Deconvolution-based peak profile analysis methods for characterization of CoCrFeMnNi high-entropy alloy

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HIGHLIGHTS
- The structure of deformed CoCrFeMnNi alloy was studied using X-ray diffraction
- Several deconvolution-based peak profile analysis methods were compared
- The conventional Williamson-Hall method is not suitable for analysis of the structure of the alloy
- The correction for elastic properties significantly reduces the approximation error
- The modified Williamson-Hall and Warren-Averbach methods show the best results

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ABSTRACT
In this study, we compare several peak profile analysis methods for the investigation of the CoCrFeMnNi high-entropy alloy (HEA) after plastic deformation. We show that conventional Williamson-Hall (cWH) approach poorly correlates with measured peak broadening and some corrections must be introduced to improve the analysis. The correction for elastic properties for different crystallographic directions or application of modified Williamson Hall (mWH) and modified Warren-Averbach (mWA) methods significantly improve the correlation between the model and experimental data. Peak profile analysis shows that the dislocation density of the CoCrFeMnNi alloy subjected to axial compression increases with increase in strain and reaches a plateau at a strain of 47.5%. At the same time, the crystallite size decreases, and dislocation structure becomes more disordered.

1. Introduction
The greatest breakthrough of the past decade in multicomponent alloys technology is the development of high-entropy alloys (HEAs). Compared to all known alloys, HEAs possess the highest values of the entropy of mixing in the liquid state. Some of them are known to have a very simple structure of a single-phase solid solution consisting of five components. Such alloys belong to the so-called first generation of HEAs [1, 2]. Most HEAs crystallize in BCC or FCC lattices, however, recently HEAs with hexagonal symmetry were discovered by Yusenko et al. [3]. According to Gorban et al. [4], the lattice type of the HEAs depends on the valence electrons concentration (VEC). BCC lattice forms in alloys with the VEC ranging from 4.2 to 7.2 electrons/atom. When the VEC exceeds 7.5 electrons/atom, the HEA crystallizes in FCC lattice.

An important feature of HEAs is the intrinsically high value of crystal lattice distortion, which is a direct consequence of their multicomponent nature. Such high values of lattice distortion strongly affect the various properties of the alloys. For instance, Pogrebnyak et al. claim that the effect of high lattice distortion leads to the higher electrical and thermal conductivity of HEAs in comparison with classical alloys [5]. High mechanical properties of the HEAs are also associated with crystal lattice distortions. It is intuitively clear, that strength and ductility of HEAs should be related to their dislocation structure and to the interaction of moving dislocations with a strongly distorted lattice. At the same time,
the dislocation structure of HEAs is rarely discussed during the analysis of their mechanical properties.

One of the most common methods for characterization of material’s microstructure is an X-ray diffraction (XRD) peak profile analysis. The broadening of diffraction maxima is known to be a consequence of an increase in crystal lattice distortions and a decrease in the size of coherent scattering regions (CSR). The latter is also frequently referred to as crystallite size. The most common and generally accepted approaches for peak profile analysis are the Williamson – Hall and Warren - Averbach deconvolution methods. By using these methods, it is possible to estimate the average CSR size and crystal lattice distortions. These methods are best suited for the analysis of elastically isotropic materials. For example, in the original study of Williamson and Hall, the experiments were carried out with aluminum and tungsten, the elastic properties of which are practically isotropic [6]. However, for elastically anisotropic cubic materials a significant deviation of the experimentally measured peak profiles from those predicted by the conventional Williamson Hall, and Warren - Averbach models (cWH and cWA) is frequently observed [7]. There are two main approaches to reduce the error of the analysis: (i) the correction for the elastic modulus in different crystallographic directions; (ii) the use of the dislocation model of strain anisotropy. For instance, for severely deformed iron the correction for the elastic modulus significantly reduced the approximation error [8]. At the same time, this approach was not so effective for the analysis of polycrystalline hexagonal materials [9]. Therefore, the most expedient approach is to use the dislocation-based models of strain anisotropy of crystal lattice. These models were described by Ungar et al. [10, 11, 12] and are widely used to analyze the defects in polycrystalline materials with various crystal structures [7, 13, 14, 15]. In particular, Ungar et al. developed the so-called modified Williamson–Hall and modified Warren - Averbach (mWH and mWA) methods, which relate the profile of diffraction maxima to the features of the dislocation structure of materials.

Although mWH and mWA methods are rapidly spreading in the materials science community, there are currently a limited number of studies that have used them to analyze the dislocation structure of HEAs [16, 17, 18, 19, 20, 21, 22]. Besides, at the moment, there are no studies in the literature on analysis of the dislocation structure of the CoCrFeMnNi alloy during cold rolling at a low strain rate.

