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PECULIARITIES OF KINETIC AND MECHANICAL PROPERTIES OF HIGH-ENTROPY ALLOY $Al_{0.5}CoCuCrNiFe$ IN RANGE ~ 300…77 К

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The temperature dependences of the fundamental kinetic coefficients and the creep rate of steady-state plastic flow of the high-entropy alloy $Al_{0.5}CoCuCrNiFe$ have been determined for interval ~ 300…77 K. The correlation analysis of the features of the investigated characteristics with known acoustic anomalies of $Al_{0.5}CoCuCrNiFe$ has been carried out. Availability of the microstructure changing $Al_{0.5}CoCuCrNiFe$ into studied temperature range has been shown. For the first time the observed structural transformation in vicinity of T ~ 160 K is assigned to the martensitic type.

INTRODUCTION

An analysis of the publications array on problems of high-entropy alloys (HEAs), see, e.g. [1, 2], reveals two aims of such works: i) improving supposed operation characteristics and ii) establishing physical laws for connection of structure with properties. It is striking that in ii) electro-physical and thermal methods are very little used [3–5] for recording structural-phase transformations (SPT) the presence of which in HEAs in a wide temperature range is not in doubt, due to their multiphase nature [6–8]. Meanwhile, if it is supposed practical using material, the issues of its phase stability are important to predicting operational properties [9–11].

The aim of the work is to reveal the temperature features of the fundamental kinetic (electrical resistance R, thermoelectric power *S*, thermal conductivity k) and mechanical characteristics of the $Al_{0.5}CoCuCrNiFe$ alloy in the range of ~ 300…77 K and try to find out their nature by comparing it with the known acoustic features of $Al_{0.5}CoCuCrCiNiFe$. The choice of the temperature range is due to small number of HEAs studies carried out in it and due to possibility of using the latter as a structural material operating at low temperatures. As for promising goal of such research in the scientific field, that is finding correlations between the fine features in the behavior of kinetic, mechanical and acoustic properties of HEAs and the structural-phase state (SPS) features of ones- both submicro- and nanoscale [12].

SAMPLES AND METHODS

Alloy $Al_{0.5}CoCuCrNiFe$ (components content in wt.%: Al – 4.46; Co – 19.48; Cu – 21.01; Cr – 17.18; Ni – 19.4; Fe – 18.46), density 7.98 g/cm^3, was obtained by fusion of the components of purity 99.9 wt.% in argon atmosphere. Fusing and making samples in form of plates ~ 0.2×3×50 mm described in [6, 13]. In addition to cast samples, we studied samples annealed at 975 °C for 12 h, as well as samples annealed in the same mode and subsequently deformed by tension. To distinguish of structural states the studied quantity has index j in the text, which takes one of the values “cst”, “an”, “an + def”.

The experiments were carried out in step-by-step (~ 2…5 K) mode by means of lowering the sample temperature by the use the moving it in temperature field (TF) with known topography of isothermal surfaces. As such, TP of a gaseous nitrogen column in cylindrical Dewar vessel with liquid N_2 was used.

To experimentation the segment of TF of length ~ 300 mm was chosen, which local temperature, as established in separate experiment, is constant on azimuth and radius in horizontal sections and monotonically decreases with approaching liquid surface. Horizontally oriented sample was located inside of copper container intended to eliminate of thermal fluctuations. Measurements were carried out in static positions of sample passed step-by-step in direction of liquid. Time of SPS thermalization was ~ 3…5 min. To measure temperature difference $\Delta T = T_1 – T_2$ between ends of sample were used two Cu-Const thermocouples connected to meet. Sensitivity of ΔT measurements ~ 0.01 K. Temperature of sample was taken to be $(T_1+T_2)/2$, it was measured with accuracy of ~ 1 K. Thermalized state instability was ≤ 0.01 K.

RESULTS

Electro-physical characteristics. Dependences $R_j(T)$ were determined with accuracy of no worse than ~ 1% according to data of measurements of potential difference U = I×R by 4-point method. Fig. 1, shows the graphs of the normalized dependences $R_j(T)/R_{j0}$ for cast, annealed and deformed samples ($R_{j0} = R_{j290\,K}$, $T_0 = 290$ K). At T_0 the values of specific resistances ρ are: $\rho_{cst0} \sim 86$ μΩ·cm, $\rho_{an0} \sim 77$ μΩ·cm, $\rho_{(an+df)0} \sim 79$ μΩ·cm. The average temperature coefficient of electrical resistance, TCR, in range 300...100 K is ~ $350\cdot10^{-6}$ K^{-1}.

