ФИЗИКА ПРОЧНОСТИ И ПЛАСТИЧНОСТИ

Mössbauer and Ultrasonic Study of the As-Cast High-Entropy Al$_x$CuCrCoNiFe Alloys

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Determination of the hyperfine-interaction parameters, acoustic and elastic properties of the high-entropy Al$_x$CuCrCoNiFe alloys (HEAs) ($x = 1$, 1.8) in the as-cast state is carried out with the use of Mössbauer spectroscopy, x-ray diffractometry and high-precision ultrasonic techniques. Wide distributions of both the hyperfine-magnetic fields on iron nuclei (5.0–36.3 T) and the isomer shifts (–0.15–+0.28 mm/s) are detected in the HEAs that is result of multiphase state and inhomogeneous short-range order in the phases. As shown, the as-cast Al$_x$CuCrCoNiFe HEAs are acoustically and elastically inhomogeneous systems. Mean elastic moduli of the as-cast equimolar HEAs ($\langle E \rangle = 172.8$ GPa, $\langle G \rangle = 65.3$ GPa, $\langle B \rangle = 161.8$ GPa) and the Debye temperature ($\langle \Theta_D \rangle = 436$ K) exceed ones for the elemental metals composing HEAs. There are the Poisson's ratio $\langle \eta \rangle = 0.312$–0.322 and the ratio $\langle B \rangle / \langle G \rangle = 2.321$–2.477. Deviation from equimolar content of Al ($x = 1.8$) results in redistribution of the spin and charge s-electron densities near iron nuclei, increases the elastic moduli and the Debye temperature by approximately 4–5%.

Keywords: high-entropy alloys, ultrasonics, elastic properties, elastic anisotropy, Mössbauer effect.

Досліджено параметри надтонкої взаємодії, акустичні та пружні властивості високоентропійних стопів (ВЕС) Al$_x$CuCrCoNiFe ($x = 1$, 1,8) у литому стані за допомогою мессбаверівської спектроскопії, рентґенівської дифрактометрії та надточної ультразвукової спектроскопії. Виявлено широкі

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розподіли надтонких магнетних полів на ядрах Феруму (5,0−36,3 Тл) й ізомерних зсувів (−0,15−+0,28 мм/с) у ВЕС, які є результатом багатофазности стану та неоднорідности близького атомового порядку у фазах. Показано, що литі ВЕС Al$_x$CuCrCoNiFe являють собою акустично та пружньо неоднорідні системи. Усереднені модулі пружности литого еквіатомового ВЕС ($\langle E \rangle = 172,8$ ГПа, $\langle G \rangle = 65,3$ ГПа, $\langle B \rangle = 161,8$ ГПа) та Дебайова температура ($\langle \Theta_D \rangle = 436$ К) перевищують аналогічні значення для чистих металів, що входять до складу ВЕС. Пуассонів коефіцієнт $\langle \eta \rangle = 0,312-0,322$, а відношення $\langle B \rangle / \langle G \rangle = 2,321-2,477$. Відхил від еквіатомового складу за Al ($x = 1,8$) приводить до перерозподілу s-електронних спінової та зарядової густин на ядрах атомів Феруму, збільшує модулі пружности та Дебайову температуру приблизно на 4−5%.

Ключові слова: високоентропійний стоп, ультразвук, пружні властивості, анізотропія пружности, Мессбаверів ефект.

Исследованы параметры сверхтонкого взаимодействия, акустические и упругие свойства высокоэнтропийных сплавов (ВЭС) Al$_x$CuCrCoNiFe ($x = 1, 1,8$) в литом состоянии с помощью мёссбауэровской спектроскопии, рентгеновской дифрактометрии и сверхточной ультразвуковой спектроскопии. Обнаружены широкие распределения сверхтонких магнитных полей на ядрах железа (5,0−36,3 Тл) и изомерных сдвигов (−0.15−+0.28 мм/с) в ВЭС, которые являются результатом многофазного состояния и неоднородности ближнего атомного порядка в фазах. Показано, что литые ВЭС Al$_x$CuCrCoNiFe представляют собой акустически и упруго неоднородные системы. Усредненные модули упругости литого эквиатомного ВЭС ($\langle E \rangle = 172,8$ ГПа, $\langle G \rangle = 65,3$ ГПа, $\langle B \rangle = 161,8$ ГПа) и температура Дебая ($\langle \Theta_D \rangle = 436$ К) превышают аналогичные значения для чистых металлов, входящих в состав ВЭС. Коэффициент Пуассона $\langle \eta \rangle = 0,312-0,322$, а отношение $\langle B \rangle / \langle G \rangle = 2,321-2,477$. Отклонение от эквиатомного состава по Al ($x = 1,8$) приводит к перераспределению s-электронных спиновой и зарядовой плотностей на ядрах атомов железа, увеличивает модули упругости и температуру Дебая примерно на 4−5%.

