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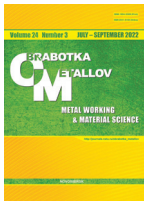

Comparison of approaches based on the Williamson-Hall method for analyzing the structure of an $Al_{0.3}CoCrFeNi$ high-entropy alloy after cold deformation

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ABSTRACT

Introduction. High-entropy alloys (*HEAs*) belong to a new and promising class of materials that are attracting the attention of both scientists and engineers from all over the world. Among all alloys of the $Al_xCoCrFeNi$ system, *HEAs* with $x \leq 0.3$ attract special attention. Materials with this composition are characterized by the presence of only one phase with a face-centered cubic lattice (*FCC*). Such alloys have high ductility, excellent corrosion resistance and phase stability at high temperatures. **The purpose of this work** is to compare several methods of profile analysis on the example of plastically deformed ingots of a high-entropy $Al_{0.3}CoCrFeNi$ alloy. **The methods of investigation.** Using several methods of profile analysis of X-ray diffraction patterns, the structures of the cold-worked high-entropy alloy $Al_{0.3}CoCrFeNi$ are studied. In addition to the classical *Williamson-Hall* method, the analysis was carried out using a modified one, as well as a method that takes into account the anisotropy of the elastic properties of the crystal lattice. **Research material.** Ingots of the high-entropy $Al_{0.3}CoCrFeNi$ alloy deformed by cold rolling with a maximum reduction ratio of 80% were used as the object of the study. Samples were cut from the obtained blanks, which were studied by the method of synchrotron radiation diffraction according to the "transmission" scheme along two (longitudinal (*RD*) and transverse (*TD*)) directions of rolled products. **Results and discussion.** It is shown that the use of the classical *Williamson-Hall* method leads to a significant error in the approximation of experimental results. The modified *Williamson-Hall* method has the smallest approximation error and can be recommended for studying the $Al_{0.3}CoCrFeNi$ alloy. An analysis of deformed samples using this method made it possible to reveal several features of the formation of defects in the crystalline structure, which are in good agreement with the classical concepts of the mechanisms of plastic deformation. First, an increase in the degree of deformation of the high-entropy $Al_{0.3}CoCrFeNi$ alloy leads to an almost uniform increase in the number of twins and stacking faults. Secondly, with an increase in the degree of reduction, there is a decrease in the fraction of edge dislocations and an increase in the fraction of screw dislocations in the material. The results obtained correlate well with the results of microhardness measurements.

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Introduction

High-entropy alloys (*HEAs*) represent a new and promising class of materials that are attracting the attention of both scientists and engineers from all over the world. [1, 2]. The most common and studied are alloys based on a combination of cobalt, chromium, iron, nickel and an additional element. In particular, many scientific works are devoted to such alloys as *CoCrFeMnNi* (Cantor's alloy) and $Al_xCoCrFeNi$ alloys [3–5].

Special attention of researchers attracts $Al_xCoCrFeNi$ alloys with $x \leq 0.3$. Materials with this composition consist of only one face-centered cubic (*FCC*) phase. Such alloys have high ductility, excellent corrosion resistance and phase stability at high temperatures. At the same time, these materials possess low hardness and yield strength. The strength of these alloys can be significantly improved by plastic deformation with subsequent heat treatment. According to a number of literary sources, the thermomechanical processing of the $Al_{0.3}CoCrFeNi$ alloy leads to its strengthening and an increase in hardness but allows retaining a reasonable level of ductility [6–8].

One of the effective methods for studying the structure of plastically deformed alloys is the peak profile analysis of the X-ray diffraction patterns. This technique makes it possible to evaluate the defects in the crystal structure of alloys. The most common peak profile analysis approach is the classical *Williamson-Hall* method. The use of this method makes it possible to estimate the distortions of the crystal lattice and the size of coherent scattering regions (*CSRs*). However, the *Williamson-Hall* method is known to have a high approximation error during the analysis of materials with high anisotropy of elastic properties. Therefore, special corrections are introduced during the analysis, that take into account the dependence of elastic properties on the direction in the crystal lattice. Even though these methods are widely used in the analysis of metals and alloys, there are no examples of exhaustive comparative analysis of peak profile analysis methods for studying the structure of high-entropy alloys. In this study, several peak profile analysis methods are compared by using the plastically deformed ingots of an $Al_{0.3}CoCrFeNi$ high-entropy alloy as an example. Using various methods, defects in the crystal structure were evaluated and its relationship with the microhardness of the deformed alloy was shown.