As can be seen from Fig. 1 the dependences $R_j(T)/R_{j0}$ are metallic, demonstrating the nonmonotonous behavior of derivative $dR_j(T)/dT$: the temperatures of the features, T_n, Table 1, are indicated by arrows. For annealed samples interval of ~ 238…200 K is distinguished. In one the value of TCR changes very slightly. The smoothest of the given $R_j(T)/R_{j0}$ curves is dependence characterizing the samples deformed after annealing. It has only one feature, namely, at T ~ 155…160 K. In our opinion, this resistive anomaly can indicate the occurrence (see [14]) of the deformation polymorphic transformation. It should be noted that in similar studies [3, 5] graphs R = f(T) did not have similar features. Perhaps this is due to the fact that the measurements [3, 5] were carried out on drift T, which could "mask" the kinetics of the processes during the SPS rearrangements. Table 2 shows the average values of TCR of cast and annealed specimens for intervals $T_n…T_{n+1}$. Temperature dependencies of TCR will be analyzed below.

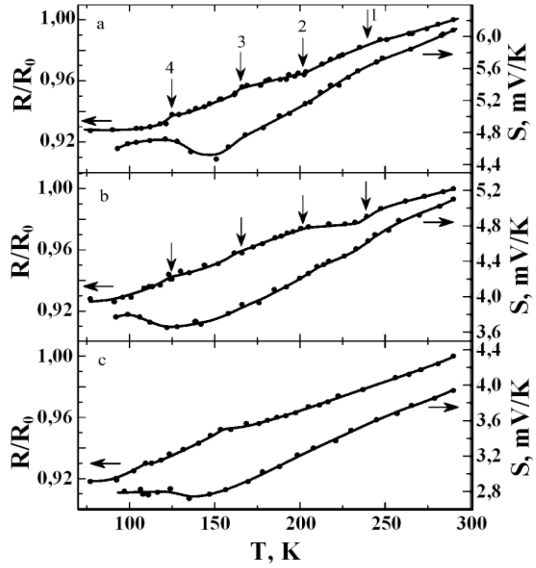

Fig. 1. Dependences $R_j(T)/R_{j0}$ and $S_j(T)/S_{j0}$: a – j=cst, b – j=an, c – j=an+df; T_0 = 290 K

Table 1

T_n of resistive anomalies

Anomaly number	1	2	3	4
T_n, K	~ 238	~ 200	~ 160	~ 125

Table 2

TCR for intervals $T_n…T_{n+1}$

Interval	$T_0…T_{0+1}$	$T_1…T_2$	$T_2…T_3$	$T_3…T_4$
ΔT, K	52	38	40	35
TCR_{cst}, 10^{-6} K^{-1}	346	474	250	371
TCR_{an}, 10^{-6} K^{-1}	404	105	450	343

To determine of absolute differential thermoelectric power S the samples were used on which resistive measurements were carried out. The potential difference $\Delta U = U_1–U_2$ between ends of sample arisen owing to difference $\Delta T = T_1−T_2$ was measured with accuracy of ~ 10^{-7} V. To making ΔT the electric heater was placed on one of sample ends. Accuracy of determining absolute value of $S = \Delta U/\Delta T$ we estimate as ~ 5…10%.

As follows from Fig. 1 i) $S < 0$ for all studied structures, *ii*) absolute values of $S_{j290\,K}$ decrease from ~ 6 to ~ 4 mV/K as result of thermo/thermomechanical treatments. This fact indicates an increase of metallic nature of alloy. Value of S decreases approximately linearly to T ~ 160 K after that there is the feature in course of S(T) consisting of change of temperature coefficient sign, dS/dT. In addition to this pronounced anomaly there are weaker features of $S_j(T)/S_{j0}$ curves. Their temperatures are coincided with T_n of resistive features designating on community of their nature. It is worth noting that the features of the dependences $S_j(T)$, as well as the upper resistive ones were observed for the first time.

