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# Influence of Technological Factors on the Structure and Properties of High-entropy FeCrMnCoNi Alloy

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The homogeneity of structure and physical properties (texture, microstructure, microhardness, magnetic susceptibility) for samples of high-entropy FeCrMnCoNi alloy cut from ingot after partial homogenization was investigated. On the example of one sample, it was showed that the microhardness, $H_V$, along the sample is not a constant, but is characterized by a certain distribution. X-ray studies of the fragments obtained by cutting this sample showed that each fragment was characterized by its texture. Degree and character of texture was estimated using the Wilson parameter. Scanning electron microscopic studies also show the heterogeneous nature of the structure distribution for each fragment, that is, the heterogeneous nature of the structure along the sample. Investigation of the magnetic properties by the Faraday method also allowed to establish the inhomogeneity of magnetic parameters (magnetic susceptibility, localized magnetic moment) along the samples, which is retained even in the case of additional deformation in the preparation of samples for research. Comparison of the presented parameter distributions and some parameters that are not included in the results (the size of coherent scattering regions, microstrains, density of the stacking faults) revealed no correlation between the investigated values. However, the study of the average composition of each fragment did not reveal a significant deviation of the composition from the nominal. On the basis of this, it is assumed that such inhomogeneity of the parameters under the condition of constant composition is a consequence of temperature gradients during the formation of the ingot and/or temperature gradients and the inhomogeneous nature of the deformation during the manufacture of the samples. Change in the balance between enthalpy and entropy contributions at low temperatures is considered one of the reasons for this heterogeneity. It is suggested that this leads to changes in the nature of the short-range order, which generally does not destroy the structure of the high-entropy alloy, but strongly affects the physical parameters.

**Keywords**: High-entropy alloy, Deformation, Microhardness, Microstructure, Magnetic susceptibility.

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## 1. INTRODUCTION

High-entropy alloys (HEAs) have been known for more than 10 years. Due to their multicomponent composition with simple crystalline structures of solid solution, such alloys exhibit a number of unique properties. As of today, there are a number of reviews on the structure and properties of such alloys [1-3], so we will not consider these properties in detail. HEAs predict the prospects of using as structural materials primarily due to their high mechanical properties. As for other properties, the prospects of using such alloys are only being considered, and their competition in this respect with traditional alloys is still rather weak.

Despite a fairly extended period of studies of these systems, a number of problems are not completely solved to this day. First of all, it concerns the role of entropy in the formation and stabilization of such a structure. Although a number of papers have been devoted to this problem [1-4], and the "entropy" principle was also used to create the so-called high entropy oxides [5], it cannot be definitively considered that entropy is the main factor, which determines the formation of a typical solid solution structure in such systems. As a result, the same alloy composition can often exhibit different microstructure characteristics and, therefore, different properties.

Recently, it has been established that alloys, which are considered as single-phase HEAs, are in fact characterized by a metastable state. Even in the well-known and studied alloy of FeCrMnCoNi composition, there is a consistent fallout of different phases at relatively low temperatures [6-8], and its structural features and certain fluctuations of the composition have been explained as a consequence of different cooling conditions, taking into account the CrFeCo-NiMn pseudo-diagram of state with unlimited solubility [9]. These and some other results indicate that the fine structure of the alloys does not always correspond to the structure, which is inherent to the completely disordered solution. Our studies also point to the important role in the formation of electrical and magnetic properties of different types of inhomogeneities with different short-range nature that can occur in alloys under conditions that deviate from equilibrium [10, 11].

In addition, it should be borne in mind that the production of alloys and the practical use of products made of these materials lead to a structural state far from equilibrium one. Usually ingots of alloys obtained by any method and samples removed from them for research are subjected to homogenization by prolonged annealing. As a result, an equilibrium structural state with recrystallized grains is formed [12]. However, economically feasible is to obtain material from structurally unstable state when, for example, the obtained ingots are cut into billets of the required shape and size, and from the latter the necessary product is formed (by rolling, drawing, forging, etc.). However,

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both the structure of the ingot itself and the features of the structure intermediate workpiece have been studied rather poorly, not to mention their relationship with the various properties and properties of the finished product.

