 mm in the wall of the specimen tube. Temperature was measured using photo-electric pyrometers of the millisecond resolution. The calibration of the pyrometers was carried out with a temperature lamp installed in the chamber in place of a specimen. The management of the experiment was provided with the computer program allowing to stop the heating by the interruption of an electrical current on given duration of process or on the achievement of a given limiting temperature. Besides the program allowed to establish the increased interval of time between measurements of temperature for a stage of the cooling.

The measurements were preceded with the numerical modeling of an expected thermal process, which purpose was study of the temperature distribution along the specimen for all stages of process of heating and cooling.

In a basis of the model the description of the process by the differential equation laid:

$$C_v \partial T / \partial t = \partial (\lambda \partial T / \partial x) / \partial x + I^2 \rho / S^2 - \varepsilon \sigma (T^4 - T_e^4) (P_1 - \varphi(x) P_2) / S, \quad (1)$$

where C_v – heat capacity of the volume unit of the metal, λ – thermal conductivity, ρ – specific electrical resistivity, ε – total hemispherical emittance, σ – the constant of the Stefan-Boltzman law, S – the cross section of the specimen, P_1 – outside perimeter of the tube, P_2 – its internal perimeter, $\varphi(x)$ – angular coefficient of irradiation in system: an elementary ring layer of an internal surface of the tube – target apertures, T – true temperature.

The equation (1) is constructed in an approach of a “thin” tube, when the radial difference of temperatures on its wall can be neglected. When calculated temperature in an elementary layer reached the temperature of the phase transformation, the right part of the equation (1) (we shall designate it as q_v) determined the rate of increase of weight of a new phase in the elementary cell considered in view of latent heat of phase transformation L :

$$dm/d\tau = q_v / L \quad (2)$$

Thus, it was possible to find the fields of temperatures in view of the expenses of heat for reorganization of structure. The features of the numerical algorithm can be found in [3].

In Fig. 2 the reorganization of the temperature fields is shown for the heating process of the titanium specimen. The value of a current is chosen in such a way as to emphasize the influence of an effect of heat removal to the cold electrodes. It is visible, that the specimen approaches to the moment of the phase reorganization beginning essentially nonisothermal on length. When the central area ($\delta = 25$ mm) enters phase transition, other part of a specimen is still underheating. Let's emphasize the fact of the consecutive expansion of the isothermal zone during an interval of time spent on phase transition in the central zone.

After its end in section of the specimen with coordinate $\delta = 25$ mm the rise of temperature begins again, and the isothermal zone is broken off and gradually collapse. In the finishing moment shown in figure, only the break on a curve $T(x)$ in the area 16 and 34 mm is evidence of the presence on the specimen two crystal structures.

Figure shows that at the low heat generation intensity it is possible not to receive approach of an indefinite - long tube, within the bounds of which one usually estimate heat capacity, latent heat, total emissivity and electrical resistance. The calculation of all these characteristics bases on results of measurements of a power failure on a site of the tube allocated with potential probes. And if in its limits a specimen is not isothermal, as it is shown in Fig. 2, the estimation of these properties can not have a satisfactory accuracy. Moreover, if the slot-hole model of a black body is used, in these conditions there is uncertainty in the estimation of its effective emittance. Hence, it is possible to obtain an apparent temperature effect of phase transformation, to be actually isothermal.

The calculations of a temperature profile allow estimating parameters of heating (current strength, length of a sample), at which it is possible to fulfill the requirements of the isothermality of the specimen working site.

Figure 3 shows two consistently taken off thermograms of heating - cooling of the specimen with the current of 500 A, interrupted at temperatures 1400 and 1600 K accordingly. Let's note the obviously expressed hysteresis of the brightness temperature at the phase transformation. On the stage of the cooling process of the return reorganization of the lattice begins at a temperature which was lower, than the temperature fixed on the stage of heating.

It would be possible to assume, that the reason of a hysteresis is the irreversible change of the spectral emissivity at an output in the area of the β - phase. It is known, that in the β - phase the acceleration of the diffusion processes can initiate to destruction oxide films on a surface of titanium. However recurrence of the process in the same conditions without depressurization of the vacuum chamber (the curve 2) has given the same temperature of the $\alpha \rightarrow \beta$ transition (zone 3) and again an underestimated temperature of the $\beta \rightarrow \alpha$ transition (zone 4).