Ключевые слова: высокоэнтропийный сплав, ультразвук, упругие свойства, анизотропия упругости, эффект Мёссбауэра.

1. INTRODUCTION

High-entropy alloys (HEAs) belong to multicomponent systems containing principal elements in the concentration range between 5 and 35 at.% [1, 2]. A characteristic feature of these alloys is that they do not have the element as host and are characterized by large mixing entropy, that can be considered as the measure of chaos in atomic distribution [1, 2]. A number of experiments have shown that majority of HEAs in as-cast state contain two or more phases with the simple crystal lattices such as f.c.c., b.c.c., h.c.p. ones as well as ordered or

amorphous microareas [2−7].

HEAs of Al$_x$CoCrCuFeNi system have attracted much attention due to their unusual structure and properties. For instance, they possess high heat resistance, corrosion resistance [8], low electrical conductivity and thermal conductivity [9, 10], low growth of grains [11], and high thermal stability [2, 12, 13]. These HEAs show high hardness 208−420 HV [3, 14], tensile mechanical properties [2, 12, 14−16], abrasive wear [14, 17], superior high-temperature strength sustained up to 800°C, enhanced plasticity, and large work-hardening capability [14]. Thus, the Al$_x$CrCuFeNiCo HEAs have a high potential for the application as the engineering materials in tools and in certain high-temperature applications.

Important fundamental characteristics of any solid are elastic moduli and the Debye temperature. Particularly, elastic moduli are needed for the determination of stress for the dislocations' movement, the fracture toughness, the work hardening rate as well as for the estimation of the interatomic-bond stiffness. At the same time, the precise information about elastic properties of HEAs is limited. There are data mainly upon the tensile Young's modulus [2, 5] or the elastic modulus E obtained by means of indentation [6, 7, 12, 18]. However, in these experiments probable chemical inhomogeneities in HEAs and their elastic anisotropy cannot be revealed. Only the elastic moduli of equimolar CrMnCoFeNi HEA were determined by more precise resonant ultrasound spectroscopy [19]. However, it was Al-free homogeneous single-phase (100% f.c.c. phase) alloy. Authors of Ref. [20] used dynamic-mechanical analyser for the measurement of the Young's modulus of the Al$_x$CoCrFeNi ($x = 0−1$) alloys in the frequency range of 1−16 Hz. In doing so, the applied technique is an effective one just to study internal friction and damping capacity of a homogeneous material. However, this method as in the case of other resonance techniques is an integral one and is not suitable for the determination of elastic characteristics in separate zones of inhomogeneous systems.

Mössbauer spectroscopy can give important information about inhomogeneous short-range atomic order as well as magnetic order, distribution of spin and charge s-electron density at nuclei of resonance atoms and, as a result, can add to the data of another measurement. The Al$_x$CrCuFeNiCo HEAs in as-cast state were not studied by means of Mössbauer spectroscopy, although, in Ref. [21], it was shown by this technique that two-phase (f.c.c. and b.c.c.) Al$_{1.5}$CoCrCuFeNi HEA is characterized by wide distribution of the hyperfine magnetic fields and inhomogeneous magnetic order. Work [22] shows the first example of Mössbauer spectroscopy study of the Al$_x$CrCuFeNiCo HEAs coatings.

Thus, there are insufficient experimental data on hyperfine interaction parameters, precise quantitative acoustic and elastic properties of

the AlCuCrCoNiFe HEAs, their anisotropy and spatial inhomogeneity. That was the reason why Mössbauer and X-ray diffraction data, the acoustic and elastic properties of the Al_xCuCrCoNiFe HEAs in the as-cast state were in the focus of the present investigation.

