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Calorimetry of amorphous alloy crystallization processes based on quasi-binary system TiNi−TiCu

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The effect of the degree of polynomial smoothing of experimental data from differential scanning calorimetry of amorphous metal alloys $Ti_{50}Ni_{25}Cu_{25}$, $Ti_{30.2}Ni_{49.8}Hf_{20}$, $Ti_{40.5}Ni_{49.8}Zr_{9.7}$, $Ti_{50.2}Ni_{24.8}Cu_{24.4}Al_{0.6}$, $Ti_{50.2}Ni_{24.8}Cu_{24.4}Fe_{0.6}$ at the defined values of exothermic effects in crystallization was studied. It was shown that the values of thermodynamic parameters obtained from this data practically do not depend on the degree of polynomial smoothing. However, such procedure does impact, and quite substantially, the kinetic characteristics of the crystallization process of amorphous metal-metal alloys when they are heated. Enthalpy of the crystallization process of such alloys turned out to be by an order less that in regular crystallization of the alloys of the same composition. The start of crystallization of these alloys may formally be described by peaking-type functions, which characterizes the initial process of crystallization as spontaneous and fast. Introduction of hard-melting components in the composition, such as Hf and Zr, somewhat suppress this trend. It is essential that upon crystallization of amorphous alloys based on the quasi-binary system TiNi−TiCu the temperatures of maximum rate of crystallization center origination and maximum rate of their growth practically coincide with each other, which is not observed during normal crystallization from alloys.

Keywords: amorphous state, enthalpy, entropy, peaking-type functions.

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1. Introduction

Calorimetric studies by method of differential scanning calorimetry (DSC) in most alloys of quasi-binary system TiNi−TiCu, usually do not end with the definition of such thermodynamic characteristics of the crystallization process as enthalpy and entropy of this phase transition (see, for example, [1–5]). Even if there is information on the thermal effect of transformation, further definition of the phase transition enthalpy and the more so entropy is not done [5,6]. Paper [7], which attempted to define these characteristics in some alloys based on quasi-binary system TiNi−TiCu, should be treated as a certain exception. However, the common patterns in crystallization processes in such amorphous alloys were not considered there. It should also be noted that in amorphous metal alloys (AMS) of the metal-metal system the patterns of the crystallization processes are studied a far smaller degree than in the study of AMS crystallization of the metal–metalloid system (see [6,8]).

The common in the study of the crystallization processes with the DCS method with AMS heating is the tendency to exclude from the consideration the random and hard-to-control factors that may impact the significant part of the measurements, while accounting for the basic component of the measurements. One of such techniques is approximation of experimental data by a polynomial of the first or higher degree. It is deemed that this approach is based on the fact that this procedure practically does not distort many characteristics defined from the DSC data, such as points of transformation start and end, temperature of maximum rate of endothermic or exothermic processes, temperature intervals of phase change courses, thermal effects of phase transformation etc. Nevertheless, we found no direct evidence of such statement. We will estimate the validity of this statement using various amorphous alloys of TiNi−TiCu system as examples. As it turned out, so far it has not been discussed whether such transformation of initial experimental data impacts the interpretation of the kinetic features of phase transformation during AMS crystallization. This study is dedicated to clarification of these issues.

2. Study method

Metal–metal AMS systems with the following composition were the object of the study: $Ti_{50}Ni_{25}Cu_{25}$ (molar weight 54.50 g/mol), $Ti_{30.2}Ni_{49.8}Hf_{20}$ (molar weight 79.4 g/mol), $Ti_{50.2}Ni_{24.8}Cu_{24.4}Al_{0.6}$ (molar weight 54.26 g/mol), $Ti_{50.2}Ni_{2524.8}Cu_{24.4}Fe_{0.5}$ (molar weight 54.57 g/mol), $Ti_{40.5}Ni_{49.8}Zr_{9.7}$ (molar weight 57.53 g/mol).