When studying HEAs by using deconvolution peak profile analysis methods, it is necessary to take into account the broadening caused by intrinsically high elastic distortions of the crystal lattice. Obviously, high lattice distortions initially exist even in undeformed HEAs, leading to a decrease in the intensity and additional broadening of the diffraction maxima. This effect can be deconvoluted using an undeformed sample of the same chemical composition. The same undeformed sample can also be used to take into consideration instrumental broadening.

In this work, using the high-entropy CoCrFeMnNi alloy (single-phase FCC alloy) as an example, we show how the diffraction maxima profiles of the HEA change during the plastic deformation. We also compare various deconvolutional methods of peak profile analysis and show that the conventional Williamson – Hall and Warren – Averbach methods are inferior to modified ones in studying the structure of HEAs. Based on the obtained data, we describe the change in the dislocation structure of the CoCrFeMnNi alloy with an increase in plastic strain.

2. Experimental procedures

CoCrFeMnNi HEA was chosen to test various peak profile analysis methods in this study. When properly prepared, this alloy has a simple fcc-based structure. Thus, there is no need to separate diffraction maxima from different phases, as well as to take into account the effects of lattice distortion due to stresses at interphase boundaries. The ingots of CoCrFeMnNi alloy were obtained from pure elements using a Büchler ArcMelter AM apparatus. Before the melting, the furnace chamber was firstly evacuated to a pressure of 2·10⁻⁸ bar and flushed with argon. A titanium ingot was used as a getter. Each HEA ingot was remelted 12 times and it was flipped after every second remelting to ensure its chemical homogeneity. The total weight of each ingot was ~30 g.

Cylindrical specimens with a diameter of 5 mm and a height of 8 mm were cut from the ingots using a Sodick AD 325 L wire discharge machine. The specimens were deformed by axial compression using an Instron 3369 universal testing machine. The experiments were performed at room temperature with the strain rate of ~8.4·10⁻³ s⁻¹. It is known that both the temperature and strain rate have significant effect on materials behaviour during the deformation [23]. We assume that the low strain rate, small sample size, and large anvil size led only to material strengthening due to the deformation, and thermal softening did not occur. A set of samples with the following engineering strain (%) was obtained: 0.00; 25.00; 37.50; 42.50; 47.50; 55.00. Subsequently, a few specimens were cut from the deformed materials for the XRD analysis and microhardness tests.

XRD experiments were carried out in a transmission mode at the beamline 5-Å (X-ray microscopy and tomography) at VEPP-4 synchrotron source (Budker’s Institute of Nuclear Physics, Novosibirsk, Russia). The X-ray wavelength was 0.0247 nm, which corresponds to the energy of photons 50.12 keV. A mar345s image plate 2D detector with pixel size 100×100 μm² and scanning area with diameter 345 mm was used to record the diffraction patterns. The samples-to-detector distance was 0.595 m. During the experiments, two-dimensional diffraction patterns were obtained. These two dimensional diffraction patterns were azimuthally integrated using pyFAI open-source software package [24]. Some of the analyzed diffraction patterns are shown in Figure 1.

The XRD patterns were fitted using whole-profile multiple pseudo-Voigt functions. The instrumental broadening, as well as the contribution of the intrinsic stresses of the CoCrFeMnNi lattice, were taken into consideration using the as-cast sample. The deconvolution of different contributions to the total peak profile was performed in two different ways. For approaches based on Williamson - Hall method the total width of diffraction maxima was defined as

\[
\beta_{\text{measured}} = \beta_{\text{structure}} + \beta_{\text{as-cast}},
\]

where \(\beta_{\text{measured}}\) is the experimentally observed FWHM before the deconvolution; \(\beta_{\text{structure}}\) is the FWHM due to the structure of the sample (crystallite size, lattice strain due to cold working, etc.) and \(\beta_{\text{as-cast}}\) is the FWHM of the reference sample (as-cast alloy). For the modified Warren - Averbach approach the correction to the real part of Fourier transform coefficients were added by using complex division \(A_{\text{measured}}(L)/A_{\text{as-cast}}(L)\), where \(A_{\text{measured}}(L)\) are coefficients before deconvolution; \(A_{\text{as-cast}}(L)\) are coefficients of the sample after deconvolution and \(A_{\text{as-cast}}(L)\) are coefficients of the reference sample. The positions of the diffraction maxima and their FWHMs (for all investigated samples) are summarized in Table 1.