Therefore, in this work, the structure and properties of the samples of the FeCrMnCoNi alloy after cutting from the ingot were investigated. In this case, the structural state of the ingot was preserved partially or completely and additional changes were made to the structure due to the cutting process.

## 2. EXPERIMENTAL METHODS

For such studies, the FeCrMnCoNi alloy ingot, the fusion method of which is described in [10, 11], was cut by the spark method into long (5 cm) specimens with a cross-section of $\sim 2 \times 1$ mm$^2$. Although the method of manufacturing the alloy included homogenizing annealing, and the final structure of the ingot was not investigated, the cutting method unambiguously made its own and quite significant contribution to the peculiarities of the alloy structure. In particular, the surface of the specimens was quite different in structure and phase composition compared to the ingot owing to thermal action and oxidation processes. Therefore, such a damaged surface layer was removed by polishing.

For the studies, the standard techniques and methods described, for example, in [10, 11] were used. X-ray diffraction studies were performed on a DRON-4 diffractometer using the Co-$K_\alpha$ radiation. Magnetic studies were performed on a Faraday magnetometer. The hardness was determined on the PMT-2 hardometer. The scanning electron microscopy studies were performed on a Teskan Vega 3 instrument.

## 3. EXPERIMENTAL RESULTS

Microhardness is the parametr that characterizes not only the mechanical properties, but also allows us to detect the local character of the inhomogeneities that occur in different systems. During the testing of the properties of HEAs, it was found that the microhardness according to Vickers, $H_V$, for the tested samples differed in value not only from sample to sample, but also showed some differences in different areas of the same sample. Such differences cannot be eliminated even by statistical processing of the multiple measurements of $H_V$. In this regard, a study of the distribution of microhardness along the sample with a length of 5 cm was carried out. The obtained dependence of $H_V$ vs. $L$ ($L$ is the distance measured from one of the two ends of the sample) for one of the FeCrMnCoNi samples is shown in Fig. 1. The data obtained for each point is the result of the averaging for each value of $L$. This averaging was performed on the results of about 20 measurements, the number of which was limited to the width of the sample. Although the number of measurements at each point is relatively small, it is clear that the microhardness monotonously changes along the sample and is not constant, as in the case of homogeneous in composition and structure systems.

Another feature that is also found in the HEAs is the texture of the samples [3, 4]. It manifests atypical

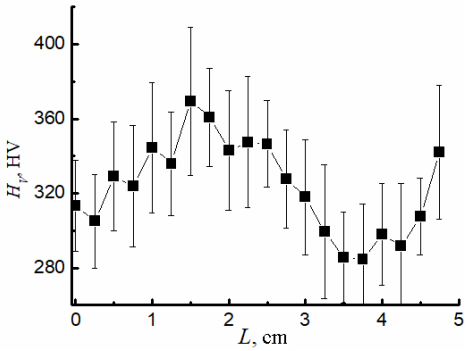



**Fig. 1** – The distribution of microhardness, HV, along the length $L$ of the sample of the FeCrMnCoNi HEA

relative intensity of the diffraction peaks. Typically, texturing arises during the deformation of metals, but can also occur in the cast specimens. Our XRD studies of fragments cut from the five samples have shown that not only the degree of texture, but also the character of the texture varies from the sample to the sample. This data will not be analyzed in detail as the cutting of the ingot into the specimens was done without controlling the position in the ingot. Instead, XRDs and other studies of the properties were conducted on the separate sample. For this purpose, the sample was used, for which the $H_V$ vs. $L$ dependence was obtained (Fig. 1). For XRD and other property studies, this sample was cut into 5 fragments (BL1-BL5). Subsequently, the BL2 fragment was additionally cut into two parts (BL2-1 and BL2-2).