Thermal conductivity. In these investigations the method of express recording thermal conductivity (k) anomalies was applied. Effectiveness of its was demonstrated by investigations of high temperature superconductors pseudogap state [15, 16]. The method advantage is the absence of need to comply with the classical method of stationary uniaxial flow [17] on maintaining ideal sample thermal insulation and stationarity of supplying heat flux Q. These conditions observing, as known [18], makes experiment extremely difficult.

As in [17], the temperature difference $\Delta T = T_1−T_2$ between the ends of the sample that established by supplying heat flux Q to one of them was used as indicator of k-value. If heat losses take a place it can write $\Delta T(T) \propto \mu Q/k(T) \times \Psi(T)$, where $\Psi > 1$ is coefficient proportionate to losses. Then ratio $k(T)/k_0$ for T and T_0 will be as follows:

$$k(T)/k_0 = [\Delta T_0/\Delta T(T)] \times \Psi_0/\Psi(T). \qquad (1)$$

The ratio of the experimental values is

$$\Delta T_0/\Delta T(T) = [k(T) \times \Psi(T)]/k_0 \times \Psi_0. \qquad (2)$$

Denoting $k(T) \times \Psi(T) \equiv \mathbb{K}(T,\Psi)$ and $k_0 \times \Psi_0 \equiv \mathbb{K}_0(T,\Psi)$ we obtain

$$\Delta T_0/\Delta T(T)] = \mathbb{K}(T,\Psi)/\mathbb{K}_0(T,\Psi). \qquad (3)$$

Coefficient $\mathbb{K}(T,\Psi)$ has the sense of normalized coefficient of thermal conductivity k. It is proportionate to k and to normalization coefficient Ψ(T). From (3) follows if dependence Ψ(T) is monotonic the anomalies of dependence $\Delta T_0/\Delta T(T) = \mathbb{K}(T,\Psi)/\mathbb{K}_0(T,\Psi)$ should be considered due to anomalies of dependence k(T).

The dependences $[\Delta T_0/\Delta T(T)] = \mathbb{K}(T,\Psi)/\mathbb{K}_0(T,\Psi)$ for alloy $Al_{0.5}CoCuCrNiFe$ were measured for samples of previous sections. Ψ(T)-monotony was achieved by applying regime of monotonically lowering temperature of sample by moving letter in TF of Dewar with liquid N_2, see above.

Fig. 2 shows the graphs of dependences $\Delta T_0/\Delta T(T)$ (j = cst, an; T_0 = 290 K). Their configurations indicate that thermal conductivity k of cast and annealed states decrease with decreasing T in range of ~ 260…160 K. The change of inclination of graphs of dependences $\Delta T_0/\Delta T(T)$ below ≈ 160 K indicates the change of thermal-physics characteristics as result of a structural transformation. As in graphs of dependences $R_j(T)/R_{j0}$ and $S_j(T)/S_{j0}$, the curves $\Delta T_0/\Delta T(T)$ have weaker features. Their temperatures coincide with T_n of dependences $R_j(T)/R_{j0}$ and $S_j(T)/S_{j0}$ indicating both reality.

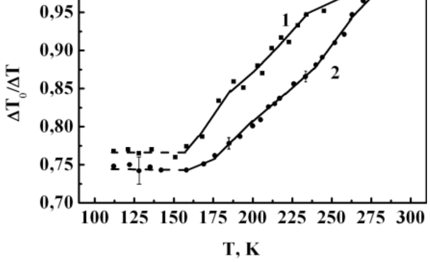

Fig. 2. Dependences $\Delta T_0/\Delta T$: 1 – j=cst, 2 – j=an; T_0=290 K (errors of measuring shown over curve 2)

Temperature dependence of creep rate. If a phase transformation in a solid be accompanied by change of energy spectrum some changes of mechanical properties are observed. It seemed probable the anomalies of thermoelectric power and thermal conductivity of $Al_{0.5}$CoCuCrNiFe occasioned in vicinity of T ~ 160 K by possible phase transformation can manifest themselves in mechanical properties, in particular, in kind of anomaly of creep rate (CR). To verify this assumption the cast and annealed samples were tested on creep.