The alloys were obtained by planar casting. The cooling rate is $\sim 10^6$ K/min. Tape thickness is $30-40\,\mu$mm.

The differential scanning calorimetry was carried out using analyzer STA „Jupiter“ 449 by Netzsch. Heating and cooling were performed at the rate of 5, 10, 20 and 40 K/min in the argon environment (99.998 % Ar). The gas flow rate was 25–30 ml/min. The masses of the specimens were within the range of 15–20 mg.

Proteus Analyses software and the Fityk package were used for processing of experimental DSC data, including the determination of critical point temperatures.

As known, AMS are most often an elastic tape with thickness of several tenths of micrometers. For manufacture of a charge in a crucible with diameter of 4 mm and height of 4 mm, the wafers of random geometry are cut from an amorphous tape, laid until the crucible volume is filled, and the crucible is closed with a cap having holes. Inside the crucible, there is no quality contact for heat transfer between wafers. This, in particular, impacts the registration of the thermal effect by the system of its measurement: one of the consequences — oscillation of the recorded values of the heat flux. To smoothen such oscillations, Proteus Analyses software includes a presentation of experimental data by polynomials of 0–8th degree. This paper considered the effect of such approximation on the results of experimental data processing by the polynomial of zero (without smoothing), fourth and eighth degree.

3. Experimental results and discussion

Figure 1, *a* shows the effect of the heating rate at the view of the DSC signal for the alloy with basic composition $Ti_{50}Ni_{25}Cu_{25}$. The so-called reduced heat capacity $C_{pexcess}$ is plotted on the ordinate axis, which comprises the DSC analysis data normalized to the heating rate (β) and mass (m_s) of the specimens (see [9]):

$$C_{p_{excess}} = \frac{\dot{Q}_s - \dot{Q}_{\mathrm{Bl}}}{m_s \beta}\,(\mathrm{Jg^{-1}K^{-1}}). \qquad (1)$$

Here $\dot{Q}_S$ and $\dot{Q}_{\mathrm{Bl}}$ — heat fluxes in heating with a specimen and without a specimen, accordingly.

As the heating rate increases, all specific points of exothermic crystallization process are displaced in the area of higher temperatures: temperatures of the start and end of transformation and the temperature of maximum rate of crystallization process development. The latter makes it possible, using Kissinger's method, [10] to evaluate the effective energy of activation of this process, E_a. It turned out to be equal to 500 ± 20 kJ/mol.

As the heating rate increases, the temperature interval of phase transformation increases, too: from 13° for heating with the rate of 5 K/min to 50° for heating rate of 40 K/min. As the heating accelerates, enthalpy and entropy of the crystallization process increase slightly. However, such changes are less than the errors of definition of the actual thermal effect of transformation using the surface area of the exothermic signal. Thus, the average value of enthalpy for all heating rates, ΔH_{av}, is equal to 1270 ± 80 J/mol; entropy, ΔS_{av} — 1.7 ± 0.1 J/(mol · K).