Fig. 2 presents the diffraction patterns obtained for each of the fragments. From these diffraction patterns it can be seen that each fragment is characterized by its distribution of the intensities of the diffraction maxima, so, by different textures. For the approximate quantification of these textures, the parameter suggested by Wilson [13] was used:

$$\Phi_{hkl} = \frac{\left(I_{T,hkl}/I_{E,hkl}\right)\sum\limits_n p_{hkl}}{\sum\limits_n \left(p_{hkl} I_{T,hkl}/I_{E,hkl}\right)},$$

where $n$ is the number of diffraction maxima detected at the given angular interval of studies, $I_{Thkl}/I_{Ehkl}$ is the ratio of the intensities of the diffraction maximum ($hkl$) of the textured (index $T$) and nontextured (index $E$)

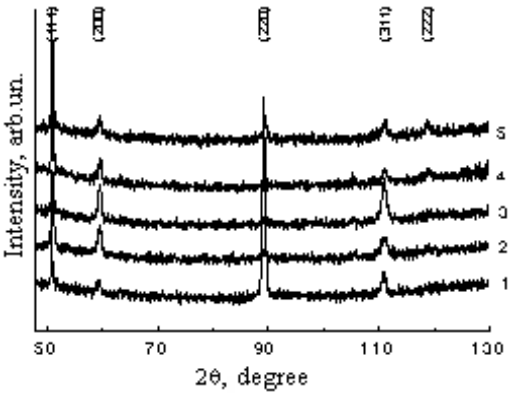



**Fig. 2** – The diffraction patterns obtained from the BL1-BL5 fragments (the number near the diffraction pattern corresponds to the fragment number)

samples, $p_{hkl}$ is the multiplicity factor of the ($hkl$) diffraction peak. The relative intensities of the diffraction maxima obtained for the FeCrMnCoNi powder diffraction patterns were used as the etalon. Only the first three diffraction maxima were analyzed: (111), (200), and (220), since the intensities of the other maxima were relatively weak because of the presence of static distortions due to the different sizes of the atoms forming the HEA.

Fig. 3 presents the dependences $\Phi_{hkl}$ vs. $L$ for maxima (111), (200) and (220). This figure shows that the texture changes along the sample. If the edges of the sample are dominated by the number of grains with the (111) planes oriented parallel to the surface of the sample, then the (200) planes dominate in the middle part. But none of the dependences $\Phi_{hkl}$ vs. $L$ correlates with the dependence of $H_V$ vs. $L$ obtained for this sample (see Fig. 1 and Fig. 2).

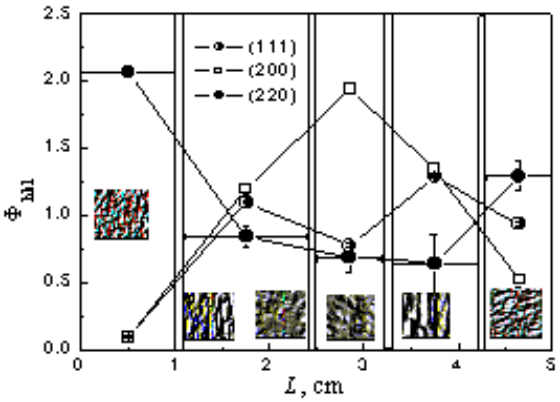



**Fig. 3** – $\Phi_{hkl}$ parameters of Wilson calculated from the intensities of the (111), (200) and (220) diffraction maxima for the BL1-BL5 fragments. The length of each fragment is limited by parallel lines, the distance between which corresponds to the part of the sample removed from the sample when cut into fragments. On the insets: the microstructure of the central part of each fragment

The microstructure along the sample is also not homogeneous. For example, Fig. 4 shows the parts of SEM-images of the BL1-BL5 fragments. As seen, each fragment is characterized by its structure and size of the components (grains). Certain heterogeneity of the structure is also found within each fragment, which is quite evident from the BL2-1 and BL2-2 SEM-images, for which diffraction study was performed as a single BL2 sample.