Experiments were carried out under uniaxial tension and fixed applied stress. At the beginning experiment the sample (length of working part l ~ 20 mm) was immersed into liquid nitrogen. The value of applied stress was $\sigma = 0.9\sigma_B$ (σ_B is the ultimate strength of this material). Temperature of sample has been taken with accuracy of ~ 0.5 K using Cu-Const thermocouple was placed in middle of working part. After reaching the stage of steady-state plastic flow and strange rate $\dot{\varepsilon}$ ~ 10^{-6} s^{-1} the temperature was being increased at rate of 0.5 K/min in spite of all this we fixed elongation of sample with accuracy of $5\ 10^{-5}$ cm.

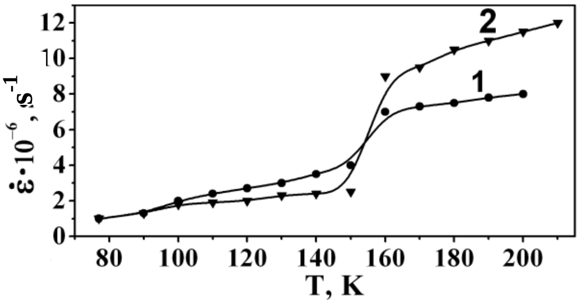

Fig. 3. Creep rate of HEA $Al_{0.5}$CoCrCuFeNi vs T: 1 – casted samples, 2 – annealed samples

As follows from Fig. 3 increasing temperature in the range 77…140 K calls into being a smooth increase of CR. But on the interval ~ 140…160 K we observed another picture, namely, jump-like change of $\dot{\varepsilon}$ for both type of samples. Above T ~ 160 K the derivative $d\dot{\varepsilon}/dT$ falls approximately on order. Since the dependences R(T), S(T), $\Delta T_0/\Delta T(T)$ also have the features at T ≈ 150…160 K the most probable cause of the sharp change in CR is the loss of crystal lattice stability during phase transformation. This assumption do not contradicts to the fact that on CR "jump" the relative growth $\Delta\varepsilon/\varepsilon_0$ for annealed samples is ≈ 2.5 times greater than one for cast: $(\Delta\varepsilon/\varepsilon_0)_{cst} \approx 1.75$ and $(\Delta\varepsilon/\varepsilon_0)_{an} \approx 4.5$. That is apparently due to different nature of changes in spectrum of dislocation barriers during phase transformation in cast and annealed states.

DISCUSSION

In accordance with existing ideas, during crystallization and subsequent cooling of HEA-ingot inside formed dendrites and interdendritic spaces the highly nonequilibrium SPS decomposes into several phases of different composition and morphology. These phases differ on a nanoscale in multicomponent chemical composition and have pronounced three-dimensional volume modulations [8, 19, 20]. The reality, however, is that when studying already formed SPS it is impossible to establish sequence and type of SFTs. The fact that they take a place in solid HEAs is indicated, for example, by series of pronounced magnetic transformations in the equiatomic alloy AlCoCrCuFeNi at high temperatures (~ 600…700 K) and weaker magnetic anomalies at low temperatures (~ 180 and ~ 100 K) [8]. Therefore, the following questions are of scientific and practical interest. What is the minimum SPT-temperature of a solid HEA? What is the nature and kinetics of SPTs? Are there hysteresis phenomena during thermal cycling? And so on.

Generally accepted [21] that the features absence in temperature behaviors of electron, magnetic and phonon properties guarantees the absence of temperature phase transformations (TPTs). If this is true, the curves configurations of Figs. 1–3 indicate some rearrangements of studied structural states in range ~ 250…125 K. In addition the weak anomalies there are also cardinal ones: the CR is changed about twice; the sign of temperature coefficient of thermoelectric power is changed and, as determined in [6, 13, 22], the anomalies of acoustic properties of $Al_{0.5}$CoCrCuFeNi take a place. In view of the last circumstance it seemed advisable to use acoustic data [6, 13, 22] as independent arguments of our analysis, especially since the samples in cited works were made using same technology as our ones.

The initial data for the correlation analysis are presented in Fig. 4. Curves 1 characterize the temperature dependences of the average values of TCR_{an} and TCR_{cst} for intervals $T_n…T_{n+1}$ (see Table 2). Let's compare them with the curves of bending vibrations with ω = 530 Hz measured by the method of mechanical resonance spectroscopy in the amplitude-independent strain region [6, 13]. The experimental attenuation curves $\delta_{anexp}(T)$ and $\delta_{cstexp}(T)$ [13] (in our notations) are shown in the inset of Fig. 4,a, and their smoothed versions are shown by curves 2 in Fig. 4,a,b. In Fig. 4,b shows the temperature dependence of attenuation of sound of 50 MHz [22].