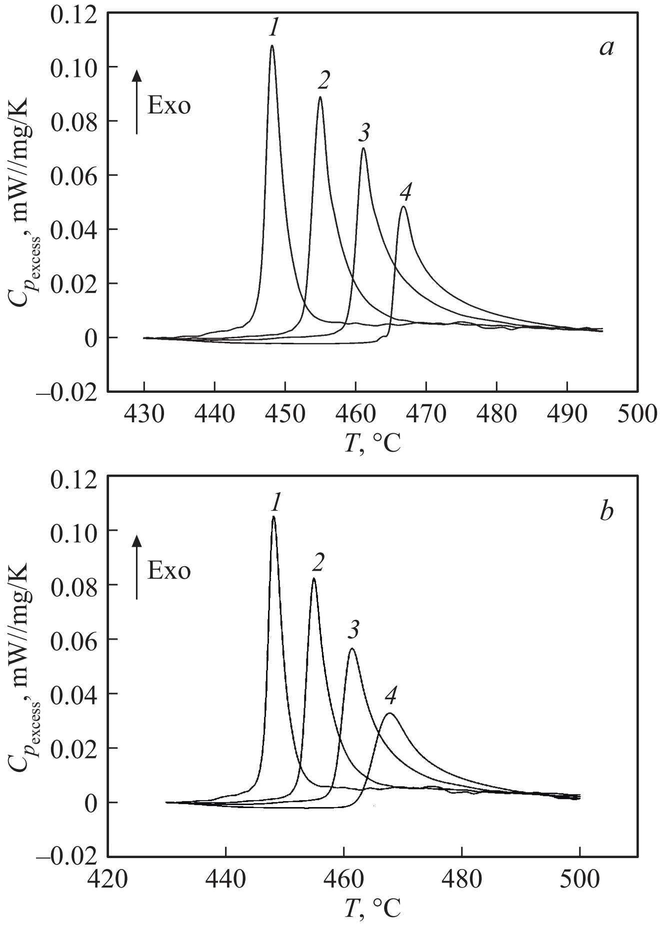

Figure 1. Effect of heating rate at change of DSC signal in AMS heating $Ti_{50}Ni_{25}Cu_{25}$: *a* — without DSC approximation; *b* — with DSC approximation. Heating rate: curve *1* — 5, *2* — 10, *3* — 20, *4* — 40 K/min.

One of the known methods to determine the type of phase transformation using DSC data is (see [11–15]) comparison of temperatures of the DSC signal maximum and maximum of its second derivative with respect to temperature. For this specific case this deviation does not exceed 0.1° for all heating rates. In this case the transformation should be deemed the phase transition of the first order (FT-I).

There is another approach to the consideration of phase transformation mechanisms, characterizing DSC curves in the heating mode by the reaction order parameter n at this phase transition [10]. The order of reaction n can be determined from the ratio presented in [10]:

$$n = 1.26\sqrt{S}. \qquad (2)$$

Here S is the shape factor of the DSC curve in the region of the thermal effect registration temperatures: the ratio of the tangent slope tangents at the inflection points of the experimental DSC dependence.

Shapes of differential thermal analysis [15] and DSC [16–18] curves were theoretically calculated for different mechanisms of phase transformation: diffusion, reaction at the phase interface, nuclei growth process etc. For the first two mechanisms of them the order of reaction is $n < 1$ taking into account the form factor. $n \rightarrow 1.3$ for the process controlled by nucleus origination and growth. In this case, the dependence $d\alpha/dT(T)$, where $d\alpha/dt$ is reaction rate, passes through a maximum and is ideally a symmetric curve $(S = 1)$.

The attempt to present the change in the DSC signal during crystallization with the heating rate 5 K/min in Fityk software failed, since in the temperature range of this process, 13°, only several measurement points are found. Therefore, this procedure was carried out for heating of the alloy with the rate of 40 K/min, when the temperature interval of transformation is significantly wider (50°). The result of such analysis is shown in Figure 2, *a*. You can see that the exothermic effect of this transformation may be presented as a superposition of two inputs. Logically the input from the process P_1 should be referred to the origination of crystallization centers, and from P_2 — to the growth of such centers. It bears noting that the temperatures of maximum rate of such processes differ from each other by fractions of a degree. Estimation of the order parameter, for example, for the heating case with the rate of 40 K/min provides value $n \rightarrow 10$. This several times exceeds value *n*, which follows from analysis of DSC data (see [15–18]). Cause — high asymmetry of DSC exothermic signal shape. The start of the process of alloy transition from the amorphous state to the crystalline one corresponds to the leap of the first derivative of the DSC signal by temperature (process rate). Therefore, the dynamics of DSC signal change at the moment of crystallization start may be presented as a peaking-type function [19]: (peaking-type modes (blow-up) — where values change due to non-linear sources in the mode of unlimited growth within the finite time), i. e. the process of crystallization of the amorphous phase is of blow-up nature. This may be interpreted as follows: upon achievement of certain temperature the main mass of the amorphous phase undergoes a transition to a new state due to spontaneous appearance of a huge number of new crystallization centers, and only then their growth is implemented at the end stage of the amorphous matrix transition to the crystalline one.