2. MATERIAL AND METHODS

The 35 g ingots of the Al_xFeCoNiCuCr HEAs ($x = 1$, 1.8) were prepared by an Ar arc melting of mixture of the elemental metals Al, Fe, Co, Ni, Cu, Cr with the purities of better than 99.9% wt. The ingots were remelted four times in order to reduce possible inhomogeneities of casting. The chemical composition of the alloys was determined by x-ray fluorescence analysis (Table 1).

The rectangular specimens were machined for both the x-ray diffraction analysis (plates of 10−15 mm in length) and the ultrasonic measurements (with $7.1 \times 5.2 \times 4.8$ mm^3 dimensions). Planes of a specimen for the ultrasonic experiment were mechanically polished in order to attain their alignment. Deviation from the specified distances between parallel planes did not exceed ± 2 μm. Before measurements, the obtained samples scanned by narrow ultrasonic beam (diameter of order 1 mm) and the most acoustically homogeneous areas were selected. For the Mössbauer measurements, foils of 20−30 μm in thickness were fabricated by mechanical polishing of thin plates and their following etching in acid.

The x-ray diffractometer 'DRON-3M' with the CoK_α radiation and Mössbauer spectrometer MS1101E with the ^{57}Co isotope of 5 mCi have been involved to characterize phase composition of HEAs and hyperfine interaction parameters. The isomer shifts were estimated with respect to α-Fe foil. The accuracy of the velocity measurements was 0.016 mm/s in the velocity range of ± 8 mm/s. The Mössbauer spectra were treated by the Window's method.

The ultrasonic measurements were executed on frequencies of 10−30 MHz at $T = 20 \pm 1°$C by automated device [23, 24]. Ultrasonic velocity was measured by the time interval between trips through a

TABLE 1. Chemical composition of the Al_xFeCoNiCuCr HEAs.

Specimen	x	wt.% (at.%)					
		Al	Fe	Co	Ni	Cu	Cr
AlFeCoNiCuCr	1.0	8.8 (17.2)	18.1 (17.1)	18.3 (16.3)	18.9 (16.9)	19.7 (16.3)	16.0 (16.2)
$Al_{1.8}$FeCoNiCuCr	1.8	14.9 (27.3)	16.8 (14.9)	17.1 (14.4)	17.7 (14.8)	18.7 (14.6)	14.7 (14.0)

zero value of identical periods (not distorted through inhomogeneity of a specimen) of high-frequency filling of the specified pair of the reflected ultrasonic radio pulses. In a number of cases, the shadow method was also used for the measurements of ultrasonic velocity.

In connection with the essential acoustic and elastic inhomogeneity of the studied HEAs, the longitudinal (v_l) and transverse (v_t) ultrasonic velocities were measured in three orthogonal directions and at least on three zones. In so doing, the extreme v_t values for each direction were determined at a rotation of polarization vector by 360 degrees. After that, all the volume velocities were averaged. The absolute instrumental error of measurement of v_{ij} was 10^{-4} on a time base of 10 μs, and the relative one was less in magnitude by one order. The real measurement error of the ultrasonic velocities in the studied objects can be estimated by spread of the measured values. The density (ρ) of specimens was measured by means of the differential Archimedes' method using quartz etalon. The error of measurements was 10^{-4} at the specimen mass of 10 g. Procedures for the measurements and analysis of experimental ultrasonic data were carried out by using the appropriate software. The calculations of elastic parameters and the Debye temperature were carried out using expressions for the isotropic polycrystals:

$$G = \rho\langle v_t \rangle^2, \tag{1}$$

$$E = GA, \tag{2}$$

$$B = GA/3(3 - A), \tag{3}$$

$$\eta = (a^2 - 2)/2(a^2 - 1), \tag{4}$$

$$A = (3a^2 - 4)/(a^2 - 1), \tag{5}$$

$$\Theta_D = \frac{h}{k}\left(\frac{9N\rho}{4\pi A_\Sigma}\right)^{1/3}\left(\frac{1}{v_l^3} + \frac{1}{v_t^3}\right)^{-1/3}, \tag{6}$$

where $a = v_l/v_t$, v_l and v_t are the averaged bulk ultrasonic velocities, h is the Planck's constant, k is the Boltzmann's constant, N is the Avogadro number, A_Σ is the total atomic weight. It could be noted that the ratio B/G as well as the Poisson's ratio η characterize plasticity of metals and alloys [25].