The following methods of peak profile analysis were tested in this study for characterization of high-entropy alloys:

- **conventional** Williamson - Hall (cWH) method (Eq. (1));
- **WHE\(E_{\text{bulk}}\) method, which stands for Williamson - Hall method corrected for elastic modulus \(E_{\text{bulk}}\) for different crystallographic directions (Eq. (2));
- **combination of modified** Williamson - Hall (mWH) and modified Warren - Averbach (mWA) methods (Eqs. (3) and (4)).

In one common form, the cWH equation looks as follows [6]:

\[
\Delta K = \frac{0.9}{D} + 2\epsilon K.
\]

In Eq. (1) \(K = \frac{\lambda}{4\pi}\) is the reciprocal space coordinate; \(\Delta K = \frac{\omega^2}{2\lambda}\) is the inhomogeneous lattice strain; \(\lambda\) is the wavelength; \(D\) is the average size of CSR.

The correction for Young’s modulus can be done by introducing the Hook’s law \(\epsilon = \sigma/E_{\text{bulk}}\) in Eq. (1):
\[ \Delta K = \frac{0.9}{D} + \frac{\sigma}{E_{\text{Ml}}} \cdot K, \]  \hspace{1cm} (2)

where \( \sigma \) is the elastic stress assumed to be isotropic; \( E_{\text{Ml}} \) is Young’s modulus along the direction normal to the plane \( \langle hkl \rangle \).

Another deconvolution approach is based on the dislocation model of strain anisotropy. This approach forms the basis for the \( mWH \) and \( mWA \) methods. For cubic crystals \( mWH \) method is written as follows:

\[ \Delta K = \frac{1}{D} + \frac{\sigma}{E_{\text{Ml}}} \cdot \left( 1 + \frac{q}{C^{\text{Ml0}}(h^2 + k^2 + l^2)^(1/2)} \right), \]  \hspace{1cm} (3)

where \( \sigma = \left( \frac{\pi a^2}{D} \right)^2 \), \( \beta = \pi A^2 b^2 / \rho \), \( A \) is the parameter determined by dislocations outer cut-off radius; \( b \) is the absolute Burgers vector value; \( \rho \) is the average dislocation density; \( C^{\text{Ml0}} \) is the average dislocation contrast factor along the \( \langle hkl \rangle \) direction; \( q \) is the parameter dependent on the elastic properties of the material; \( H^2 = (h^2 + k^2 + l^2) / (h^2 + k^2 + l^2) \).

In a similar way, the \( mWA \) equation can be written as:

\[ \ln A(L) = \ln A(L) - \frac{\pi \rho}{2} \left[ L^2 \ln \left( \frac{R_e}{L} \right) \left( \frac{g^2 C^{\text{Ml0}}(h^2 + k^2 + l^2) (1 + q)}{L} \right) + O \cdot \left( \frac{g^2 C^{\text{Ml0}}(h^2 + k^2 + l^2) (1 + q)}{L} \right) \right], \]  \hspace{1cm} (4)

where \( g \) is the diffraction vector \( g = K \) in Bragg’s positions; \( L \) is the Fourier transform length, defined as \( L = \pi a_2 \) (where \( n \in Z \); \( a_2 = \lambda / (2 \sin \theta_1 - 2 \sin \theta_2) \) is the unit of Fourier transform along the diffraction vector \( g \); \( \theta_1 - \theta_2 \) is the angular range of diffraction profile; \( C^{\text{Ml}} \) is the average contrast factor along the \( \langle hkl \rangle \) direction; \( R_e \) is the dislocation outer cut-off radius; \( O \) is the higher-order term.

A detailed description of these models and examples of their practical applications can be found in the publications by Ungar et al. [10, 11, 12, 25] as well as in our previous study [9].

### 3. Results and discussion

From Table 1 it follows, that the FWHM of the diffraction peaks increases with an increasing strain. At the same time, the FWHM varies unevenly for different peaks. This is a consequence of the dislocation contrast: distortions of the crystal lattice around dislocations are inhomogeneous in different directions and affect the broadening of different maxima in a different way. This effect is enhanced by the anisotropy of the elastic properties of the samples. This significantly affects the results.