At the analysis of the phase transformations of the first order always there is a question on possible overcooling of a system. The presence of the poorly expressed intermediate minimum on the thermogramm of cooling can be a certificate of such an overcooling. In the point of a minimum of the thermogramm the expense of heat are compensated for the account of the heat generation accompanying the reorganization of the crystal structure. The intensity of thermal losses determines the rate of the formation of a new phase. This process under condition of isothermality of a controllable site of a specimen is described by the equation

$$H_{\beta\alpha} \frac{dM}{d\tau} = \int_F q_{\text{rad}} dF \quad (3)$$

in which $H_{\beta\alpha}$ – latent heat of the transition from β - in α -phase, $dM/d\tau$ – the rate of increase of weight α -phase in the volume of the working site of a specimen, q_{rad} – density of thermal flow, loosed from an unit of the specimen surface, F – the total area of a surface of the working site participating in heat exchange with an environment.

After the structure reorganization completed the value $dM/d\tau = 0$ and thermal losses cause the further decrease of temperature. The further changes in time of

brightness temperature and electrical resistance are shown. The points of a maximum and minimum of the resistance curves determine an interval of time $\Delta\tau_R$ reorganization of the phase structure of all working site. If it were isothermal, then temporary parameter $\Delta\tau_R$ would be equaled to the appropriate interval on thermogram $\Delta\tau_T$. Unfortunately, it is necessary to ascertain a difference of these intervals. This difference determines a possible error in the calculation of the latent heat of transformation.

For the isothermal zone all electrical energy, entered during the phase transformation, and is spent on the change of the structure of the working site and heat losses from its surface:

$$\int_{\Delta\tau_R} U \cdot I \, d\tau = H_{\alpha\beta} \cdot S \cdot L \cdot \gamma + Q_{hl} = Q_{isot}, \quad (4)$$

where S – the area of cross section of the tube, L – a length of a working zone, γ – density, $H_{\beta\alpha}$ – latent heat of the transformation, U, I – instant values of a voltage on a working site and a current, Q_{hl} – the heat losses.

In case of a unisothermal site the equation of a balance should be added with terms which take into account an expenses of heat on the change of temperature of zones in the specimen, underheated up to temperature of the transformation at the beginning of this process:

$$\int_{\Delta\tau_R} U \cdot I \, d\tau = Q_{isot} + \int_L C_{v\alpha} S \Delta T_1(x) \, dx + \int_L C_{v\beta} S \Delta T_2(x) \, dx + 2 \int_{\Delta\tau_R} S \lambda \left| \frac{dT}{dx} \right| \, d\tau. \quad (5)$$

Here $\Delta T_1(x)$ is the value of the underheating of a working zone up to the transformation temperature at the beginning of the phase transition in any point of the zone. $\Delta T_2(x)$ is the value of the overheating of this zone at the moment, when on its most cold ends the phase transition was finished, (see Fig. 5).

Unfortunately, both in our work and in realizations of the subsecond pulse heating, known for us, the distribution of temperatures along a sample are not supervised during experiments. Therefore, the use of the equations (5) is not possible for the estimation of latent heat. The indirect certificate of a unisothermality of a working site at a stage of heating is the inequality of values $\Delta\tau_R$ and $\Delta\tau_T$. With reference to the parabolic distribution of temperature the determination of latent heat using $\Delta\tau_R$ will give the overestimated value, and the account on characteristic points of the $T(L/2)$ thermogram will give understating the considered thermal effect. Only in case of equality $\Delta\tau_R$ and $\Delta\tau_T$ it is possible to expect for a correct estimation of the thermal effect.

For the experiments shown on Fig. 4, the value of the thermal effect of $\alpha \rightarrow \beta$ transformations calculated using $\Delta\tau_T$ was found to be equal of $3.7 \div 4.0$ kJ/mol. The calculation with $\Delta\tau_R$ has given the value of $4.7 \div 5.9$ kJ/mol. The losses of energy on radiation from the surface of the working site have made ~ 14 J, i.e. 6.3 % from energy entered during transformation. At the variation of conditions of experiment the share of these losses can be reduced at increase of the heat generation intensity.