Samples preparation. Methods for studying the structure and properties of materials

In this work, the ingots of the $Al_{0.3}CoCrFeNi$ high-entropy alloy were used. The ingots were obtained from commercially pure metals by argon-arc melting in a water-cooled copper crucible. To distribute chemical elements evenly, remelting was carried out at least 10 times. Weight loss during smelting did not exceed 0.2 %. The elemental composition of the ingots was evaluated by energy dispersive X-ray spectroscopy using a scanning electron microscope *EVO50 XVP* (Carl Zeiss) equipped with detector *X-Act* (Oxford Instruments). According to data obtained, the deviation of the actual composition did not exceed 0.6 at. %.

It is well known that the structure of materials obtained by melting and casting methods is characterized by the presence of large dendrites, as well as a heterogeneity of the chemical composition (i.e., dendritic segregation). In order to obtain a more homogeneous composition and a fine-grained structure, thermomechanical processing of ingots was carried out. It was carried out by cold rolling with a reduction of 20 % and long-term low-temperature annealing (400 °C during 24 hours). The higher annealing temperatures were not used because some high-entropy alloys of the $Al_xCoCrFeNi$ system have a phase transition with formation of the *B2* and $L1_2$ ordered phases (space group $Pm\bar{3}m$) at temperatures exceeding 400 °C [6, 9]. The results of X-ray diffraction analysis indicate that this thermomechanical processing contributed to the relaxation of the structure and did not lead to the formation of new phases (Fig. 1).

After the thermomechanical treatment, the high-entropy alloy ingots were subjected to cold rolling with reduction of 20; 40; 60 and 80 %. The reduction during the single rolling pass was ~ 2 %.

Afterall, the samples were cut for X-ray diffraction analysis and microhardness testing. The structure and properties of materials along the rolling direction (*RD*) и transverse direction (*TD*) were investigated.

The X-ray diffraction analysis was carried out by the synchrotron X-ray diffraction method in transmission using the beamline *P07* of *Petra III* source of *Deutsches Elektronen-Synchrotron* (*DESY*). The wavelength of the radiation used was 0.14235 nm. It corresponds to energy of 87.1 keV. A *2D PerkinElmer XRD 1621* detector was used to record the diffraction patterns. The screen resolution of the detector was 2048 × 2048 px. The screen area was 409.6 mm × 409.6 mm. The distance from the sample to the detector was 1.05 m.

The resulting diffraction patterns were reduced to a one-dimensional form by azimuthal integration using the *pyFAI* library [10]. Examples of the obtained two-dimensional and one-dimensional diffraction patterns are shown in Fig. 1.

For peak profile analysis, one-dimensional diffraction patterns were described by a function of the following form:

$$I_{pattern}(2\theta) = \sum_{i=1}^{10} I_i(2\theta) + \sum_{i=0}^{7} a_j(2\theta)^j, \quad (1)$$

where the first sum determines the contribution to the intensity of ten diffraction maxima, and the second sum is a 7th order polynomial to describe the background. In turn, the profile of each of the diffraction maxima was described by the pseudo-Voigt function, which is generally written as:

$$I_i(2\theta) = I_0\left[\eta L(2\theta) + (1-\eta)G(2\theta)\right], \quad (2)$$

where I_0 – the value of the maximum intensity of the diffraction peak; η – *Lorentz* function contribution; $L(2\theta)$ and $G(2\theta)$ – *Lorentz* and *Gauss* functions, respectively. These functions look like:

$$L(2\theta) = \frac{\left(0.5\beta[1-A]\right)^2}{\left(0.5\beta[1-A]\right)^2 + (2\theta - 2\theta_0)^2} \quad (3)$$

and

$$G(2\theta) = \exp\left(\frac{-\pi(2\theta - 2\theta_0)^2}{\left(0.5\beta[1-A]\sqrt{\pi / \ln 2}\right)^2}\right), \quad (4)$$

where $2\theta_0$ – angular position corresponding to the maximum value of the peak intensity; β – full width at half maxima (*FWHM*); A – diffraction peak asymmetry parameter ($-1 \le A \le 1$).

