MEANS FOR MEASURING THE THERMAL QUANTITIES

ELECTROKINETIC PARAMETERS AND THERMAL STABILITY OF MULTICOMPONENT METALLIC GLASSES

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Abstract. The paper analyzes the results of research on the temperature coefficient of resistance, thermo-e.m.f. and the thermostability of multicomponent metal meshes depending on the presence of an alloying component, with the aim of applying them in electrical thermometry.

Keywords: Metallic glasses, Metallic amorphous alloys, Amorphous state, Crystalline state, Alloying component, Electrical thermometry.

1. Introduction

Metallic amorphous alloys demonstrate several advantages over their crystalline counterparts, including high strength, improved corrosion resistance, and optimal magnetic properties. However, the metastability of amorphous structures makes them sensitive to thermal and mechanical influences, which limits their use in complex engineering applications, especially at high temperatures. Production and application require an understanding of the fundamental principles that govern their formation, structural dynamics, and thermal stability. Critical factors affecting the formation and stability of the amorphous state include thermodynamic, kinetic, and physicochemical aspects. Although research in this area is actively developing, the effects of alloy composition, cooling conditions, and external factors on the thermal stability of metallic amorphous materials remain subjects of intensive scientific investigation. Our study is aimed at a systematic analysis and evaluation of the mechanisms affecting the structural stability of metallic amorphous alloys in the context of their long-term application in engineering systems at high temperatures. The approach used in this study includes advanced experimental techniques and theoretical analysis, providing an understanding of the properties and application possibilities of amorphous metallic alloys.

2. Drawbacks

Methodological constraints. The research is based on a comprehensive approach; however, it may be limited by methods that do not take into account recent developments in solid-state physics and electrokinetic phenomena. This could lead to the oversight of potential findings.

Mismatched conditions. Real operational conditions can significantly differ, thus results obtained in laboratory settings might not fully correspond to actual operational conditions.

Measurement errors: There is a risk of specific errors not related to the inaccuracies of measuring instruments. These errors can affect the accuracy of results and their interpretation.

Insufficient attention to long-term stability: For a complete understanding and application of metallic amorphous alloys, it is important to further investigate the long-term stability of these materials at high temperatures.

3. Goal

The goal of the research is to create a concept and develop primary thermotransducers with improved metrological and operational characteristics based on new materials, which contributes to the development of practices for creating precise temperature measurement instruments.

4. Research methodology

The methodological foundation of the scientific research is a comprehensive approach to analyzing the stability of metrological characteristics of temperature measuring instruments from the perspective of stabilizing the thermometric properties of materials in response to the deviating effects of structural and operational factors. The theoretical analysis conducted in the dissertation is based on the principles of solid-state physics, particularly the theory of electrokinetic phenomena in metals and alloys, thermodynamics, materials science, the theory of elasticity and plasticity of materials, theoretical foundations of special foundry technologies, methods of increasing the accuracy of measuring instruments, the theory of errors and processing of measurement results, and certain sections of the theory of electrical circuits and the theory of statistical research. Experimental studies of the electrophysical properties of primary thermotransducer materials

and their stability depending on operational factors are carried out using original methodologies.

While working on this research, there was a need to develop a complex for researching the electrophysical properties of both new thermo-electrode materials and for verifying standardized temperature-measurement instruments, due to the absence of domestic systems for this purpose. To conduct the research, a universal computerized complex was created, which can also be applied in the network of metrological laboratories in Ukraine, scientific research institutes, special design bureaus, and enterprises engaged in the development, manufacturing, operation, and metrological certification of primary thermometry instruments.

The complex includes: a personal computer (PC); standard temperature measurement instruments covering the range of 4.2–1800 K; UTT6-VMC device; interface unit with PC; furnace control unit; MTP-2M type furnace unit; steam TP-5 and zero TN-12 thermostats; a measurement cell. External devices of the complex are connected to the computer via their adapters.

The measurement cell is designed to study the temperature dependence of electrical resistance, temperature coefficient of resistance, and thermo-electromotive force (thermo-e.m.f.) of samples within the range of 4.2–1200 K, influenced by operational factors.