Figure 1, *b* shows the estimated effect of heating rate at the change of the DSC signal when it is presented as a fourth-degree polynomial. As you can see from the given data, the above patterns of heating rate effect on the DSC data are maintained in this case. The crystallization process maximum temperature differs by less than 1° from the initial state, without approximation. The same is true for the enthalpy and entropy values of the processes at different heating rates. Moreover, the effect of the heating rate at the crystallization development temperature interval is reproduced with the precision of up to several degrees.

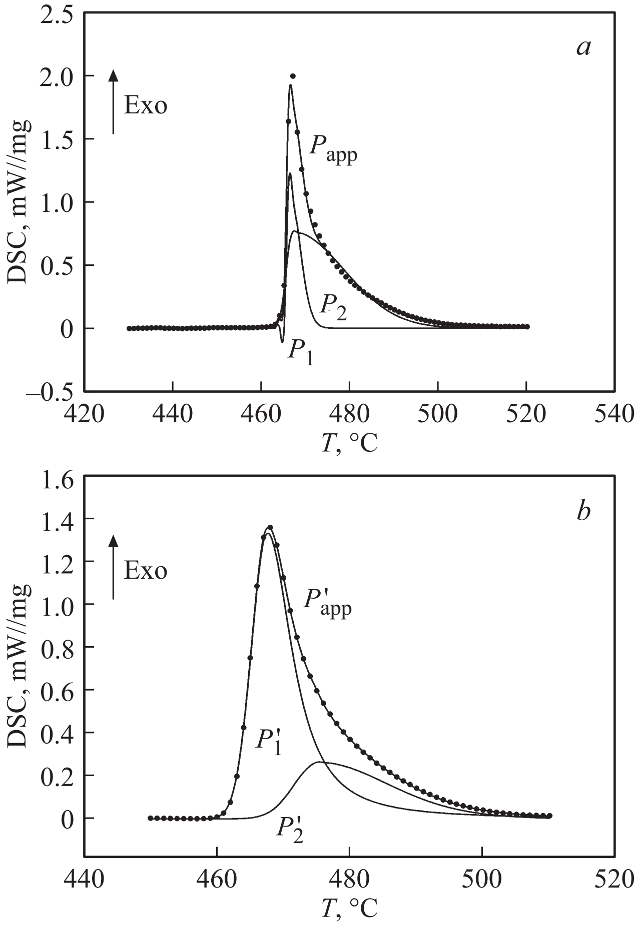

Figure 2. Structure of exothermic effect when heated with the rate of 40 K/min, AMS $Ti_{50}Ni_{25}Cu_{25}$: *a* — without DSC approximation; *b* — with DSC approximation. Points — experimental data; P_{app} (P'_{app}) — approximation result; P_1 (P'_1) and P_2 (P'_2) — subpeaks.

When the DSC signal is approximated with polynomial of the 4th degree, the shape of the DSC signal changes in the region of exothermic effect registration temperatures (Figure 2, *b*). The exothermic peak in this case is much less asymmetric than without approximation. The parameter of the reaction order *n* has the value 2.7 and does not correspond to the data of DSC analysis in different literature sources. From the results given in Figure 2 it follows that the crystallization process may be imagined as a superposition of two subprocesses: origination of nuclei and their subsequent growth. Without approximation (Figure 2, *a*) the temperatures of the maximum rate of these two processes ($T_{1\text{m}}$ and $T_{2\text{m}}$) coincide with each other (468.4 °C).