The instrumental contribution was taken into account by using the *Caglioti* function. The parameters of the function were determined by analyzing the diffraction pattern of the *HEA* sample after cold rolling and long-term annealing at 400 °C.

To carry out X-ray diffraction analysis the classical *Williamson-Hall* model was used. According to this model, the peak broadening depends on the parameters of the sample microstructure as follows:

$$\Delta K = \frac{0.9}{D} + 2\varepsilon K, \quad (5)$$

where $K = \frac{2\sin\theta}{\lambda}$ – reciprocal coordinate; $\Delta K = \frac{\cos\theta 2\Delta\theta}{\lambda}$; ε – relative lattice distortion; λ – wavelength; D – average «visible» size of *CSRs*.

As noted in the introduction, the anisotropy of the elastic properties of materials causes a high error in the approximation of diffraction data using the classical methods of peak profile analysis. Therefore, in this work, in addition to the classical *Williamson-Hall* model, several other models were used.

In some cases, the approximation error can be reduced by introducing a correction based on the assumption that crystal lattice distortions in one of the directions depend on the elastic modulus of the crystal along this direction [11]. This model can be written in the following way:

$$\Delta K = \frac{0.9}{D} + \frac{\sigma}{E_{hkl}} K, \quad (6)$$

where σ is isotropic elastic stress; E_{hkl} – modulus of elasticity along the normal direction to the plane (*hkl*).

In addition, the obtained data were analyzed using a model based on the assumption of the dislocation nature of crystal lattice distortions. This approach is called the modified *Williamson-Hall* method [12]. In the case of cubic polycrystalline materials, the equation underlying this model has the following form:

$$\frac{\Delta K^2 - [\alpha + \beta \cdot W(g) / a]}{K^2} \cong A\overline{C_{h00}}[1 - qH^2], \quad (7)$$

where $\alpha = (0.9/D)^2$; β – parameter that shows the probability of detecting stacking faults and twins; $W(g)$ – coefficients depending on crystallographic direction indices [*hkl*] [13, 14]; *a* – lattice parameter; *A* – parameter depending on the average density of dislocations, the average length of the Burgers vector and the arrangement of dislocations; $\overline{C_{h00}}$ – average dislocation contrast factor along [*h*00] direction; *q* – parameter depending on the elastic properties of the material; $H^2 = (h^2k^2 + h^2l^2 + k^2l^2) / (h^2 + k^2 + l^2)^2$.

According to literature, the modified *Williamson-Hall* method has the lowest approximation error [11, 15]. A more detailed description of the implementation of this method is described elsewhere [11, 15, 16].

The microhardness of the samples was evaluated by using the *Vickers* method on a *Wolpert Group 402MVD* semi-automatic hardness tester. The load on the tetrahedral diamond indenter was 0.98 N, the holding time under load was 10 s.

Research results

It is believed that the multielement composition of high-entropy alloys leads to significant distortions of its crystal lattice even before plastic deformation. This feature can possibly cause an additional broadening of the diffraction peaks of undeformed samples. In addition, the instrumental broadening of diffraction maxima arises due to the instrument which is used for the diffraction experiment. In order to take into account, the contribution of both factors and analyze only the effects caused by a change in the structure of the samples, an undeformed *HEA* sample of the same composition with a homogeneous structure was used as a reference. For this purpose, preliminary thermomechanical processing of *HEA* was carried out. This processing consisted in plastic deformation and subsequent long-term low-temperature annealing. According to the results shown in Fig. 1 *a*, *b*, the structure of the alloy after the deformation and low-temperature annealing is characterized by a more uniform spatial orientation of crystallites (which is evidenced by the presence of complete diffraction rings) and a low level of microstresses (which is evidenced by the small width of diffraction maxima). Subsequent cold rolling (Fig. 1, *c*, *d*) leads to a significant broadening of the diffraction maxima, which indicates an increase in the number of defects in the crystalline structure.