It is quite difficult to establish certain correlation features between the microstructure, texture parameters and microhardness, as seen from Fig. 3. This figure contains the parts of the images from Fig. 4, which are shown for each fragment.

The length distributions of the parameters obtained by the profile analysis of the diffraction maxima (the size of the coherent scattering regions, microdeformation, density of the stacking faults) were also not uniform. For this purpose, both the approximation method and the Fourier analysis were used. These results will not be considered, and the methods themselves and their capabilities are described in the classical literature on the diffraction methods [13].

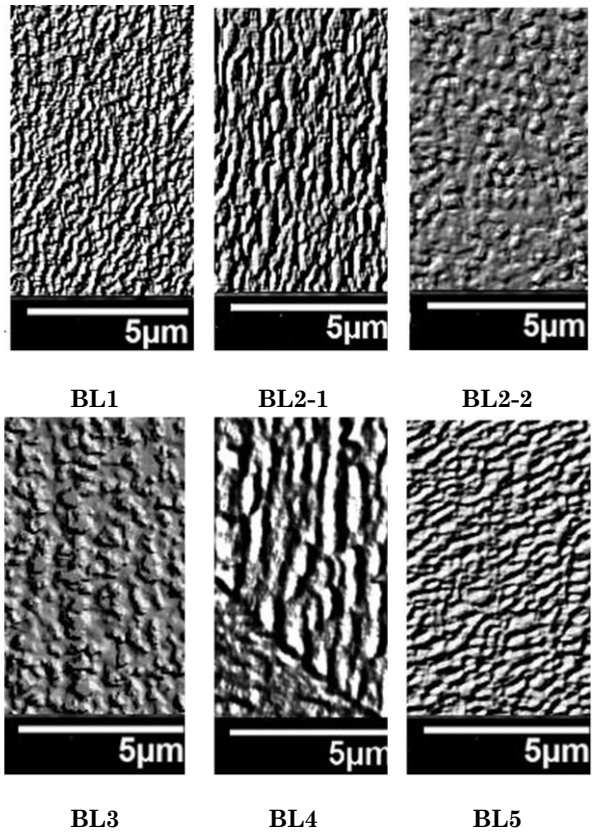



**Fig. 4** – SEM images of the surface of the fragments BL1-BL5

To determine the cause of such a heterogeneous structure on the sample, the integral composition of each fragment BL1-BL5 was investigated. Fig. 5 shows the result of such research. As can be seen, the deviation of the content of each component does not exceed ± 0.2-0.3 at. % of the nominal (20 at. %), and the fluctuations of the content of each component along the sample are no more than ± 0.1 at. %. The only exception is Cr content, which changes on a slightly larger value (+ 0.5 at. %) along the sample. Such fluctuations in composition are typical of the CoCrFeMnNi alloys [7, 14] and cannot explain the structural differences that are found in the diffraction studies. Among the impurities found in the sample is Si, which is obviously an admixture of technically pure iron (content does not exceed 0.01 at. %). In addition, fragments BL2-1 and BL2-2 contained a small amount (less than 0.01 at. %) of Zn and Cu, which are apparently the products of erosion of the elements contained in the arc melting device (brass components).

Considering the obtained result, it can be assumed that the composition along the sample is approximately constant, and its insignificant fluctuations and impurities cannot explain the heterogeneous nature of the structure along the sample.

Magnetic properties have a rather high sensitivity to the nature of order, in particular to the short-range order. In this regard, the temperature dependences of the magnetic susceptibility, $\chi(T)$, of the edges of each fragment BL1-BL5 were investigated. The character of the $\chi(T)$ dependences proved to be quite similar for all measurements and similar to the $\chi(T)$ dependences in [11].