3. RESULTS AND DISCUSSION

3.1. X-Ray Diffraction Analysis

XRD analysis has revealed in the as-cast equimolar AlFeCoNiCuCr HEA one b.c.c. α-phase and two f.c.c. phases γ_1 and γ_2 (Fig. 1, a).

Treatment of the XRD data was carried out by means of program PowderCell 2.4. The lattice parameter of the b.c.c. phase is $a_\alpha = 0.2868$ nm and of the f.c.c. phases are $a_{\gamma1} = 0.3625$ nm and $a_{\gamma2} = 0.3592$ nm that is consistent with the data in [5]. The ratio of the integral intensities of the diffraction lines of the γ_1, γ_2 and α-phases is $I_{\gamma1}:I_{\gamma2}:I_\alpha = 7:42:51$. The presence of α-phase as well as of two γ_1- and γ_2-phases in the as-cast equimolar HEA indicates its chemical inhomogeneity.

In case of increase in Al content, the α- and γ_1-phases were retained in the $Al_{1.8}FeCoNiCuCr$ alloy, while γ_2-phase disappeared. On the whole, the content of phase with the f.c.c. structure decreases and the content of α-phase a little bit increases. The lattice parameter of α-phase and γ_1-phase increases to 0.2882 nm and 0.3648 nm, respectively, that is consistent with their concentration dependences [2, 4] and means dissolution of Al in both the b.c.c. and f.c.c. phases.

It is necessary to point out the fact that ratio of the x-ray diffraction lines intensities does not correspond to standard one that caused by a texture in the as-cast HEAs.

3.2. Mössbauer Data

Mössbauer spectra of the as-cast $Al_xFeCoNiCuCr$ ($x = 1$, 1.8) HEAs are superposition of sextet with the broadened and diffused lines and singlet (Fig. 2). Distributions of hyperfine magnetic fields $p(H)$ and isomer shifts $p(\delta)$ reduced from the spectra using the Window's approach are presented in Fig. 2, b, c, e, f. Wholly satisfactory approximation of the spectra was obtained at relatively low $\chi^2 = 1.32$ and 1.40, respectively, for the HEAs with $x = 1$ and $x = 1.8$.

The shape of the $p(H)$ function indicates a set of more probable values ($p(H) > 0.001$) lying in wide interval 5.0–36.3 T in equimolar HEA
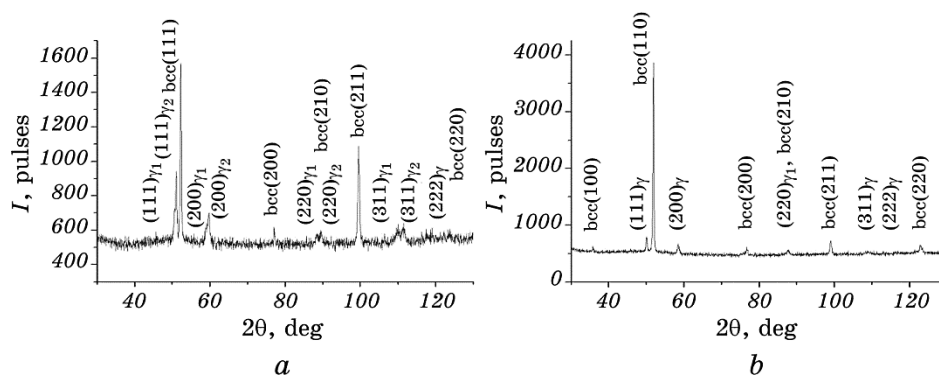
Fig. 1. X-ray powder diffraction patterns of the as-cast AlFeCoNiCuCr (*a*) and $Al_{1.8}FeCoNiCuCr$ (*b*) HEAs.

($x = 1$). Magnetic fields within the interval are most likely associated with Fe atoms in different atomic surroundings in f.c.c. phases (γ_1 and/or γ_2) detected in x-ray diffraction experiment (Fig. 1, *a*). The most probable neighbourhoods are with the Fe, Ni, Co atoms that are similar to those in Invar Fe–Ni–Co alloys [26]. Magnetic fields around 33 T can be attributed also to Fe atoms in b.c.c. phase having different kinds of atoms as nearest neighbours increasing (Ni, Co) and decreasing (Al, Cr) field on iron nuclei.