The peak profile analysis of diffraction patterns of plastically deformed alloys makes it possible to estimate the number and the type of defects in the crystal structure based on the parameters of diffraction maxima. Thus, the assessment of the width of diffraction maxima using the classical *Williamson-Hall* method (Equation 5) makes it possible to determine the relative distortions of the crystal lattice and the *CSRs* sizes. However, it is known that this method is the least accurate with the significant error of the approximation of experimental results. Therefore, some corrections based on the anisotropy of crystal properties are often introduced during the analysis of X-ray diffraction data by using the peak profile analysis methods. The simplest way to account for anisotropy is to introduce into the calculation the elastic modulus for the normal to the planes (*hkl*) crystallographic directions (Equation 6). Table shows the values of the elastic moduli of the $Al_{0.3}CoCrFeNi$ alloy for the diffraction maxima analyzed in the work.

Another, less common, but in many cases more effective way to improve the approximation accuracy is to use a model based on the dislocation theory of elastic distortions of the crystal lattice. This type of models is called modified in the literature. They were described in detail in the works of

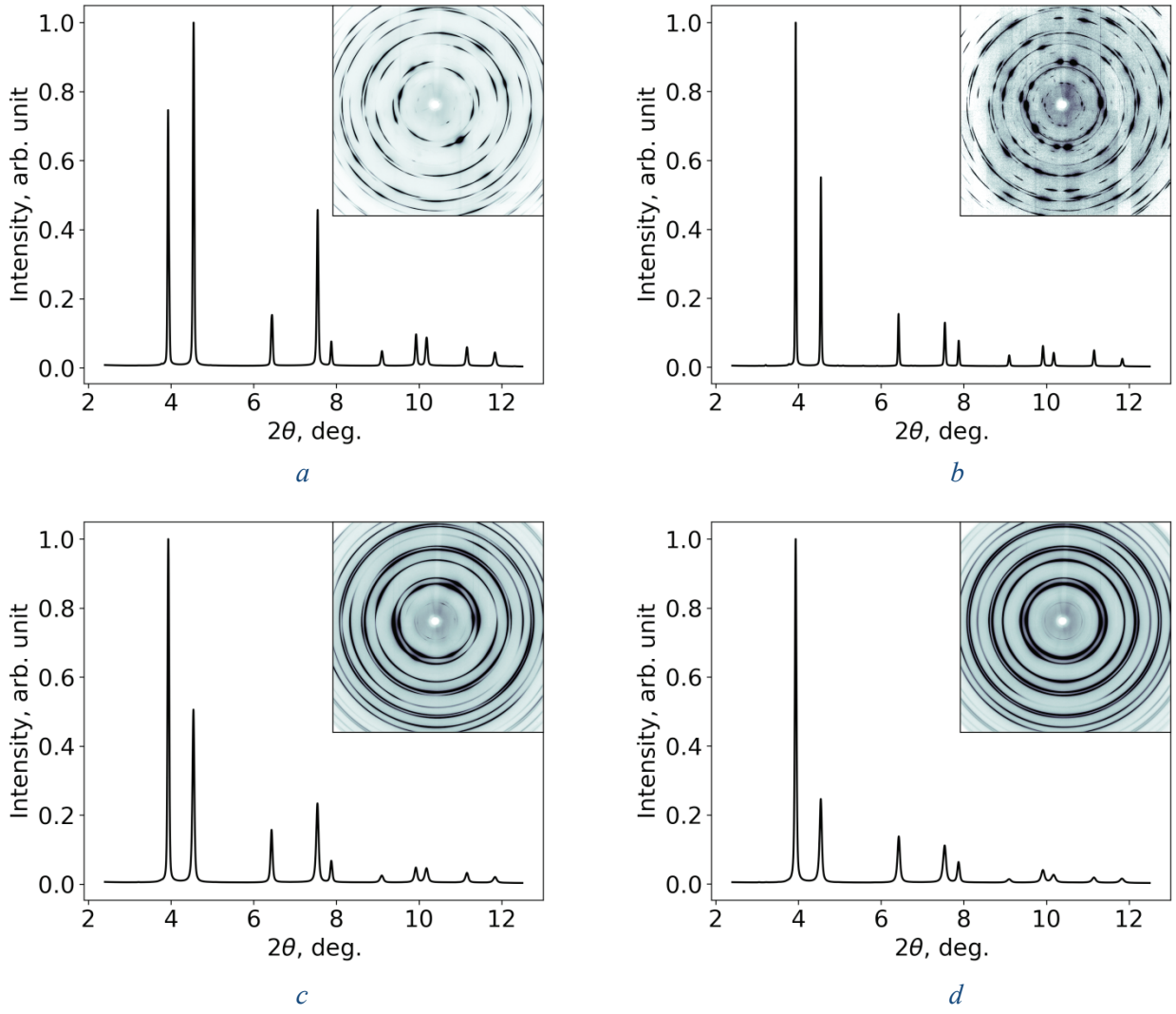


Fig. 1. X-ray diffraction patterns of the $Al_{0.3}CoCrFeNi$ high-entropy alloy in pre-deformed state (*a*); after annealing at 400 °C (*b*); after cold rolling with 40 % (*c*) and 80 % (*d*) reduction

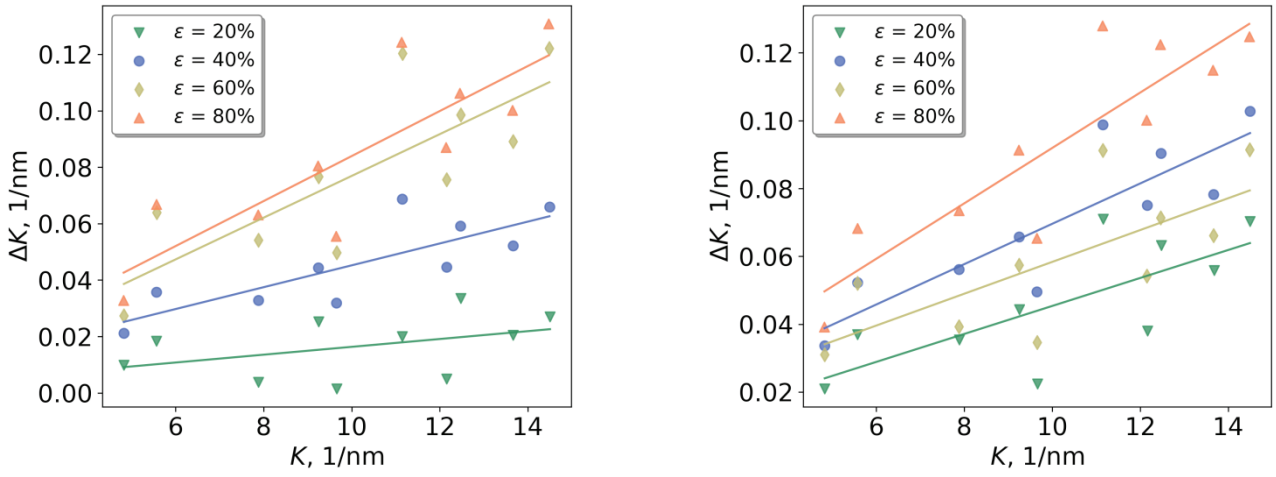
Table 1

Young's modulus of $Al_{0.3}$CoCrFeNi alloy in different directions

Direction	[111]	[200]	[220]	[311]	[222]	[400]	[311]	[420]	[422]	[333]
E_{hkl}, GPa	432	178	318	246	432	178	345	248	318	432