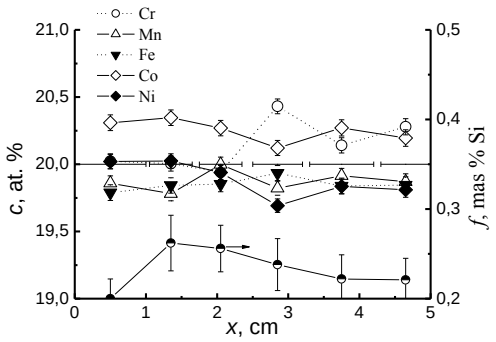



**Fig. 5** – Distribution of element concentration (at. %) over the length of the sample obtained from the average concentration of elements of BL1-BL5 fragments (right axis – concentration (at. %) of impurity silicon along the length)

The first measurement cycle showed irreversibility, which significantly decreases or almost disappears in subsequent heating-cooling cycles. The magnitude of irreversible $\chi(T)$ changes can be seen in Fig. 6a, which shows the magnitude of the magnetic susceptibility at room temperature ($T = 300$ K), $\chi_{300}$, in the initial state (before the first heating cycle) and at the end of the measurements (after the second cooling cycle) for the edges of each fragment (for the beginning and for the end). Fig. 6b shows the distribution of magnetic moments obtained from the $\chi(T)$ dependences on the assumption that the low-temperature part of the dependence follows the $\chi \sim 1/T$ dependence, that is, the Curie dependence or the Curie-Weiss dependence in the limiting case $T \gg \Theta$, where $\Theta$ is the Curie temperature. The arguments for this are discussed in [11].

As can be seen, in the first measurement cycle, both $\chi_{300}$ and $\mu$ change slightly enough in length, which can be considered as a result of additional deformation in the preparation of samples. The relaxation processes that take place in the measurement process partially remove such deformation effects and a certain equilibrium state is established. Such structure is the result of the imposition of deformation on the structural state, which established in this part after the casting. Thus, the dependence of $\mu$ vs. $L$ obtained in the last cooling cycle only approximately reflects the actual distribution of magnetic moments in the length. But, as can be seen from Fig. 6b, $\mu$ is not a constant value, and like other parameters, varies along the length.

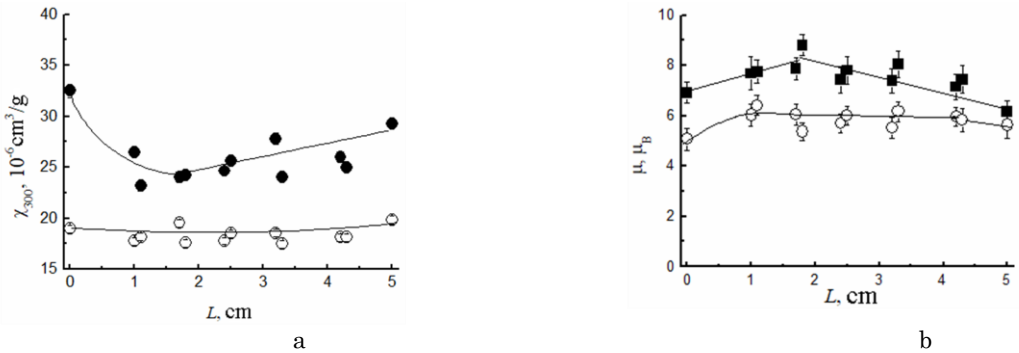
In the case of a composition constant in length and provided that large values of $\mu$ are to be considered from the point of view of clustered structures united by magnetic interaction [11], it can be considered that the heterogeneity of distribution $\mu$ is a consequence of the difference in the short-range order in such clusters along the sample length. Obviously, such inhomogeneity in the short-range order is formed already upon obtaining of the ingot, and it determines the heterogeneity of the distribution of structure and physical properties.