A broadened singlet in the spectrum (Fig. 2, *a*) and an intensive peak nearby zero field (Fig. 2, *b*) are caused by Fe atoms in f.c.c. paramagnetic phase with Fe, Ni, Co atoms in the first coordination shell stabi-
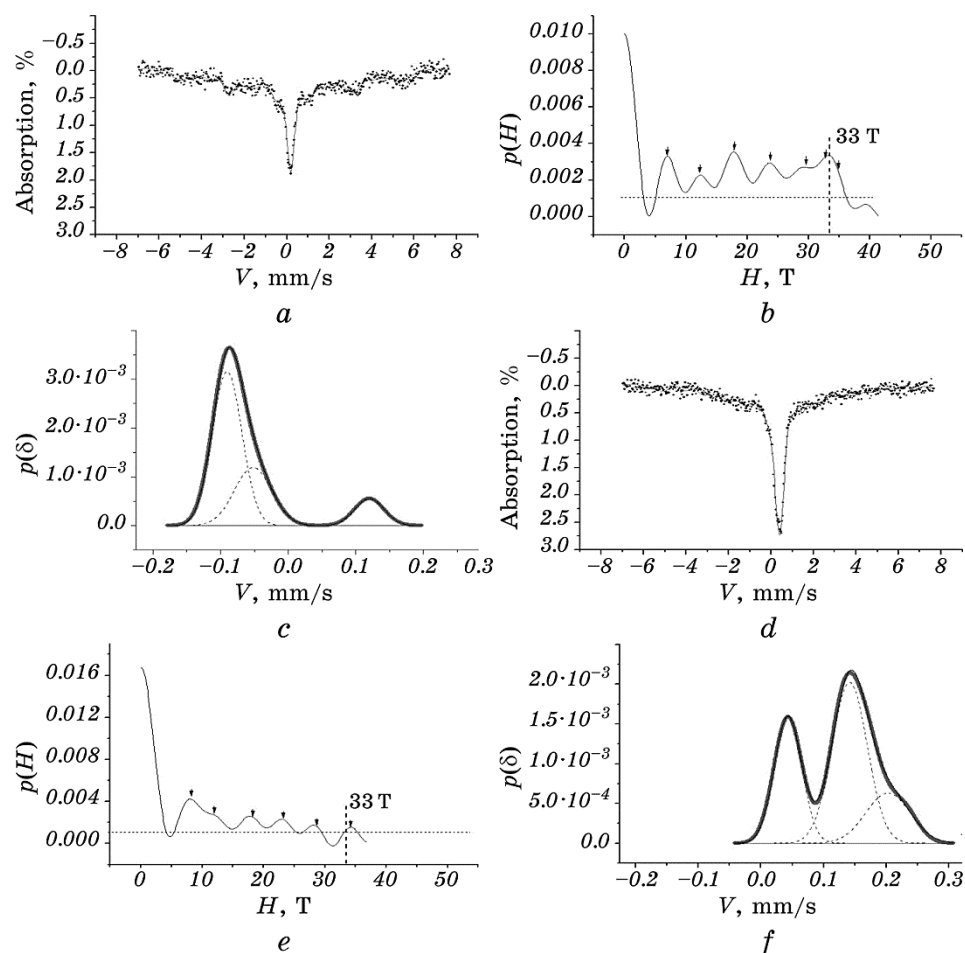

Fig. 2. *X*-ray Mössbauer spectra of the as-cast Al$_x$FeCoNiCuCr HEAs: $x = 1$ (*a*), $x = 1.8$ (*d*); distribution of hyperfine magnetic fields $p(H)$ (*b*, *e*) and isomer shifts $p(\delta)$ (*c*, *f*).

lizing an f.c.c. structure. It is possible that zero field peak is attributed to Fe atoms in copper precipitates, which according to TEM data are distributed inhomogeneously mainly on DR-ID boundaries [27, 28]. However, this contribution could be low through low mutual dissolution of iron and copper.