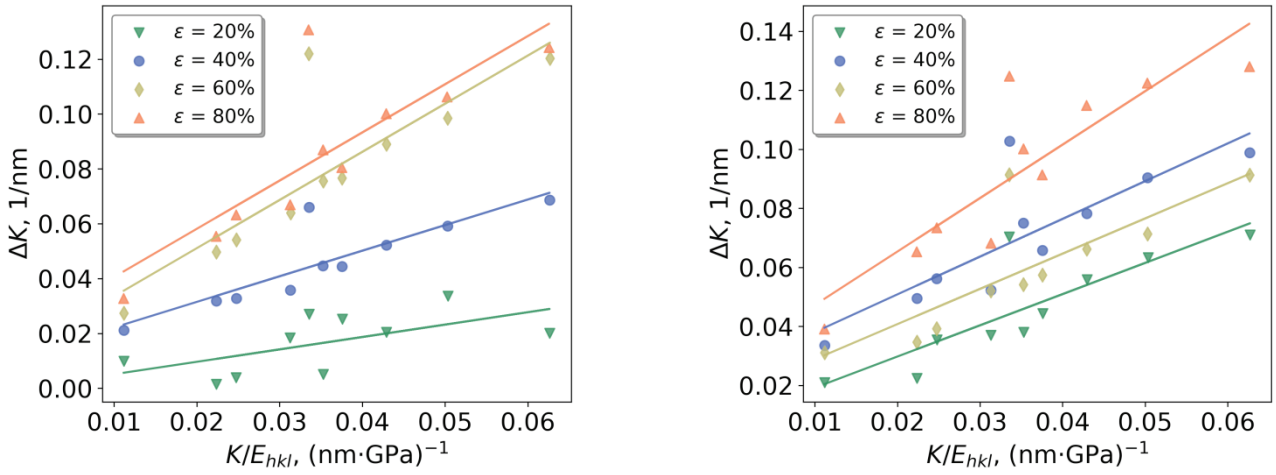
Ungar et al. [17, 18]. In particular, such models include the modified *Williamson-Hall* model used in this work (Equation 7). It is known that the structural defects are the reason of occurrence of stresses of the crystal lattice. The most common defects in the crystal structure are point defects, dislocations, stacking faults, twins, as well as grain and subgrain boundaries [19]. In addition to reducing the approximation error, the use of the modified *Williamson-Hall* method makes it possible to obtain additional information about the features of the defect structure of the crystal lattice. Thus, in the case of the analysis of polycrystalline materials with a cubic crystal lattice, it becomes possible to determine such microstructure parameters as the distribution of dislocations by type (screw/edge), as well as the probability to find stacking faults and twins. Experimental data and figures obtained using various models are presented in Figs. 2, 3 and 4.

From the presented figures it can be concluded that the entering of adjustments allows reducing the variance of values and bringing the trend closer to a linear one. This conclusion is confirmed by the analysis of



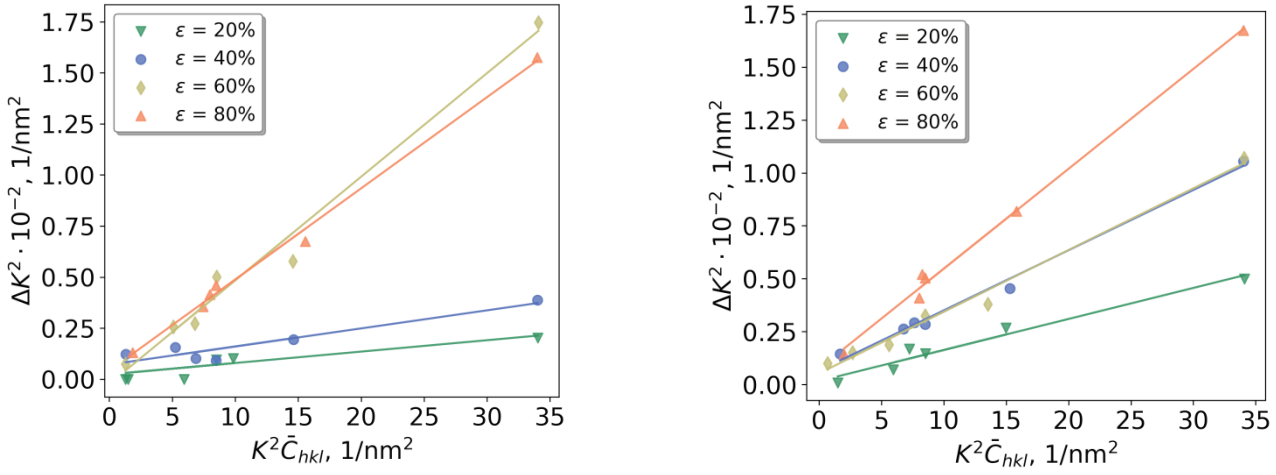
a *b*

Fig. 2. Williamson-Hall plots for $Al_{0.3}CoCrFeNi$ alloy after cold rolling for (*a*) *RD* and (*b*) *TD* directions