The input of each of the processes organizing the total exothermic effect, at the heating rate of 40 K/min: process P_1 — $\Delta H_{P1} = 316$ J/mol, $\Delta S_1 = 0.4$ J/(mol · K); process P_2 — $\Delta H_{P2} = 1020$ J/mol, $\Delta S_{P2} = 1.4$ J/(mol · K).

The input of each of the processes organizing the total exothermic effect, at heating rate 40 K/min (approximation

Table 1. DSC data analysis results

Degree of polynomial	Characteristic	$Ti_{50.2}Ni_{24.8}Cu_{24.4}Fe_{0.6}$	$Ti_{50.2}Ni_{24.8}Cu_{24.4}Al_{0.6}$	$Ti_{50}Ni_{25}Cu_{25}$	$Ti_{30.2}Ni_{49.8}Hf_{20}$	$Ti_{40.5}Ni_{49.8}Zr_{9.7}$
0	E_F, kJ/mol	470 ± 20	470 ± 30	500 ± 20	650 ± 30	680 ± 200
	n	> 100	> 100	$\gg 10$	$\rightarrow 5$	$\rightarrow 3$
	ΔH_{av}, J/mol	1470 ± 50	1250 ± 70	1270 ± 80	780 ± 40	760 ± 80
	ΔS_{av}, J/(mol · K)	2.0 ± 0.1	1.7 ± 0.1	1.7 ± 0.1	1.0 ± 0.1	1.0 ± 0.1
	T_{1m}, °C	466.1	468.4	466.0	534.1	499.1
	T_{2m}, °C	466.3	468.5	466.9	534.4	499.9
	ΔH_{P1}, J/mSol	256	520	316	322	321
	ΔP_1, J/(mol · K)	0.3	0.5	0.4	0.4	0.4
	ΔH_{P2}, J/mol	1280?	790	1020	570	770
	ΔS_{P2}, J/(mol · K)	1.7	0.8	1.4	0.7	1.0
4	E_F, kJ/mol	470 ± 20	470 ± 30	500 ± 20	650 ± 30	680 ± 200
	n	2.5	2.2	2.7	1.7	2.0
	ΔH_{av}, J/mol	1580 ± 100	1230 ± 60	1310 ± 200	790 ± 60	760 ± 60
	ΔS_{av}, J/(mol · K)	2.2 ± 0.1	1.7 ± 0.1	1.8 ± 0.3	1.0 ± 0.1	1.0 ± 0.1
	T'_{1m}, °C	469.4	468.9	467.6	535.3	499.5
	T'_{2m}, °C	481.6	471.9	485.2	546.5	503.9
	$\Delta H_{P'1}$, J/mol	1160	610	480	740	390
	$\Delta S_{P'1}$, J/(mol · K)	1.6	0.7	1.3	0.9	0.5
	$\Delta H_{P'2}$, J/mol	360	840	370	170	500
	$\Delta S_{P'2}$, J/(mol · K)	0.5	0.9	0.4	0.2	0.6
8	E_F, kJ/mol	470 ± 20	470 ± 30	500 ± 20	650 ± 30	680 ± 200
	n	1.5	1.4	1.5	1.3	1.8
	ΔH_{av}, J/mol	1450 ± 100	1190 ± 80	1380 ± 100	760 ± 60	700 ± 50
	ΔS_{av}, J/(mol · K)	2.0 ± 0.1	1.6 ± 0.1	1.8 ± 0.1	0.9 ± 0.1	0.9 ± 0.1

of DSC data with polynomial of the fourth degree), may be imagined as follows: process $P'_{1,}$ — $\Delta H_{P'1} = 480$ J/mol, $\Delta_{S1} = 1.3$ J/(mol · K); process $P'_{2,}$ — $\Delta H_{P'2} = 370$ J/mol, $\Delta S_{P'2} = 0.4$ J/(mol · K). Such low entropy value may indirectly indicate mild shuffling of atoms in the process responsible for exothermic effect P'_2.