Unlike electrical resistance, where the measurement error using a four-wire scheme is at the level of the potentiometric method error, the measurement of thermo-e.m.f. involves specific errors not associated with the errors of measuring instruments. These primarily constitute the major portion of error when measuring thermo-e.m.f.

Additional research involved integrating machine learning (ML) for analyzing material properties and the influence of structural operational factors on the stability of thermometric characteristics. The use of advanced analytical models, based on ML and data collected using the measurement cell, facilitated the analysis of the temperature dependence of electrical resistance, thermo-electromotive force (thermo-e.m.f.), and the determination of optimal parameters under various operational influences. It became possible to analyze the impact of adding alloying elements, modifying cooling regimes, and the influence of mechanical stresses on the stabilization of thermometric properties. Crucially, these models were employed to identify and analyze subtle dependencies that might be overlooked by traditional analysis methods but can affect the stability of measurements during operation.

5. Analysis of the study results on electrokinetic parameters

Analyzing the results of our studies, we can conclude that binary metallic glasses are less thermally stable compared to multicomponent ones. However, alloying them with a certain amount (approximately 3 at. %) of a third component significantly enhances the stability of the electrophysical properties of many metallic alloy systems and increases their tendency to transition into an amorphous state. This work examines the results of studies on metallic glasses with the following compositions: Ti-Cu-Co, Ti-Cu-Ni-Si, Ti-Cu-Co-Si.

Regarding the aforementioned materials, as evident from Fig. 1, they all exhibit a negative temperature coefficient of resistance (TCR), which is typical for this class of materials with a specific resistance above 150 μΩ·cm. In the range from nitrogen (77.35 K) to room (300 K) temperatures, the electrical resistance of the materials studied changes almost linearly with temperature, with a notable non-linearity in the temperature dependence of resistance from helium (4.2 K) to nitrogen temperatures. These anomalies, in our view, are caused either by the Kondo magnetic effect [1] or tunneling scattering [2], as they are specific manifestations of the amorphous structure. Concerning tunneling scattering in the first approximation – it refers to two-level energy state systems; the existence of which requires a certain degree of local order combined with a large number of atoms involved in the process, which reduces the energy imbalance.

In the case of metallic glasses, there's an additional likelihood that these two-level systems interact with conduction electrons. Likely, as the temperature is lowered, this circumstance leads to scattering effects, equivalent to the Kondo effect. These are realized in the form of a potential well with two energy minima of the same level, so that the primary states are symmetric and antisymmetric tunneling states, separated by a gap of 2Δ (~0.2 meV).

The temperature dependence of resistance can then be described by an expression similar to the Kondo-type formula:

$$\rho(t) = -c \ln\left(k_B^2 T^2 + \Delta^2\right), \qquad (1)$$

where Δ<1 K.

The tunneling model for the materials under study suggests their negative TCR. The high value of specific electrical resistance and similarity with the universal Muij correlations allow us to conclude the presence of saturation effects in metallic glasses, hence rendering traditional theories for analyzing their electrokinetic properties unsuitable.

An important finding is the observed effect of alloying on TCR of the materials studied. For instance, upon alloying (Fig. 1, Table 1) metallic glasses composed of Ti-Cu-Co with silicon, the absolute value of TCR increases from $1.372 \cdot 10^{-4}$ K^{-1} to $-2.167 \cdot 10^{-4}$ K^{-1}, and replacing cobalt with nickel leads to a decrease in the absolute value of TCR. This phenomenon is explained by the influence of average valence electron concentration of the alloying components.

From a practical application perspective, an important property of metallic glasses is the ability to control their TCR by adjusting the concentration of the base components and quenching conditions [3].

High-temperature studies of the Ti-Cu metallic glass system, alloyed with nickel, cobalt, and silicon, have shown that the absolute value and sign of their TCR are the same as those in the low-temperature range, namely: the resistance linearly decreases with increasing temperature up to the onset of crystallization temperatures.