It is quite logical to ask how far the inhomogeneities found can affect the structure and properties of products that can be obtained from such a relatively inhomogeneous ingot. Consider this an example of the effect of deformation as one of the possible methods of obtaining products.

The effect of deformation is quite clearly traced to the nature of the dependence of microhardness $H_V$ on the rolling deformation $\varepsilon$. In Fig. 7, this dependence is shown for both the sample subjected to deformation without heat treatment and the sample annealed at $T = 950$ °C.

Annealing at $T = 950$ °C corresponds to the temperature where recrystallization processes which intensively occur [12]. Therefore, the annealed sample has a microhardness $H_V = 250 \pm 25$ HV, practically corresponding to the value of microhardness, which is most commonly found in the literature for recrystallized FeCrCoNiMn alloy (125 ÷ 220 HV according to the results [12, 15, 16]). Deformation of this sample leads to an increase in the microhardness with deformation, which agrees with the general ideas about the deformation effect on this value and is consistent with the results for the recrystallized FeCrCoMnNi HEAs of other works [16]. This is also consistent with the general understanding of the mechanisms of deformation (strengthening) during cold rolling of steel and other metal alloys [17], according to which the strength (including hardness) increases in the initial stages of deformation. Further, the intensity of strengthening with enhancement of the deformation decreases. Also, such the nature of the deformation effect is observed for the dependence of $H_V$ on $\varepsilon$ for the annealed specimen (Fig. 7): $H_V$ increases up to $\varepsilon \sim 50$ %, and at large values of $\varepsilon$ remains almost unchanged. And only when deformation $\varepsilon \sim 95$ %, there is a slight increase in $H_V$.

In contrast to the influence of $\varepsilon$ on the $H_V$ discussed above, the nature of the $H_V(\varepsilon)$ dependence for a non-annealed specimen exhibits a somewhat anomalous be-




a b

**Fig. 6** – Distribution of magnetic susceptibility values at a temperature $T = 300$ K, $\chi_{300}$, (a) and localized magnetic moments, $\mu$, (b) obtained from the first heating cycle (light symbols) and from the second cooling cycle (black symbols) along the length of the sample

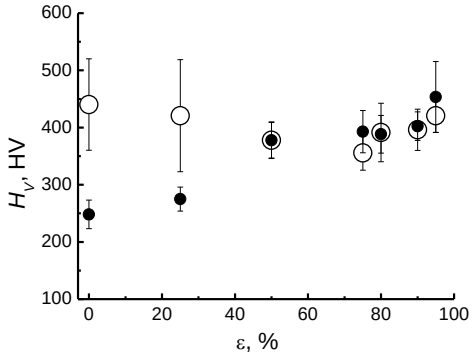



**Fig. 7** – Dependence of microhardness, $H_V$, on the magnitude of rolling deformation, $\varepsilon$, of samples deformed without annealing (light circles) and annealed at 950 °C for 30 min before rolling (dark circles)

havior: when deforming up to $\varepsilon \sim 50$-80 %, a decrease in $H_V$ is observed from the initial state ($H_V = 440 \pm 80$ HV) to a value close to $H_V$ of the sample deformed after annealing. At the large deformations, the dependences of $H_V(\varepsilon)$ almost coincide.

Since, according to Fig. 1, the value of microhardness varies from 220 to 440 HV over the length of the sample, it should be expected that the nature of the "anomaly" in the initial stages of deformation will differ for different fragments up to some value of $\varepsilon$. At a certain value of deformation (about 50 %), the "inhomogeneity" will disappear, and the differences in the $H_V(\varepsilon)$ dependences of different samples should vanish.