The brightness temperature of the $\beta \rightarrow \alpha$ - transition, fixed on a stage of the natural cooling of a specimen got $T_{\lambda=0.65} = 1116$ K. The equilibrium temperature of the $\alpha \rightarrow \beta$ transition for technical titanium containing up to 0.2 mass % of technological impurity

was found 1155 K [11]. It allows to estimate spectral emittance of the surface of our specimen. It is known, that

$$\varepsilon_{\lambda} = \exp[c_2(T_{\lambda} - T)/(\lambda T \cdot T_{\lambda})], \quad (6)$$

whence $\varepsilon_{\lambda=0.65} = 0.512$.

Using this value, the true temperature of the $\alpha \rightarrow \beta$ – transition was calculated at the stage of the specimen heating ($T_{\lambda} = 1156$ K). It has appeared equal $T_{\alpha-\beta} = 1200$ K.

Thus, at the rate of the heating ~ 600 K/s, the polymorphic transformation in technical titanium begins at the temperature exceeding equilibrium value up to ~ 45 K. We have also to note the fact of the development of a non-isothermality of the phase transformation at a stage of the repeated heating of the specimen.

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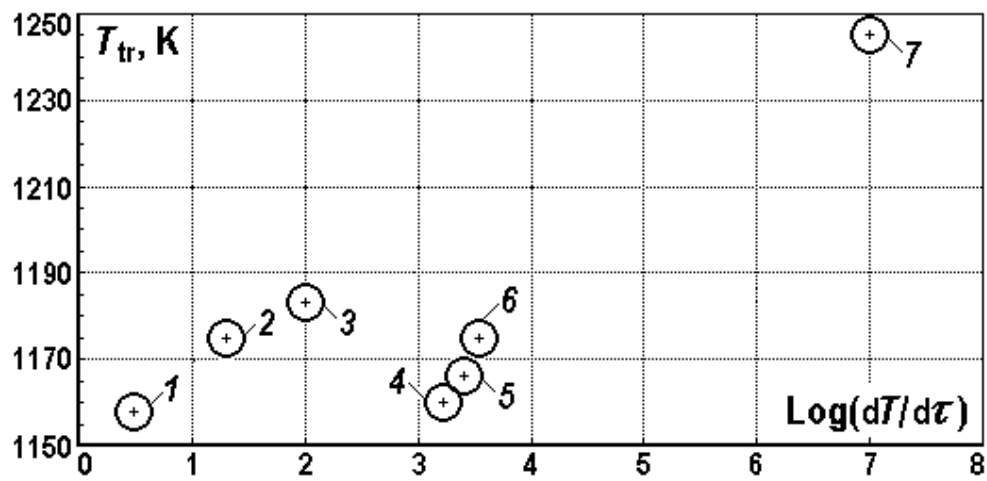


Fig. 1. Influence of speed of heating on temperature of polymorphic transformation: 1 – [4], 2 – [5], 3 – [6], 4 – [7], 5 – [8], 6 – [9], 7 – [10].