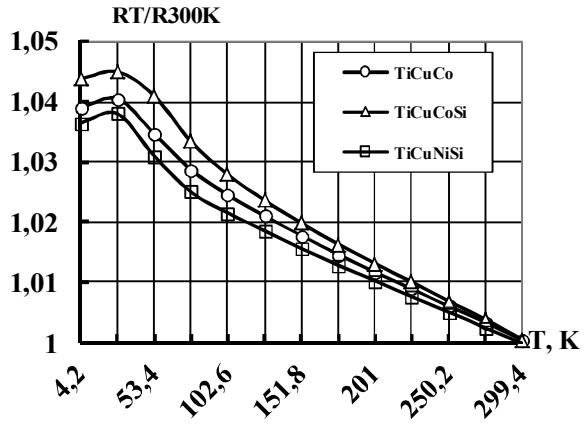

Fig. 1.The impact of alloying on the TCR of the materials studied

Analyzing the resistance values of the samples in both amorphous and crystalline states, it is important to note that in the amorphous state, the predominant contribution to the electrical resistance comes from structural disorder.

Table 1. Electrophysical properties of the studied samples

Alloy	TKO, 10^4 K^{-1}	$T_{кр.}$, K	$R_{273}/R_{4.2}$	$R_A/R_{кр.}$	ρ_{273}, μΩ·см
Ti-Cu-Co	–1.372	710	0.956	1.55	160
Ti-Cu-Co-Si	–2.167	700	0.943	1.53	160
Ti-Cu-Ni-Si	–1.221	720	0.959	1.67	160

The absolute thermo-electromotive force S of the samples was explored and determined relative to a copper Cu standard in the range of 4.2–500 K. An integral method was used: one junction of the sample and the Cu standard was thermostatted, while the temperature of the other was varied, measuring its value in the examined range using a copper-constantan (type T) thermocouple, simultaneously measuring the e.m.f. of the formed thermocouple. The derivative of the measured voltage – the differential thermo-e.m.f. of the thermocouple formed by the studied sample and the Cu standard (Fig. 2). The absolute thermo-e.m.f. of the studied sample is the difference between the absolute thermo-e.m.f. of the Cu standard and the differential thermo-e.m.f. of the thermocouple formed by the studied sample and Cu standard (Fig. 3). The error in determining the $S(T)$ dependence does not exceed ±0.075 μV/K.

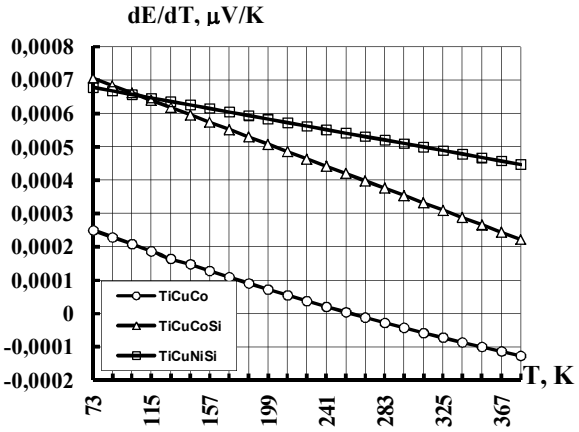

Fig. 2. Differential thermo-e.m.f. of thermocouples formed by the studied samples relative to the Cu standard

In Fig. 2, the differential thermo-e.m.f. of thermocouples formed by the studied samples and the Cu standard are almost indistinguishable from each other (maximum within 0.6–0.7 μV/K), implying that their absolute thermo-e.m.f. is nearly identical, a typical example of which is shown in Fig. 3. The $S(T)$ dependence for the studied samples is approximated by a polynomial: $S(T)=\sum_i^n A_i T^i$. The coefficients A_i of the approximation polynomial for each of the studied materials are provided in Table 2.

From the results of the conducted studies, it follows that unlike the TCR, the thermo-e.m.f. of the studied materials is practically independent of the presence and type of alloying component in the alloy. However, the thermo-e.m.f. of metallic glasses is quite sensitive to their structural state.

Curve 1 (Fig. 3) – the thermo-e.m.f. of the metallic glass composition Ti-Cu-Co-Si is typical for each of the studied materials that have undergone stabilization of the structural state following a specially developed methodology [4].

Curve 2 (Fig. 3) – the thermo-e.m.f. of the metallic glass of the same composition, but which did not undergo the aforementioned stabilization. As seen from Fig. 3, the $S(T)$ dependence for the metallic glass composition Ti-Cu-Co-Si, which has not undergone stabilization of the structural state, exhibits two inflection points in the cryogenic temperature region, which in our view is caused by a permanent transformation of the structural state triggered by heating.