### Table 1. Parameters of the diffraction maxima obtained by curve fitting.

| \( \varepsilon \), % | \( 2\theta_{\text{max}} \) FWHM | \( 2\theta_{\text{max}} \) FWHM |
|---------------------|------------------|------------------|
|                    | (111)            | (200)            |
| 25.00              | 6.8260           | 7.8910           | 0.0297 |
| 37.50              | 6.8353           | 7.8999           | 0.0359 |
| 42.50              | 6.8302           | 7.8864           | 0.0435 |
| 47.50              | 6.8329           | 7.8932           | 0.0451 |
| 55.00              | 6.8302           | 7.8870           | 0.0479 |
|                    | (200)            | (311)            |
| 25.00              | 11.1700          | 13.1057          | 0.0347 |
| 37.50              | 11.1693          | 13.1199          | 0.0431 |
| 42.50              | 11.1691          | 13.1041          | 0.0492 |
| 47.50              | 11.1712          | 13.1110          | 0.0596 |
| 55.00              | 11.1632          | 13.1014          | 0.0588 |
|                    | (222)            | FWHM             |
| 25.00              | 13.6884          | 0.0261           |
| 37.50              | 13.6991          | 0.0285           |
| 42.50              | 13.6896          | 0.0355           |
| 47.50              | 13.6959          | 0.0422           |
| 55.00              | 13.6878          | 0.0413           |

Figure 1. Diffraction patterns of the CoCrFeMnNi alloy before (a) and after (b–d) plastic deformation by axial compression with different strains.
of conventional peak profile analysis methods. According to Figure 2a, d, the cWH method has a poor correlation with experimental data (the adjusted corrected coefficient of determination $R^2_{adj}$ was used as a criterion to estimate the correlation quality). The $R^2_{adj}$ values for the cWH method are in the range 0.1–0.5 for all samples, which indicates that this method is not suitable for the analysis of the structure of such materials.

As mentioned above, some corrections should be used to reduce the approximation error of peak profile analysis. One of the most well-known approaches is to introduce the correction for elastic properties of the crystal lattice (Eq. (2)). In this case, the resulting values of lattice strain ($\varepsilon$) are normalized to the value of the elastic modulus ($E_{hkl}$) for each diffraction maximum. Figure 2b, d shows the results of this method and the values of the coefficients $R^2_{adj}$. The values of the elastic moduli (Table 2) were obtained using the elastic constants calculated by Zaddach et al. [26] using the exact muffin-tin orbital method combined with coherent potential approximation (EMTO-CPA). The correction for elastic properties significantly reduces the approximation error, which is in good agreement with the results obtained for other materials with cubic symmetry [8].

Another effective method to reduce the approximation error is the use of an approach based on the dislocation theory of elastic distortions of the crystal lattice. In this case, it is necessary to calculate the contrast factor in the direction [h00] ($C_{h00}$) for the crystal structure, which can be done using the same elastic tensor which was used to calculate $E_{hkl}$. The values of the contrast factor with different anisotropy coefficient ($A_i$) are shown in Figure 3 (a). According to the obtained calculations, the average value of $C_{h00}$ for the CoCrFeMnNi alloy is 0.3355. The values of $C_{h00}$ are later used in $mWH$ method (Figure 2 c, d). The application of this method significantly reduces the approximation error. Even though the approximation error of the $mWH$ method is lower than that of (WH$E_{hkl}$) method, both approaches are effective for improving the quality of peak profile analysis for the HEA.

From the values of parameter $q$, which is used in Eq. (3), one can deduce the fraction of edge and screw dislocations in the samples (Figure 3 b). An increase in strain leads to an uneven increase in the density of edge and screw dislocations. It means that upon deformation of the CoCrFeMnNi alloy not only does the dislocation density increase but also other, more complex changes in the dislocation structure occur. By using the values of $q$ the values of $C_{hkl}$ were calculated. Using $C_{hkl}$ it is possible to apply the mWA method to the XRD patterns (Eq. (4)), and define the «size» ($A^s(L)$) and «distortion» ($A^d(L) = \frac{2}{\pi} \cdot \frac{\ln(\frac{E}{1})}{\rho}$) parts of Fourier-transform. The example of obtained results for the sample $\varepsilon = 55.00\%$ are presented in Figures 4 and 5 a and b.