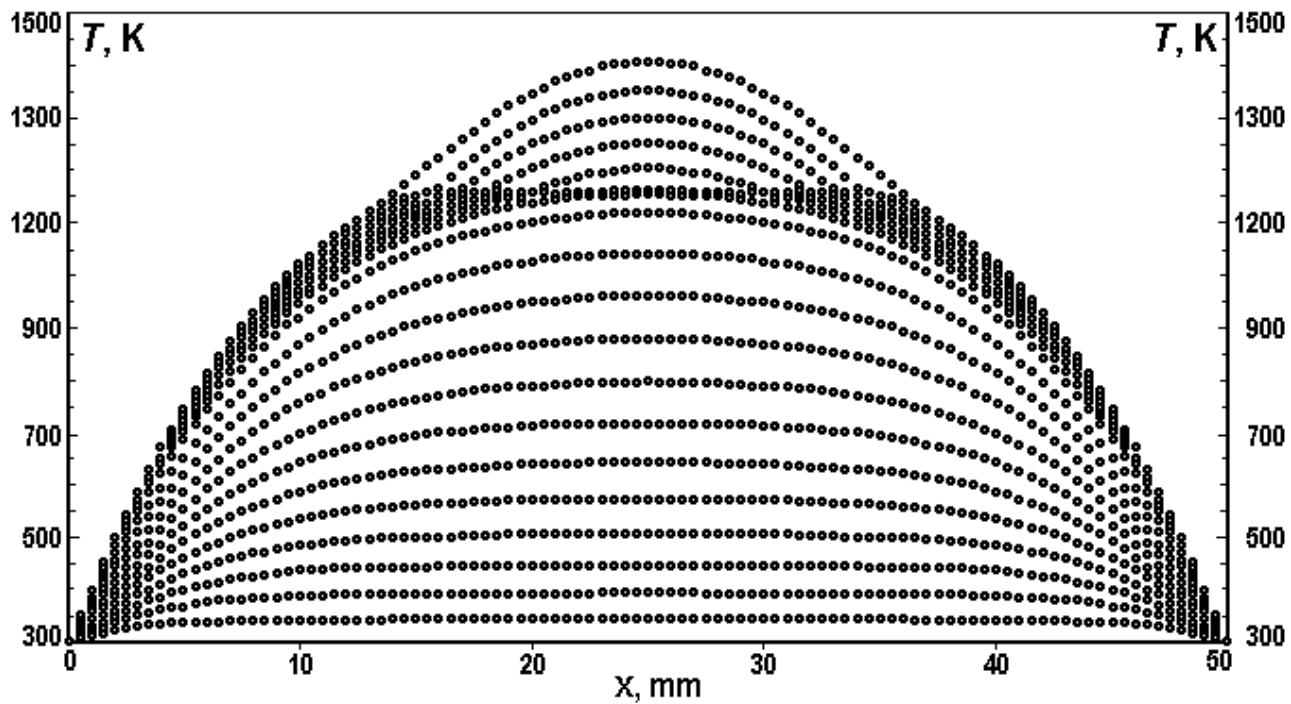


Fig. 2. Temperature fields on a sample during its heating.

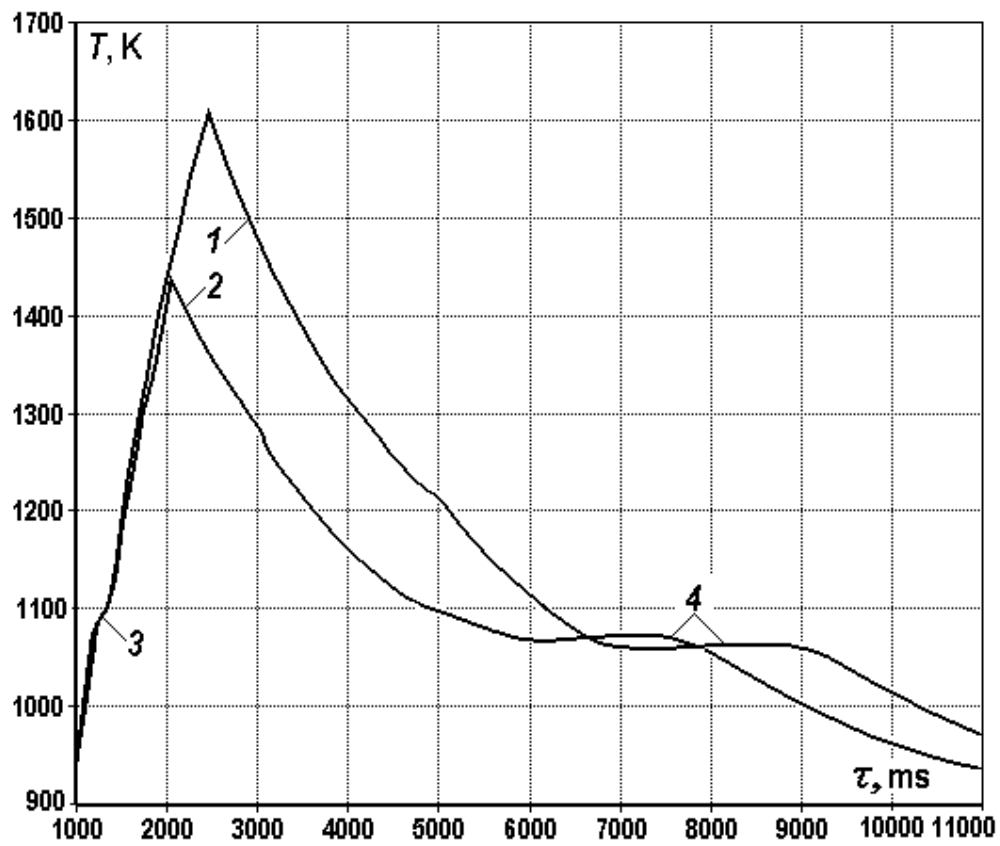


Fig. 3. Thermogram of heating and cooling of a sample: *1* - first heating; *2* - second heating; *3* - phase transformation at heating; *4* - phase transformation at cooling.