Starting from 200 K and higher, the character of the $S(T)$ dependence for this alloy becomes similar to that of the structurally stabilized sample, but the absolute values are different.

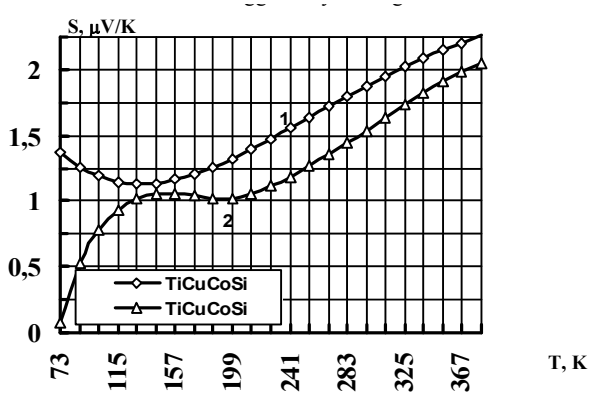

Fig. 3. Temperature dependence of the absolute thermo-e.m.f. of the studied samples

Table 2. Coefficients A_i of the approximation polynomial for the studied materials

Alloy	Ti-Cu-Co	Ti-Cu-Co-Si	Ti-Cu-Ni-Si
A_1	2.703	2.702	2.251
A_2	–0.028	–0.029	–0.019
A_3	$1.752\cdot10^{-4}$	$1.751\cdot10^{-4}$	$9.306\cdot10^{-5}$
A_4	$-3.76\cdot10^{-7}$	$-3.759\cdot10^{-7}$	$-1.162\cdot10^{-7}$
A_5	$2.86\cdot10^{-10}$	$2.865\cdot10^{-10}$	–

It is evident that this reflects a mismatch of the structural states of the studied materials, which could explain the apparent discrepancies in the research results of different authors who work with structurally non-identical systems. In our studies, for the purpose of accurate comparison of results, samples are processed according to the above-mentioned methodology [4].

Atomic configurations in metallic glasses can not be unequivocally and directly determined as in the case of crystalline materials. Therefore, information about their structural state is obtained by comparing experimental data with calculations based on various models [5–7]. Based on these comparisons, it is concluded that the phenomena of electrical charge transport in metallic glasses and metal melts are considerably similar [5], and additional experimental data further support this.

Thus, if the properties of liquid metals are similar to the corresponding properties of metallic glasses, it is reasonable to assume that theoretical aspects describing the phenomena of electron transport in liquid systems are also applicable to explaining electron transport phenomena in metallic glasses. Considering that the electron mean free path in a liquid is determined by the formula:

$$\frac{1}{l}+=\frac{1}{l_l}+\frac{1}{l_d}+\frac{1}{l_p-l}+\frac{1}{lp}, \qquad (2)$$

where l_l is determined by specific "liquid" scattering due to the disruption of short-range order; l_d accounts for scattering at local abrupt structural defects; l_{p-1} describes "phonon-liquid" scattering, proportional to temperature and dictated by less stringent selection rules in electron-phonon scattering; l_p is determined by conventional phonon scattering.

From this, it follows that impurities in the melt only partially modify one of the four components ($l_Д$) of the total electron mean free path, whereas in the solid state, defect scattering is commensurate with phonon scattering. Thus, in molten metals and metallic glasses, the effect of impurities on thermometric properties is significantly less than in crystalline materials, and even in single crystals.

In Ziman's theory, being Fourier-transformed, in wave vector space, the radial distribution function of atoms $G(r)$ gives the structural factor $S(k)$, which is introduced as an integral part of the structure in the expression for the electron mean free path l_p in a liquid metal:

$$\frac{1}{l_p}=\frac{3\pi}{2\sqrt{2}}\frac{z\sqrt{m}}{\hbar\sqrt{E_F^3}}\frac{1}{4k_F^4}\int_0^{2k_F}S(k)\left|t(k)\right|^2k^3dk \qquad (3)$$

where: m – mass of the electron; z – valency.