As for curves 3 depicted by dashed lines, that of them which concerns to annealed structures (i.e. on Fig. 4,a) was obtained in [13]. It represents the temperature (background) term $\delta_{an,t}(T)$ of the experimental value $\delta_{an,f}(T)$ amounting to:

$$\delta_{anexp}(T) = \delta_{anT}(T)+\delta_{anres}(\omega,T), \qquad (4)$$

where $\delta_{anres}(\omega,T)$ is the resonance term [23]. The area shaded represents the region of the sound resonance absorption [13].

By analogy with [13] we have identified the temperature component $\delta_{cst,T}(T)$ of curve $\delta_{cstexp}(T)$ for the cast samples. It is shown by dashed line 3 in Fig. 4,b. Thus, we revealed the temperature range of the sound resonance absorption, but now it is the range ~ 200…160 K.

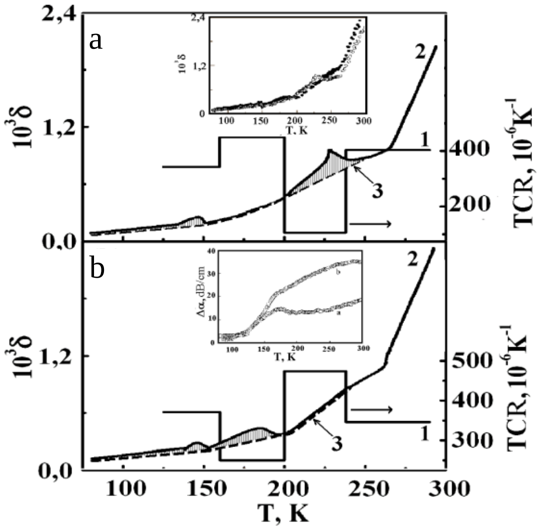

Fig. 4. The dependences: TCR = f(T) – (1); δ_{exp}(T), [13] – (2); δ_T(T) – (3), [13]. Areas shaded represent the regions of resistance absorption: a – annealed state; b – casted state. Insert on Fig. 4,a: the dependences δ_{cst}(T) – black circles and δ_{an}(T) – white circles [13]. Insert on Fig. 4,b: attenuation of ultrasound of 50 MHz parallel (a) and perpendicular (b) to dendrites axis in annealed $Al_{0.5}$CoCuCrNiFe [22]

It is clearly seen TCR_j and δ_{jexp} are changing in "antiphase" to each other on intervals of ~ 238…200 and ~ 200…160 K: decreasing TCR_j coincides with appearance of resonance component, and increasing TCR_j coincides with its disappearance. Such inversion correlation of TCR_j with δ_{jres} can be explained by assumption about of the presence of some TPTs happened on these intervals – one by one for each kind of microstructure. For cast state that is interval ~ 200…160 K, where TCR_{cst} has minimum and the resonance component $\delta_{cstres}(\omega,T)$ take a place. For annealed structure that interval is ~ 238…200 K corresponding to minimum of TCR_{an} and to the presence of the resonance component δ_{anres} with Koiva-Hasiguchi peak at 228 K.

As regards the nature of the structure changes over these enough extent extensive intervals, it predestined probably by heterogeneity of nanoscale distribution of elements entering in alloy composition. It was marked [21], in microstructure of $Al_{0.5}$CoCuCrNiFe on the one hand, the regions are available [21] in form of strips of ~ 15…20 nm wide having essentially distinction of elements concentrations. Such stripes are formed an un-regular three-dimensional lattice which knots may be effective barriers for different quasiparticles. Other type of structure heterogeneity observed also in [21] are clusters of atoms having relatively big atomic radius. Ones also create local distortions of crystalline lattice and represants substantial obstacles for dislocations. It is impossible to exclude the configurations of both types of obstacles are changing smoothly with temperature that have been fixed by resistive and acoustic date. The reason of why intervals of existence of the resistive and acoustic anomalies are spaced in T in cast and annealed specimens is apparently the difference of initial structure states before sample cooling from 300 K. This point of view supported by the fact that as result of annealing $Al_{0.5}$CoCuCrNiFe at 975 °C for 12 h the contents of Cu and Al has been decreased into interdendritic spaces, quantity of other components has been increased and microvolumes have been formed enriched by Ni, Al, and Cu near dendrites boundaries [5]. These data confirm noticeably different of the microstructures of the cast and annealed alloy $Al_{0.5}$CoCuCrNiFe at 300 K and not surprising their posterior temperature evolutions are different.