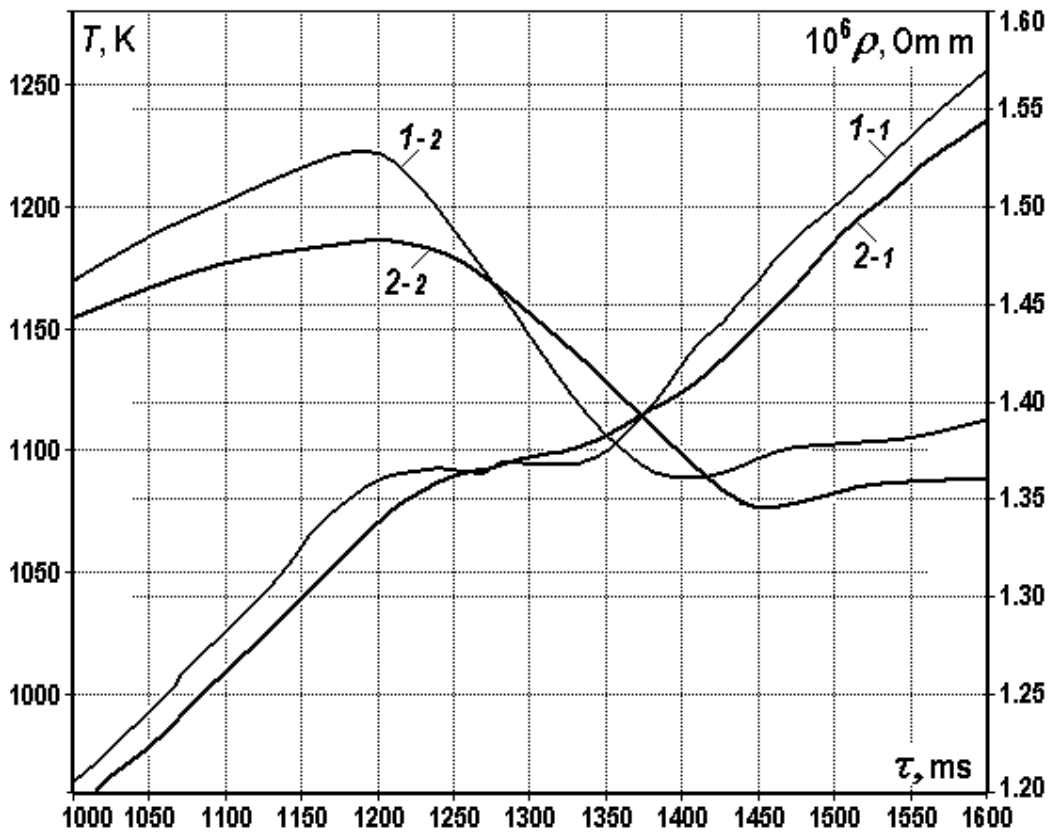


Fig. 4. Change of temperature and electroconductivity in time in a zone of phase transformation at heating: *1-1* - change of temperature at the first heating; *2-1* - change of temperature at the second heating; *1-2* - change of electroconductivity at the first heating; *2-2* - change of electroconductivity at the second heating.

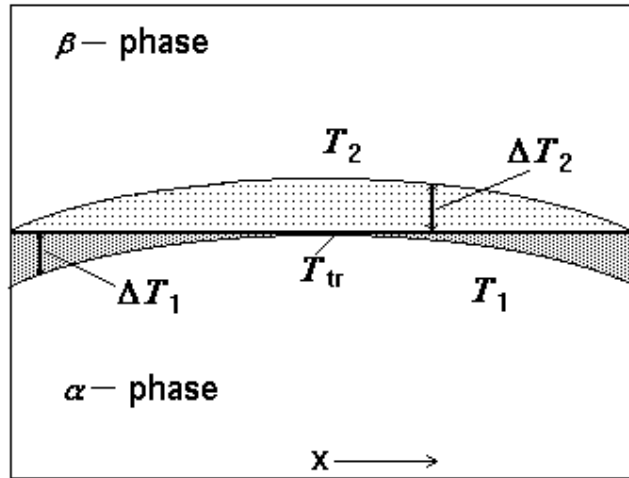


Fig. 5. Phase transformation passage on non-isothermal specimen.