Fermi energy E_F and wave vector k_F on the Fermi surface are determined within the framework of the free electron model:

$$Å=\frac{\hbar}{2m}k_F^2=\frac{\hbar}{2m}\sqrt[3]{(3\pi N)^2}=\frac{\hbar}{2m}\sqrt[3]{(3\pi^2\frac{A}{\mu}d\cdot z)^2}, \qquad (4)$$

where: A is Avogadro's number; μ is the atomic mass; d is the density.

The matrix element of the pseudopotential $t(k)$ is determined by the ion potential, as well as the screening ability of the free electron gas, hence $t(k)$ practically does not depend on external influences. Thus, the temperature dependence of the electron mean free path in these systems is completely determined by the temperature change of the structural factor, and their electrical resistance in the free electron approximation is determined by the dependence:

$$\rho=\frac{3\pi}{2}\frac{m}{\hbar e^2E_F}\frac{V}{N}\frac{1}{4k_F^4}\int_0^{2k_F}S(k)\left|t(k)\right|^2k^3dk \qquad (5)$$

The impact of impurities on the change in electrical resistance can be assessed by the expression:

$$\Delta\rho=\frac{3\pi}{2}\frac{m}{\hbar e^2E_F}\frac{V}{N}\times \times\int_0^{2k_F}C_AC_B\left[1-S(k)\right]\left[t_B(k)-t_A(k)\right]^2k^3dk \qquad (6)$$

where: C_A and C_B are the concentrations of the base and the impurities; $t_A(k)$ and $t_B(k)$ are the matrix elements of their pseudopotentials.

The similarity of the electrokinetic properties of liquid and amorphous systems is also manifested in experimental studies of thermo-e.m.f. [8].

Within the framework of Ziman's theory for the amorphous phase, the value of thermoelectric power is determined from the dependence:

$$S = -\frac{\pi^2 k_B^2 T}{3|e|E_F}(3 - 2q - \frac{1}{2}r) \qquad (7)$$

where: $q = S(2k_F)|t(2k_F)|^2 / 4\int_0^{2k_F} |t(k)|^2 S(2k_F)^{-4} k^3 dk$;

and r is significantly determined by the energy dependence of t matrix. For the wave vector $2k_F \approx k_p$, the term – $2q$ predominates and $S>0$ while TCR < 0.

6. Analysis of the study results on thermal stability

Since metallic amorphous alloys are structurally metastable, when developing a new or choosing an existing metallic glass for applications at temperatures exceeding even 300 K, particularly in primary thermotransducers, the issue of thermos stability and influencing factors arises. Factors influencing the formation and stability of the amorphous state in metallic glasses can mainly be grouped into three main directions: thermodynamic, kinetic, and physicochemical. The latter direction is the least studied; however, it is the most effective concerning applications in electrothermometry.

Currently, based on the available literature data, it can be concluded that the structural stability and properties of metallic glasses depend on the properties of the components that make up their composition. Considering the well-known empirical dependence T_K/T_T = 0.4–0.6, it can be concluded that the most stable amorphous systems are those based on the use of metals with a high melting temperature. However, this is not entirely accurate. The results of the conducted studies show, the ratio of the transition temperature to the crystalline state and the melting temperature of the metallic glasses is not constant, but rather, it tends to increase around the middle part of the state diagram. According to [9], this is caused by the formation of intermetallic compounds with complex-packed crystalline structures in this area, more details of which classifications can be read about in [10].

Since the onset temperature of the transition of metallic glasses to the crystalline state (T_K) is important characteristic, in our view, it is more appropriate to use an empirical dependency that links the temperature T_K to the concentration dependence of the enthalpy of formation of vacancy-type defects ΔH_C [kJ/mol]: $T_K \approx (7–8)\cdot\Delta H_C$. Based on the analysis of this relationship, it can be concluded that due to the structural relaxation of the studied materials, their onset temperature of transition to the crystalline state can significantly change.

Similarly, just as the onset temperature of transition to the crystalline state, the glass transition temperature T_w can also serve as a measure of the thermal stability of metallic glasses. Thus, for the majority of metallic amorphous systems, it is true that $T_w/T \geq 0.45$, which allows obtaining an amorphous structure of materials that are quenched at rates not exceeding 10^6 K/c. Thus, the higher the glass transition temperature and the lower the melting temperature, the more stable the amorphous state.