Figure 3 shows the effect of heating rate at the change of the DSC signal when it is presented as a 8th-degree polynomial. As you can see from the given data, the above patterns of heating rate effect on the DSC data are maintained in this case. The crystallization process maximum temperature differs by less than 1° from the initial state. However, in this case for the heating with the rate of 40 K/min it is not possible to correctly analyze the components of the thermal effect. Therefore, the data was averaged for the heating rates 5–10–20 K/min. The following results were obtained: $\Delta H_{av} = 1380 \pm 100$ J/mol · K, $\Delta S_{av} = 1.8 \pm 0.1$ J/(mol · K), $n = 1.5$. The differences in the average values of enthalpy and entropy of phase transformation from the initial state in this case are in the range of 5 ± 1 %. For approximation by the fourth-degree polynomial this difference does not exceed 1–2 %.

Analysis of the DSC data for alloys $Ti_{30.2}Ni_{49.8}Hf_{20}$, $Ti_{50.2}Ni_{24.8}Cu_{24.4}Al_{0.6}$, $Ti_{50.2}Ni_{24.8}Cu_{24.4}Fe_{0.6}$, $Ti_{40.5}Ni_{49.8}Zr_{9.7}$ shows practically complete quality match with the data obtained for alloy $Ti_{50}Ni_{25}Cu_{25}$. The results of the study of these alloys are given in Table 1.

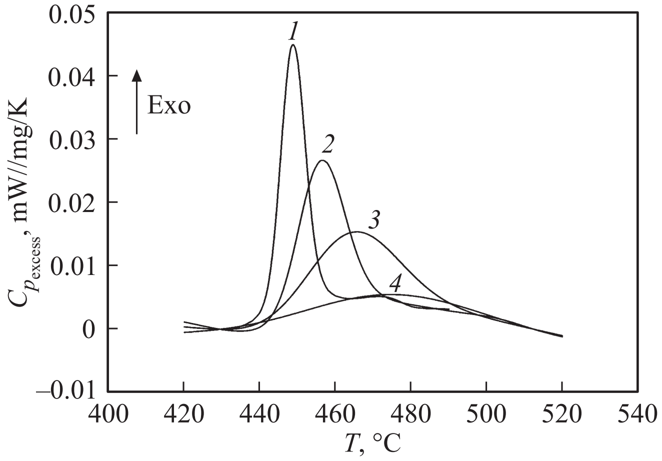

Figure 3. Effect of heating rate at change of DSC signal in AMS heating $Ti_{50}Ni_{25}Cu_{25}$ — with DSC approximation by the 8th-degree polynomial. Heating rate: *1* — 5, *2* — 10, *3* — 20, *4* — 40 K/min.

As for the effect of the degree of polynominal approximation of the source data at the definition of the reaction order parameter *n*, as this degree increases, the DSC signal asymmetry decreases in the registration of the thermal effect of crystallization without a major change in the value of enthalpy and entropy of such phase transformation.

Table 2. Values of enthalpy and entropy in regular crystallization of the studied alloys

Alloy	ΔH^*, kJ/mol	ΔS, J/(mol $\cdot$ K)
$Ti_{50}Ni_{25}Cu_{25}$	15.3	8.7
$Ti_{30.2}Ni_{49.8}Hf_{20}$	18.7	9.4
$Ti_{50.2}Ni_{24.8}Cu_{24.4}Al_{0.6}$	15.2	8.8
$Ti_{50.2}Ni_{24.8}Cu_{24.5}Fe_{0.5}$	15.2	8.8
$Ti_{40.5}Ni_{49.8}Zr_{9.7}$	16.7	9.1

Providing that the process P_1 is related to origination of the crystallization centers, and the process P_2 — with their further growth, it follows from the data given in Table 1 that the thermal effect of the first process is less than that of the second one. Accordingly, the entropic characteristics of this phase transition are correlated. A relatively low entropy value should be noted. Indirectly it may be interpreted as a low degree of atom shuffling in implementation of the first crystallization stage. The latter may be a certain explanation for the assumption on the reason for the change of the DSC signal as the peaking-type function.