Some words for cardinal anomalies of S, k, and CR below ~ 160 K. Their reality are confirming also by observing anomalies of attenuation of ultrasound of 50 MHz (see insert on Fig. 4,b). Is the assumption was made concerning these anomalies (about the phase transformation) non-alternative? In this connection it is worth to consider as possible objection the results of [24, 25], were shown that when the temperature of alloy $Al_{0.5}$CoCuCrNiFe is decreased from 300 to 77 K the process of macrotwinning is changing by the process of microtwinning. That leads to increasing density of twin boundaries being the obstacles for dislocation movement. That decreases CR at cooling. However a change of the kind of the twinning occurs gradually as rule – due to decrease of contribution to plastic deformation of one kind of twinning and due to gradual increase of another one and do not accompanied by jump of CR. Therefore sharp change of CR cannot be associated with gradual change of dislocation barriers but indicates qualitative changes of material structure. Most probably in our case it is martensitical transformation (see also [14]).

CONCLUSIONS

The temperature dependences: of the electrical resistance R and the thermoelectric power S, of the sign of temperature coefficient of thermal conductivity k and the creep rate under uniaxial tension have been determined for samples of high-entropy alloy $Al_{0.5}$CoCuCrNiFe in temperature range ~ 300…77 K.

We have ascertained that values of $\rho_{300\,K}$ for cast, annealed and deformed samples are ~ 86, ~ 77, ~ 79 μΩ·cm, respectively. Average temperature coefficient of resistance in the range 300…100 K is ~ $350 \cdot 10^{-6}$ K^{-1}. It has been shown the annealing of the samples leads to decreasing $S_{300\,K}$ from ~ (–6 mV/K) to ~ (–4 mV/K).

It was shown that discovered firstly in range ~ 250…130 K the correlating features of the studied characteristics are the consequences of microstructure changes. Most radical one corresponds to structural-phase transformation, apparently, of martensitic type.

By correlation results analysis with known acoustic spectra of similar samples it has been shown that anomalous lowering electrical resistance with lowering temperature correlates with appearance of resonance term in acoustic spectra. Both kinds of anomalies have been provoked presumably by nanoscale changes in structure.

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ОСОБЕННОСТИ КИНЕТИЧЕСКИХ И МЕХАНИЧЕСКИХ СВОЙСТВ ВЫСОКОЭНТРОПИЙНОГО СПЛАВА $Al_{0,5}$CoCuCrNiFe В ИНТЕРВАЛЕ ~ 300…77 K

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Определены температурные зависимости фундаментальных кинетических коэффициентов и скорости ползучести установившегося пластического течения высокоэнтропийного сплава $Al_{0,5}CoCuCrNiFe$ в интервале ~ 300…77 K. Проведен корреляционный анализ особенностей исследовавшихся характеристик с известными акустическими аномалиями. Показано наличие изменений микроструктуры $Al_{0,5}$CoCuCrNiFe в изученном интервале температур. Впервые наблюденное структурное превращение вблизи T ~ 160 K отнесено к мартенситному типу.

ОСОБЛИВОСТІ КІНЕТИЧНИХ І МЕХАНІЧНИХ ВЛАСТИВОСТЕЙ ВИСОКОЕНТРОПІЙНОГО СПЛАВУ $Al_{0,5}$CoCuCrNiFe В ІНТЕРВАЛІ ~ 300…77 К

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Визначено температурні залежності фундаментальних кінетичних коефіцієнтів і швидкості повзучості усталеної пластичної течії високоентропійного сплаву $Al_{0,5}$CoCuCrNiFe в інтервалі ~ 300…77 К. Проведено кореляційний аналіз особливостей вивчених характеристик зі знаними акустичними аномаліями $Al_{0,5}$CoCuCrNiFe. Продемонстрована наявність змін мікроструктури $Al_{0,5}$CoCuCrNiFe у дослідженому інтервалі температур. Вперше спостережене структурне перетворення поблизу T ~ 160 K віднесено до мартенситного типу.