Primarily, models of thermal stability of the amorphous state are based on the assumption that the rate of transition to the crystalline state, determined by diffusive processes, is proportional to an amount inversely proportional to viscosity. So, in the theory of entropic viscosity, the dependence of viscosity changes on temperature is determined as follows:

$$\eta = C\exp(\Delta E/ST), \qquad (8)$$

where C is a constant; ΔE is the activation energy for viscous flow; S is the configurational entropy.

In the solid state at T_K the entropy does not change significantly, and the crystallization of an amorphous alloy, as is known, occurs at the moment of reaching a critical viscosity value, which is approximately $\approx 10^{12}$ Pa·s. Thus, with an increase in the viscosity of the melt, the glass transition temperature correspondingly increases, which in turn increases the temperature of the transition to the crystalline state of the metallic glass.

With the aim of determining the value of the phase transition temperature (for a certain stage of the crystallization process – the onset temperature of the transition to the crystalline state T_K for the studied metallic glasses, we shall use the temperature dependences of their reduced electrical resistance (see Fig. 4; heating rate 20 K/s). Considering the crystallization rate V_K ($V_K = \Delta(R_T/R_{273K})/\Delta T$), the phase transition temperature will be considered at the point where the crystallization rate is maximum. The results of the studies conducted for typical materials are presented in Table 3.

The results of the conducted studies indicate that there is no clear correlation between the tendency of alloys to transition to an amorphous state and their resistance to crystallization. It seems more likely that the resistance of the studied materials to crystallization increases with the rate of their quenching.

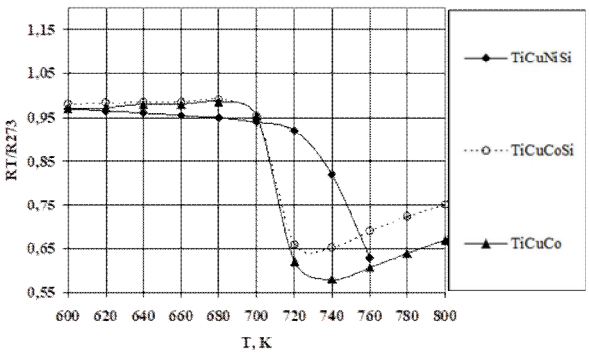

Fig. 4. Typical temperature dependencies of the reduced electrical resistance of the studied materials

Table 3. Melting, glass formation, phase transition temperatures, and crystallization rates of the studied metallic glasses

Склад стопу	T_T, K	$T_{ш}$, K	T_K, K	V_K, K^{-1}
Ti-Cu-Ni-Si	1677	883	720	–0.00297
Ti-Cu-Co-Si	1687	788	710	–0.015
Ti-Cu-Co	1674	782	700	–0.0093

To study the kinetics of crystallization of metallic amorphous alloys, the method of electrical resistance was used. For this purpose, a series of isothermal annealings of samples at various temperatures were conducted, with the dynamics of their resistance change being recorded. The process was analyzed using the modified Avrami equation:

$$\log\left[\ln\left(1-\frac{R_A-R(t)}{R_A-R_K}\right)^{-1}\right]= \\ =n\left[\log\left(\tau_0\exp\left(\frac{-E_K}{kT}\right)\right)^{-1}+\log t\right] \quad (9)$$

where R_A is the initial electrical resistance of the sample in the amorphous state, Ohm; $R(t)$ is the electrical resistance of the sample at time t, Ohm; $R_к$ is the final electrical resistance of the sample in the crystalline state, Ohm; n is a factor determined by the nature of the nucleation and growth of crystals; τ_0 is the frequency factor; $E_к$ is the activation energy of the crystallization process, eV; k is the Boltzmann constant, eV/K.

To determine the thermal stability of metallic glasses as a function of temperature and operating time, they were subjected to continuous isothermal annealing at a temperature $T_в$ which is $0.75T_ш$ for a time $t_в$, which greatly exceeds the crystallization time $t_к$, determined during the study of the crystallization dynamics.