However, in approximation of the experimental data by the fourth-degree polynomial (Table 1) the noted pattern changes its sign. At the same time the difference between the temperature of the maximum rate of the crystallization center origination and the temperature of the maximum rate of their growth increases in this type of phase transformation. Approximation of experimental data with the eighth-degree polynomial maintains this trend, and the difference in the temperatures of such maxima increases.

The smoothing degree of DSC experimental data within the errors of their measurement has no principal effect on the average values of enthalpy and entropy of crystallization of metal amorphous alloys based on the quasi-binary system TiNi–TiCu.

If you address the data on the value of enthalpy in crystallization of the base metals included in the composition of the considered amorphous alloys, these values lie in the interval of 15–25 kJ/mol [20], therefore, for entropy - 8–10 J/(mol $\cdot$ K). For crystallization from the liquid state of the compositions considered in this paper, such data is unavailable. You may attempt in the additive approach to rather roughly evaluate the possible values of enthalpy and entropy in the regular crystallization of the studied alloys. The following values were obtained, which are summarized in Table 2.

These values are close in the magnitude to the corresponding values in process of crystallization from the liquid state of the base metals and differ noticeably from the values obtained experimentally in crystallization of the amorphous alloys of the same composition (Table 1).

Hence it appears that in process of regular crystallization of the base element alloys, the enthalpy of crystallization of the amorphous alloy is less than this value by an order or more. And for entropy values — more than 5 times. The reason for this is related, seemingly, to the high thermodynamic stimulus of transformation and presence of the significant quantity of „frozen“ crystallization centers in the amorphous alloy [21,22]. Both of these factors are substantially less pronounced in the regular crystallization of metal alloys. However, such trend is observed in this case as well [23].

4. Conclusion

Estimation of the thermodynamic characteristics of AMS crystallization, such as activation energy of crystallization process, enthalpy, entropy, maximum rate of process development etc., practically does not depend on the presentation of the experimental data by the polynomial of zero or higher degree. However, the method of experimental data smoothing distorts the estimation of the kinetic characteristics of the studied phase transformations.

The process of metal-metal amorphous alloy crystallization always includes two components: origination of crystallization centers and their subsequent growth. The initial stage of crystallization in heating of AMS on the basis of quasi-binary system TiNi–TiCu (metal–metal) in the kinetic plan may be described by the peaking-type functions. The manifestations of such kinetic effect are especially strongly affected by small, less than 1 at.%, additives of aluminum or iron.

Relatively low values of enthalpy and entropy of phase transformation compared to these characteristics in crystallization of the alloys with similar composition, may serve as indirect, integral proof of existence of many „frozen“ crystallization centers in the X-ray amorphous AMS.

It is essential that upon crystallization of the studied alloys the temperatures of maximum rate of crystallization nuclei origination and maximum rate of crystal growth coincide with each other, which is not observed during normal crystallization from alloys. Therefore, it may be assumed that a more pronounced degree of the structural organization of metal alloys in amorphous state exists, contrary to the models of the dense random packing of liquid.

Substitution of copper in the quasi-binary system TiNi–TiCu with hard-melting components, hafnium or zirconium, increase the activation energy value of the crystallization process, enthalpy and entropy of phase transformation and somewhat levels it out at the initial stage of the blow-up nature of crystallization.

Effect of the small additives of iron or aluminum on the manifestation of the blow-up nature of the initial crystallization stage is seemingly the consequence of the fact that their presence promotes complete amorphization of such alloys at superfast cooling (see, for example, [5]).

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Conflict of interest

The authors declare that they have no conflict of interest.

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