The increase of Al content ($x = 1.8$) above equimolar one narrows interval of the H values to 5.3–35.1 T (Fig. 2, *b*, *d*). Moreover, addition of Al in HEA sufficiently changes the $p(\delta)$ function (Fig. 2, *c*, *f*). Particularly the isomer shifts in this case markedly increased. This impact of Al results from decreasing in spin and charge *s*-electron densities at iron nuclei caused by change of interatomic distances (a_α and a_γ increased) as well as short-range atomic order.

Besides, the $p(\delta)$ function can be markedly divided on three most probable ranges both in case of equimolar HEA and of alloy with higher Al content (Fig. 2, *c*, *f*). It means there are at least three kinds of Fe atoms in different nearest surroundings. An important point is that with the increasing Al content the probabilities $p(\delta)$ are redistributed between low and high values of δ that can be caused by changing in a short-range atomic order. This is reflected also on *s*-electron spin density at iron nuclei and respectively on distribution of hyperfine magnetic fields (Fig. 2, *b*, *d*).

Thus, wide range of hyperfine magnetic fields on iron nuclei and of isomer shifts reflects an existence in the as-cast AlFeCoNiCuCr and Al$_x$FeCoNiCuCr HEAs at least three kinds of most probable nearest neighbourhoods of iron atoms which can be apparently well connected with the atomic configurations in the revealed γ_1- , γ_2-, α-phases. Deviation from equimolar content of HEA results in redistribution of spin and charge *s*-electron densities at iron nuclei caused by change of phase content, redistribution of atoms in crystal lattices of phases and between phases.

3.3. Ultrasonic Data

Since hyperfine parameters are sensitive to a change of Al concentration one can expect that interatomic bond stiffness will respond to this changing. A measure of the interatomic bond in a solid is the Debye temperature and elastic moduli. To estimate these values, the ultrasonic technique described above was applied. Taking into account that the HEAs are multiphase systems and contain chemical inhomogeneities, the ultrasonic velocities and elastic moduli were averaged using methodology described above.

Table 2 represents the obtained values of the mass density (ρ), the averaged ultrasonic velocities ($\langle v_l \rangle$, $\langle v_t \rangle$) and elastic moduli ($\langle E \rangle$, $\langle G \rangle$, $\langle B \rangle$), the ratio $\langle B \rangle / \langle G \rangle$ and the Poisson's ratio ($\langle \eta \rangle$), the Debye temperature (Θ_D) of the as-cast AlFeCoNiCuCr and Al$_{1.8}$FeCoNiCuCr HEAs.

The scatter of data on ultrasonic velocities v_l ($\cong 0.4-1.5\%$) and v_t ($\cong 30-46\%$). Probable causes of this spread are existence of several phases and spatial chemical inhomogeneity in studied alloys as well as anisotropy due-to texture. Distortion of the x-ray diffraction intensities ratio indicates an existence of texture in the as-cast HEAs (Fig. 1). In this connection, a special attention was paid to the extreme values of the ultrasonic velocities using procedure averaging of experimental data described above.

As a result, the longitudinal $\langle v_l \rangle$ and transverse $\langle v_t \rangle$ ultrasonic velocities increase with the increasing Al content that is opposite to behaviour of mass density ρ which decreases. Apparently, the decrease of ρ is due to lower mass density of Al. Besides, the ratio between the f.c.c. and b.c.c. phases was changed at the expense of increasing content of α-phase. This can result in the increase of the $\langle v_l \rangle$ and $\langle v_t \rangle$ values. In addition, refinement of the grain structure can also results in some increase of ultrasonic velocities. One can assume that refinement of the dendrite structure observed in the Al$_x$FeCoNiCr HEAs with Al content $x > 1$ [27] has similar action upon acoustic parameters.

The Young's modulus $\langle E \rangle$ and the Debye temperature Θ_D of the as-cast equimolar AlFeCoNiCuCr HEA is 172.8 GPa and 436 K, respectively (Table 2). The obtained value of $\langle E \rangle$ is higher than the tensile Young's modulus of the Al$_x$FeCoNiCr HEA ($x = 1$) 163 GPa [2] and the E values estimated by indentation of the AlFeCoNiCr HEA 168 GPa [18]. On the other hand, the $\langle E \rangle$ value is less than 180–182 GPa obtained in mechanical testing of the equimolar AlCoCrCuFeNi HEA [5–7]. The differences between dynamical ultrasonic and mechanically testing data are usually observed for the majority of metals and alloys that caused by different internal losses of mechanical energy in both experiments. At the same time, the Young's modulus and the Debye temperature of the as-cast equimolar HEA are high, which exceed those for the metals composing HEA and indicate high interatomic bond stiffness. This is consistent with the strong binding effect in HEAs mentioned in [2].