Analyzing the results of these studies and the crystallization kinetics for metallic amorphous alloys, we found that the characteristic parameters we proposed [11], such as the temperature exposure $B = (0.75T_ш; t_в)$ which determines the onset of crystallization processes in the studied samples and the crystallization time $t_к$ are appropriate for estimating their probable operating time $t_{iк}$ (without the risk of crystallization development) depending on the operating temperature (T_e). As a result of the conducted studies, an empirical dependency of the searched values was obtained, which aligns well with the experimental data:

$$t_{iк} = A\sqrt{\frac{B\,t_к}{T_e}}, \quad (10)$$

where A is a factor whose value varies within the range of 600–800 and depends on the individual characteristics of the studied sample.

Fig. 5 shows the dependency of $t_{iк}$ on the operating temperature for the metallic glass Ti-Cu-Ni-Si.

To achieve extended analytics and an understanding of the relationships between thermoelectric properties of materials and operational factors, the following machine learning models were utilized in the research. Random Forests, an ensemble of decision trees, was used for classification and regression analysis. This model was applied to determine the influence of operational factors such as temperature, mechanical stress, and chemical composition on the thermoelectric properties. Gradient Boosting Machine was applied for optimal interpretation of the impact of external factors on the stabilization of alloy properties. Support Vector Machines (SVM) were used for distinguishing between material states when it is necessary to separate two classes of data with a large number of input parameters.

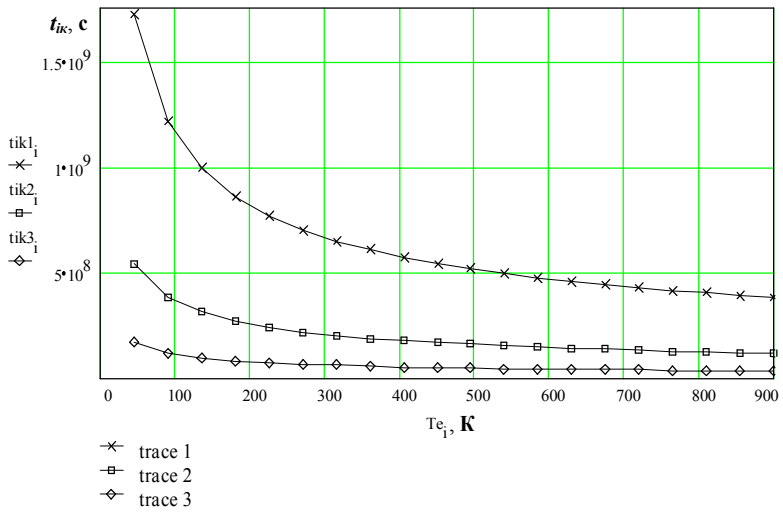

Fig. 5. Dependence of $t_{iк}$ on the operating temperature and the dynamics of the crystallization process for the metallic glasses Ti-Cu-Ni-Si (curve 1 for $t_к$ = 1000 s; curve 2 for $t_к$ = 100 s; curve 3 for $t_к$ = 10 s)

It was found that the electrical resistance and thermoelectric power of materials depend on the temperature range from 4.2 to 1200 K, and from the influence of operational factors, such as mechanical stress or changes in chemical composition. It was observed that the introduction of specific alloying elements changes the thermometric properties of materials, aiding in the stabilization of electrical resistance and thermoelectric power at high temperatures. It was also discovered that optimal cooling parameters affect the electrophysical properties of the alloys used for thermoelectric converters.

It was established that with an increase in temperature from 4.2 K to 300 K, the electrical resistance of iron-nickel alloy samples decreases by 15 %. The addition of copper in 5 % concentration is capable of stabilizing the resistance at temperatures above 500 K, where the decrease was 3 %. Modeling identified conditions for quality control; cooling to 100 K within 10 hours after thermal treatment improves the stability of electrical resistance by ±2 %.

7. Conclusions

It should be noted that due to the homogeneity of the structural state, as well as the absence of dislocations and grain boundaries in metallic glasses, their electrophysical properties are characterized by high stability and reproducibility. This makes these materials highly promising, especially for application in electrical thermometry for creating precise measurement thermotransducers in the range from cryogenic to medium temperatures.

Gratitude

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Conflict of Interest

The authors state that there are no financial orother potential conflicts regarding this work.

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