a *b*

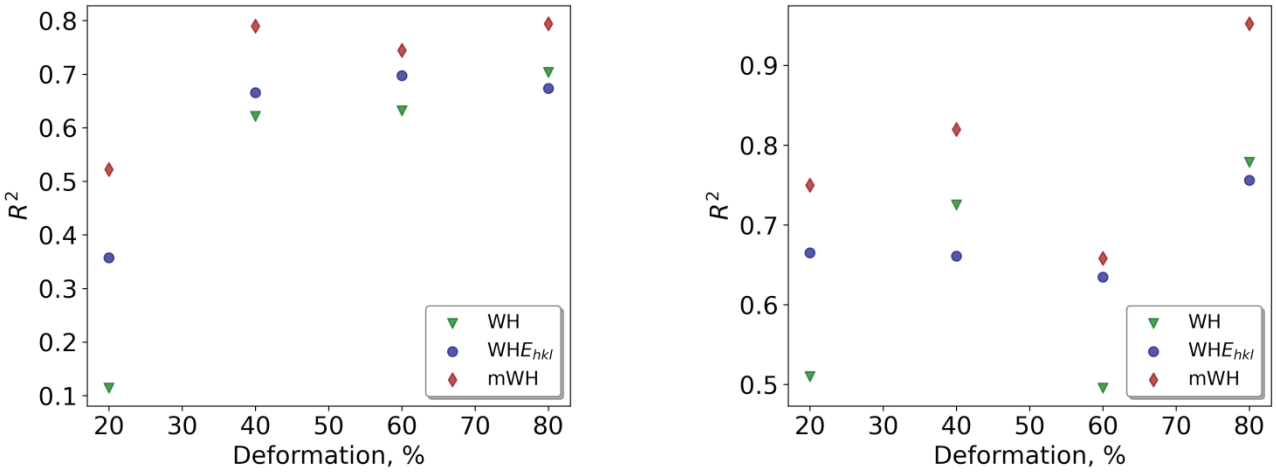
Fig. 3. Williamson-Hall corrected by elastic modulus plots for $Al_{0.3}CoCrFeNi$ alloy after cold rolling for (*a*) *RD* and (*b*) *TD* directions



a *b*

Fig. 4. Modified *Williamson-Hall* plots for $Al_{0.3}CoCrFeNi$ alloy after cold rolling for (*a*) *RD* and (*b*) *TD* directions

the values of the coefficient of determination (R^2). According to Fig. 5, the values of the coefficient R^2 in the case of the classical *Williamson-Hall* method can be lower than 0.5. This fact indicates that only half of the variance of the values ΔK is described by the model. The entering of adjustments significantly reduces the approximation error. The best result is observed for the modified *Williamson-Hall method*.



a *b*

Fig. 5. The coefficient of determination for various peak profile analysis methods used in this study: the classical *Williamson-Hall* (*WH*) method; classical method corrected for the modulus of elasticity (WHE_{hkl}); modified *Williamson-Hall* (*mWH*) method. The results for *RD* (*a*) and *TD* (*b*) directions are presented

A number of parameters of the modified *Williamson-Hall* model make it possible to evaluate the features of defects in the crystal structure of the materials. So, the dynamics of the parameter *q* (Equation 7) makes it possible to draw conclusions about the change of the relative fraction of edge/screw dislocations. Furthermore, the values of the parameter β (Equation 7) are directly related to the formation of stacking faults and twins in the material. An increase in the number of twins and stacking faults is indicated by an increase in the values of the parameter β. At the same time, the decrease in the parameter *q* values indicate a decrease in the relative fraction of edge dislocations.

The Fig. 6 shows the results of the analysis of the dislocation density and the parameter β depending on the degree of plastic deformation, as well as its relationship with the measured values of microhardness. It can be seen that plastic deformation leads to a significant increase of the number of twins and stacking