More information on dislocation structure can be received from a combination of $mWH$ and $mWA$ methods: the absolute dislocation density, the distribution of dislocations by type, and the spatial distribution of dislocations. These results are shown in Figure 6. An increase in strain leads to a gradual increase in the dislocation density. However, at the strain $\varepsilon = 47.5\%$, dislocation density reaches a plateau, which indicates that the saturation stage has been reached. This result is in good agreement with the classical concepts of plastic deformation of metallic

![Figure 2](image)

**Figure 2.** Peak profile analysis of deformed CoCrFeMnNi samples using: (a) cWH; (b) WH$E_{hkl}$; (c) mWH methods. (d) The values of $R^2_{adj}$ which was used to compare the methods.

| $E_{hkl}$ (GPa) | (111) | (200) | (220) | (311) | (222) |
|-----------------|-------|-------|-------|-------|-------|
| 334.16          | 92.01 | 201.55| 139.70| 334.16|

Table 2. Values of $E_{hkl}$ in the directions normal to the diffraction planes that were used for the peak profile analysis.
materials, which predict that at later stages of deformation the nucleation and annihilation rates of dislocations become equal, and subsequently the dislocation density doesn’t increase [27]. The dynamics of the screw dislocations density differs from that of the total density of dislocations. Up to $\varepsilon = 42.5\%$, the screw dislocation density almost does not change and increases only in the range $42.5\% - 47.5\%$.

During the deformation, there are also significant changes in the CSR size ($x_{area}$) and the dislocations outer cut-off radius ($R_e$) (Figure 7a). The values of these parameters decrease up to $\varepsilon = 41.5\% - 47.5\%$ and practically do not change with subsequent increase in strain. At the same time, the constant growth of the Wilkens parameter ($M$) indicates disordering of the dislocation structure with an increase in strain (Figure 7b). According to classical concepts, the cell-like structure appears when plastic strain is high. The structure of such type possesses a so-called self-screening effect leading to a decrease in $R_e$ and $M$. However, in this case, it can be assumed that the strain was not sufficient for the formation of a cell structure.

From a practical point of view, the correlation of structural parameters deduced by peak profile analysis with the mechanical properties of the alloy is of great importance. In this study, the microhardness ($HV_{0.1}$) of the alloy was measured at various stages of deformation (Figure 8). It is typically believed, that $HV_{0.1}$ depends on dislocation density ($\rho$) and CSR size ($x_{area}$) according to Taylor equation ($\sigma_y = \alpha Gb/\sqrt{\rho}$, where $\sigma_y$ is material yield strength; $G$ is shear modulus $b$ is the magnitude of the Burgers vector; $\alpha$ is a geometric factor depending on the distribution of interacting dislocations and their type) and Kuhlmann-Wilsdorf equation ($\sigma_y = k_c l^{-2}$, where $k_c$ is the constant of the resistance of boundaries to penetration of dislocations; $l$ is cell diameter). In several works, it was shown that there is a strong correlation between structural parameters measured using XRD and the strength and hardness of the sample [8, 9, 28]. In the current study, the correlation between $HV_{0.1}$ and $\sqrt{\rho}$ is 0.87; between $HV_{0.1}$ and $1/x_{area}$ is
0.94. A significant increase in microhardness occurs with an increase of strain to 42.5%, however, with a further increase in strain, the values of microhardness almost do not change.

4. Conclusions

In this study we compared several peak profile analysis methods to study the structure of CoCrFeMnNi HEA. The following conclusions can be drawn as a result of the research:

1. An undeformed sample of the same composition as deformed ones can be used to take into account the lattice distortions inherent in the HEA.
2. The broadening of peak profiles predicted by cWH method for the CoCrFeMnNi HEA differs significantly from the experimental results, making cWH approach unsuitable for the analysis of CoCrFeMnNi-based alloys and possibly other FCC-based HEAs.
3. The correction for the elastic modulus in different crystallographic directions (WH_Ehkl method) can significantly reduce the approximation error of the peak profile analysis.
4. However, the most effective approach for analysis of CoCrFeMnNi alloy is mWH method. It provides the values of R_Ehkl above 0.8 and can be recommended to other researchers studying the structure of deformed HEAs.
5. Under axial compression of the CoCrFeMnNi alloy, the growth of the dislocation density occurs up to ε = 47.5%, while its average value is ~40 \cdot 10^{14} \text{ m}^{-2}. A further increase in strain does not change the total dislocation density. The dynamics of the CSR size (x_{area}), dislocations outer cut-off radius (R_e), and the Wilkens parameter (M) indicate the formation of a disordered dislocation structure.

Declarations

Author contribution statement

Ivan V. Ivanov: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.
Kemal I. Emurlaev: Performed the experiments; Contributed reagents, materials, analysis tools or data.
Konstantin E. Kuper, Sergey A. Akkuzin: Performed the experiments.
Ivan A. Bataev: Conceived and designed the experiments; Wrote the paper.

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Data availability statement

Data will be made available on request.


**Declaration of interests statement**

The authors declare no conflict of interest.

**Additional information**

No additional information is available for this paper.

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