It should be noted, despite the different dependence of $H_V$ on $\varepsilon$ for annealed and not annealed specimens in the initial stages of deformation, in both cases, there is an increase in the "deformation" thermoelectric force, $E_\varepsilon$, with $\varepsilon$ [10]. The value of $E_\varepsilon$ is determined by the defectivity of the structure, so in the case of deformation the number of defects increases in both cases. Since defects also determine the mechanical parameters, it must be expected to increase the microhardness for both samples, because the defectivity is one of the reasons for strengthening. However, according to Fig. 7, annealed and non-annealed samples exhibit different $H_V$ behavior with $\varepsilon$ in the initial stages of deformation. Therefore, only the changes in defectivity cannot explain this behavior.

## 4. DISCUSSION

In general, it is considered that the structure of a single-phase HEA is a completely statistically disordered solid solution with no short-range order in it. The absence of the short-range order was neither confirmed, nor rejected by the results of XRD, electron microscopy, etc. [18]. But just in terms of a completely disordered solid solution, the properties of the HEAs are analyzed.

However, there are many examples where different types of heterogeneities were observed in the structure of the same HEAs (some of them were analyzed in [19]). Recent published results also confirm the view that due to the evolution of the entropic contribution during cooling, different types of elemental segregation, precipitates, chemical ordering and spinodal decomposition regions, etc. can be formed. But in this case there is a considerable amount of the disordered phase and the main advantages of the structure of HEAs remain.

Thus, the result of the inhomogeneous distribution of the parameters of the structure and some physical properties is not relatively new at present, and is a reflection of those technological factors that influence the sample from the moment of melting of the alloy to the time of production of the objects for research or operation. Since the final stage of forming the structure of HEAs is the casting of a liquid alloy into the mold, the ingot structure will be determined by the nature of the heat sink during cooling (even in the case of relatively high cooling rates, which realizes during the casting of small HEA mass into a copper form). The structure of the ingot, quite well known in the field of materials science, will obviously take place also during the formation of the HEA, and its character will be inherited even with homogenizing annealing. It follows that the distribution of structural parameters will be determined by how the ingot is cut out for research. Unfortunately, in the initial stages of research this factor was not taken into account.

Another factor that can affect the structure of the samples is the ingot cutting on the samples for research. The thermal and mechanical effects, especially in the case of small samples, can significantly alter the structure and nature of its heterogeneity. Again, this factor was hardly controlled. In this respect, the results of [20], where the structure, composition, and microhardness distribution of a wire radius of 16.5 mm diameter obtained from the ingot by drawing with the total diameter change (reduction) of 60 % are rather interesting. The microhardness on the surface is found to be 15 % lower than that of the axis, which is associated with dynamic annealing during elongation. This example shows the significant role of technological factors.

Studies of the microstructure parameters and the nature of the distribution of physical properties, such as microhardness and magnetic susceptibility, clearly indicate that the heterogeneity of the physical properties is also related to the structural heterogeneity. Specifically, which of the parameters (different grain sizes, microstrains, defects, etc.) determines a particular property is difficult to establish. Most likely, in this case, the short-range order that arises from the competition of the entropy and enthalpy contribution under heterogeneous external factors has an important influence. In this case, the multicomponentity of the material can be reflected in the short-range order, even when the average composition is close to the nominal one.

Qualitatively, the obtained results can be explained as follows. The formation of alloys is associated with a configurational increase in the entropy contribution of $\Delta S$. But since it is not $\Delta S$ but $T\Delta S$ that determines the energy of different processes, it is clear that such a contribution depends on the temperature, also. Therefore, during the formation of HEAs, the ingot formation process will take place with a temperature gradient and, as a consequence, a gradient of the $T\Delta S$ contribution.

In this respect, the results of the formation of amorphous metal alloys by melt spinning should be considered when, at enormous cooling rates (up to $10^6$ K/s), the formed structure of the thin (about tens of micrometers) amorphous tape reveals a difference along the thickness of the tape. One of the main reasons for this is the different speed of quenching caused by the different con-

tact environments – metal drum and ambient atmosphere. Therefore, the pseudo-binary CrFeCo-NiMn diagram of the state proposed in [9] can be considered as the basis for explaining the different structural state of the FeCrCoMnNi alloy under the gradient condition, that is, at different cooling rates.