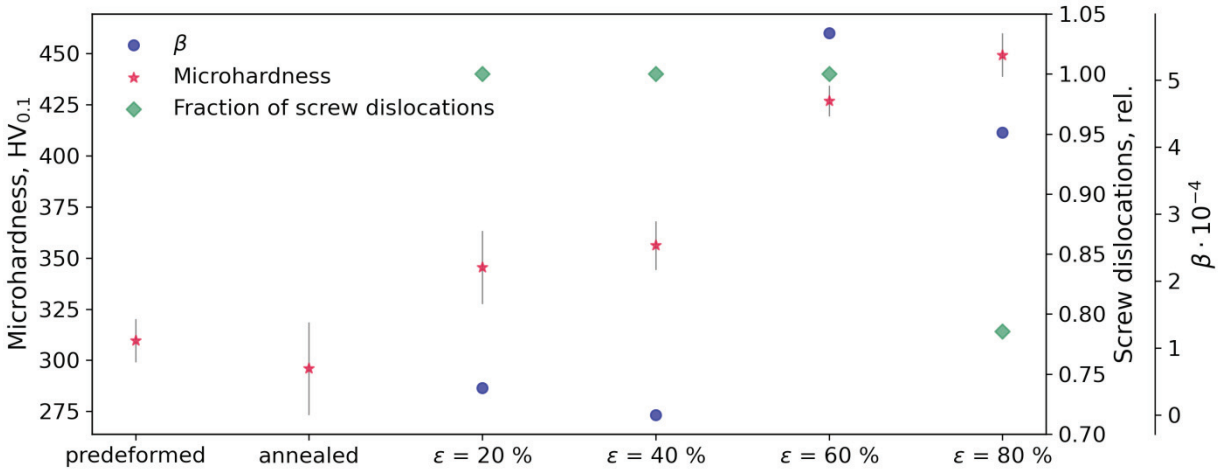


Fig. 6. Relative fraction of edge dislocations, parameter β and microhardness of the $Al_{0.3}CoCrFeNi$ alloy during cold rolling

faults. The values of the parameter β increase by more than an order of magnitude with an increase of the deformation degree up to 60 %. A further increase of the deformation degree leads to a slight decrease in the number of defects of this type. The effect of an increase of the number of twins and stacking faults during cold plastic deformation is known and well-studied for many high-entropy alloys with a *FCC* lattice [20, 21, 22]. The last stage (the stage of slight decrease) is apparently associated with the saturation of the structure with defects of this type. Furthermore, it can be seen that at the stage of increasing the deformation degree up to 60 %, the material is characterized mainly by the presence of screw dislocations. A further increase of the deformation degree leads to a decrease of the relative fraction of this type of defects. The effect of the dominance of screw dislocations at relatively low strains was demonstrated by *Schafler et al.* on the example of commercially pure copper deformed by equal channel angular pressing [23]. According to the obtained results, an increase of the deformation degree leads to a gradual decrease in the fraction of screw dislocations and an increase of the fraction of edge dislocations. A similar effect was also observed in the study of the dislocation structure of the aluminum alloy *Al-5.9Mg-0.3Sc-0.18Zr* with *FCC* crystal lattice [24]. This alloy was deformed by high-pressure torsion, and an increase of the number of revolutions from 0.5 to 5 led to a decrease in the proportion of screw dislocations in the system from 30 to 8 %.

The obtained results of microstructural studies correlate well with the values of microhardness. It can be seen (Fig. 6) that an increase of the deformation degree leads to a significant increase of the microhardness. It can be noted that the $Al_{0.3}CoCrFeNi$ alloy has a high capacity for work hardening.

Conclusions

1. In this study the possibilities of peak profile analysis methods for assessing defects in the crystal structure are shown by using the high-entropy alloy $Al_{0.3}CoCrFeNi$ as an example. Due to the presence of internal stresses associated with the nature of *HEA*, it is advisable to take into account the instrumental contribution using a preliminarily prepared annealed alloy of the same composition as the investigated samples.

2. The anisotropy of the elastic properties of the $Al_{0.3}CoCrFeNi$ alloy leads to an error of the approximation of the results by using the classical *Williamson-Hall* method. Entering of adjustments is an effective way to reduce the approximation error.

3. The smallest approximation error is typical for the modified *Williamson-Hall* method. The use of this method makes it possible to obtain the most reliable results concerning the defective structure of the $Al_{0.3}CoCrFeNi$ alloy.

Plastic deformation by cold rolling leads to an increase of the number of stacking faults and twins. Screw dislocations dominate in the structure of the alloy at a deformation degree up to 60 %, and an increase in the fraction of edge dislocations occurs only with an increase of the deformation degree up to 80 %. This dynamics of defects in the crystal structure is in good agreement with the data provided in the literature. The $Al_{0.3}CoCrFeNi$ alloy has a high tendency to work hardening.

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Conflicts of Interest

The authors declare no conflict of interest.