The relatively large share modulus $\langle G \rangle = 65.3$ GPa (Table 2) means high resistance to dislocations movement in the as-cast HEAs. The ra-

TABLE 2. The values of mass density (ρ), the averaged bulk ultrasonic velocities ($\langle v_l \rangle$, $\langle v_t \rangle$), the elastic moduli ($\langle E \rangle$, $\langle G \rangle$, $\langle B \rangle$), the ratio $\langle B \rangle / \langle G \rangle$, the Poisson's ratio ($\langle \eta \rangle$), and the Debye temperature (Θ_D) of the as-cast HEAs.

Alloy	ρ, g/cm^3	$\langle v_l \rangle$, m/s	$\langle v_t \rangle$, m/s	$\langle E \rangle$, GPa	$\langle G \rangle$, GPa	$\langle B \rangle$, GPa	$\langle G \rangle / \langle B \rangle$	$\langle \eta \rangle$	Θ_D, K
AlFeCoNiCuCr	7.378	5907.4	2975.0	172.8	65.3	161.8	2.477	0.322	436
Al$_{1.8}$FeCoNiCuCr	6.8465	6054.3	3142.9	180.1	68.7	159.4	2.321	0.312	458

tio $\langle B \rangle / \langle G \rangle = 2.477$ is lower as compared to pristine Al (2.93–3.08) and Cu (2.76–2.90) and higher than for Fe (1.99), Ni (2.02), Co (2.32) [29], Cr (2.27) [30]). These data along with the relatively low Poisson's ratio $\langle \eta \rangle$ indicate mean level of the plasticity of the studied HEAs.

Deviation from equimolar content of the $Al_x CuCrCoNiFe$ HEAs on Al ($x = 1.8$) increases the $\langle E \rangle$, $\langle G \rangle$ values by approximately 4–5%. The Θ_D also increased in magnitude of 5% (Table 2). This indicates growth of the interatomic bond stiffness and the resistance to dislocations movement. Plasticity of the HEA at $x = 1.8$ evidently decreased that is followed by decreasing the Poisson's ratio $\langle \eta \rangle = 0.312$ and the ratio $\langle B \rangle / \langle G \rangle = 2.321$. The obtained data are consistent with the increasing hardness and microhardness when concentration of Al riches 1.8 [7, 14, 27].

4. CONCLUSION REMARKS

1. Wide distributions of both the hyperfine magnetic fields on iron nuclei and the isomer shifts were detected in the as-cast $Al_x FeCoNiCuCr$ HEAs ($x = 1, 1.8$) that results from multiphase state (γ_1-, γ_2-, α-phases) and inhomogeneous short-range order in the phases. Deviation from equimolar content results in redistribution of the spin and charge s-electron densities at iron nuclei caused by change of phase content, redistribution of atoms in crystal lattices of phases.

2. The as-cast $Al_x CuCrCoNiFe$ HEAs are elastically inhomogeneous systems which are characterized by wide spread of acoustic parameters. Particularly, it concerns share ultrasonic velocity. The cause of the observed effect is their multiphase content and inhomogeneous distribution of chemical elements that becomes stronger due to texture.

3. The averaged elastic moduli ($\langle E \rangle$, $\langle G \rangle$, $\langle B \rangle$) and the Debye temperature Θ_D of the as-cast AlCuCrCoNiFe HEAs exceed ones for the elemental metals composing the HEA and indicate high interatomic-bond stiffness and high resistance to dislocations movement. The Poisson's ratio $\langle \eta \rangle = 0.312$–$0.322$ and the ratio $\langle B \rangle / \langle G \rangle = 2.321$–$2.477$ points to the mean plasticity of the alloys. Deviation from equimolar content upon Al ($x = 1.8$) increases the elastic moduli and the Debye temperature by approximately 4–5%.

4. The acoustic and elastic properties of the as-cast inhomogeneous HEAs can be adequately described by using ultrasonic equipment and measurement technique described in this work.

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