In addition, in the presence of the $T\Delta S$ gradient, the structure of the HEAs will be restructured so as to compensate for the decrease in $T\Delta S$ due to the enthalpy component $\Delta H$. Such compensation can occur with atomic jumps within the nearest neighbors, as opposed to moving atoms over long distances, as in the case with diffusion. This was considered qualitatively in [11]. This will mean the presence of different short-range order for different values of $T\Delta S$, which is formed by the condition of the gradient $T$.

The deformation energy, especially when it is heterogeneous in nature, can also disrupt the balance created between the entropy and enthalpy. This can also be seen as the reason for the formation of a new character of short-range order. Such process will especially reveal itself under the conditions of plastic deformation, when the processes of atomic displacement occur. Such displacements will occur primarily in the least "connected" structural units, leading to the formation of new structural units that also reduce the energy of the system by changing $\Delta H$ if $T\Delta S$ is reduced due to low temperatures.

Although the process under consideration requires a deeper theoretical analysis, it provides a logical explanation for the occurrence of all kinds of heterogeneities under the condition of constant composition.

## 5. CONCLUSIONS

Consequently, the studies show that the structure of the samples is significantly determined by the regimes of thermal and mechanical treatment. The heterogeneous nature of the temperatures and deformations leads to the inhomogeneous structure, which reveals itself in the heterogeneity of the physical properties (the microhardness, texture and magnetic properties). At the homogeneous composition, such features can be explained only in terms of the formation of certain types of the short-range orders (the cluster structures) due to the competition of the entropy and enthalpy contributions at low temperatures.

Such features should be taken into account when forming products at low temperatures and when operating products in the conditions of the rigid (near plastic) deformation.

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## Вплив технологічних факторів на структуру та властивості високоентропійного сплаву FeCrMnCoNi


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У статті досліджено однорідність структури та фізичних властивостей (текстура, мікроструктура, мікротвердість, магнітна сприйнятливість) зразків високоентропійного сплаву FeCrMnCoNi, вирізаних із злитку після часткової гомогенізації. На прикладі одного із зразків показано, що мікротвердість, $H_V$, вздовж зразка є не постійною величиною, а характеризується певним розподілом. Рентге-

нівські дослідження фрагментів, одержаних при розрізуванні цього зразка, показали, що кожен із фрагментів характеризується своєю текстурованістю, яка оцінювалась з використанням параметру Вільсона, що визначає характер текстури. Растрово-електронно мікроскопічні дослідження також показують і неоднорідний характер розподілу структури для кожного із фрагментів, тобто неоднорідний характер структури вздовж зразка. Дослідження магнітних властивостей методом Фарадея дозволили також встановити і неоднорідність магнітних параметрів вздовж зразків (магнітна сприйнятливість, локалізований магнітний момент), що зберігається навіть у випадку додаткової деформації при приготуванні зразків для досліджень. Співставлення представлених розподілів параметрів та деяких параметрів, що не увійшли у статтю (розмір областей когерентного розсіювання, мікронапруги, густина дефектів упаковки) не виявило ніяких кореляції між дослідженими величинами. При цьому, дослідження усередненого складу кожного з фрагментів не знайшло суттевого відхилення складу від номінального. На основі цього зроблено припущення, що така неоднорідність параметрів при умові постійності складу є наслідком градієнтів температур при формуванні злитку та/або градієнтів температур та неоднорідного характеру деформування при виготовленні зразків. Зміна балансу між ентальпійним та ентропійним внеском при низьких температурах розглядається як одна з причин такої неоднорідності. Припущено, що це призводить до змін характеру ближнього порядку, що в цілому не руйнує структуру високоентропійного сплаву, але сильно впливає на фізичні параметри.

**Ключові слова**: Високоентропійний сплав, Деформація, Мікротвердість, Мікроструктура, Магнітна сприйнятливість.