Microstructural investigation of plastically deformed Ti$_{20}$Zr$_{20}$Hf$_{20}$Nb$_{20}$Ta$_{20}$ high entropy alloy by X-ray diffraction and transmission electron microscopy

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The microstructure evolution in body-centered cubic (bcc) Ti$_{20}$Zr$_{20}$Hf$_{20}$Nb$_{20}$Ta$_{20}$ high entropy alloy during quasi-static compression test was studied by X-ray line profile analysis (XLPA) and transmission electron microscopy (TEM). The average lattice constant and other important parameters of the microstructure such as the mean crystallite size, the dislocation density and the edge/screw character of dislocations were determined by XLPA. The elastic anisotropy factor required for XLPA procedure was determined by nanoindentation. XLPA shows that the crystallite size decreased while the dislocation density increased with strain during compression, and their values reached about 39 nm and $15 \times 10^{14}$ m$^{-2}$, respectively, at a plastic strain of ~20%. It was revealed that with increasing strain the dislocation character became more screw. This can be explained by the reduced mobility of screw dislocations compared to edge dislocations in bcc structures. These observations are in line with TEM investigations. The development of dislocation density during compression was related to the yield strength evolution.

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1. Introduction
Since the pioneering works by Yeh et al. [1] and Cantor et al. [2] high-entropy alloys (HEAs) have triggered a lot of expectation as potential new family of structural component materials [3,4]. Actually, HEAs refer to multi-elementary (disordered) solid solutions with at least 5 elements whose concentration is close to equimolarity. One of the proposed empirical parameters involved in HEA concept – and also in the formation of the multicomponent solid solutions – is the contribution of the mixing entropy term to the Gibbs free energy of mixing for the alloy system [5]. For HEAs the value of the mixing entropy is high and therefore sufficient to overcome the enthalpies of compound formation and phase separation. In such a case, solid solution is expected to be stabilized [6]. However, a recent work [7] has revealed that although the above concept can explain the single-phase state of some multi-element alloys [2], it cannot be used as a general rule for all HEAs. Indeed, many alloys meeting the proposed criteria, including HEAs, possess complex microstructures with multiple phases [8,9], since other quantities, such as mixing enthalpy or valence electron concentration, also influence the phase stability [10].

HEAs with simple solid solution structures, whatever face-centered cubic (fcc) or body-centered cubic centered (bcc) are reported to exhibit excellent properties such as high hardness and strength [1, 11–13], good resistance to softening at high temperatures [11,14–16], outstanding wear and fatigue properties [17], good corrosion resistance [18] and biocompatibility [19], making them promising materials for industrial and structural applications [13]. Among all the attractive new compositions, refractory HEAs have been intensively studied by many groups, particularly by Senkov and collaborators [14–16,20–23]. In particular, the equimolar bcc TiZrHfNbTa is a promising HEA for the applications performed in a wide temperature range from room temperature (RT) to approximately 600 °C [21]. The effect of the crystal structure, the chemical composition and the testing temperature on the mechanical behavior has been studied in different HEAs [13]. However, the effect of the lattice defects, such as dislocations, formed during plastic straining on the mechanical performance (e.g. the strength) has been not investigated in details. Recently and for the first time, detailed transmission electron microscopy (TEM) investigations have been carried out in order to comprehend the dislocation controlled mechanisms of plastic deformation in equimolar TiZrHfNbTa HEA alloy during compression at RT [24]. The operation of thermally activated deformation processes has been evidenced by the joint observation of low apparent activation volumes compatible with a Peierls mechanism and the glide of screw dislocation with the Burgers vector $b = a/2\langle 111 \rangle$ in the first stage of plastic deformation.

In the present work, the density, the arrangement and the edge/screw character of dislocations in an equimolar bcc TiZrHfNbTa...
HEA are monitored during compression at RT. According to the knowledge of the authors, this is the first study which gives a detailed characterization of the dislocation structure in a HEA material applying the combination of X-ray line profile analysis and post mortem TEM investigations. For a correct evaluation of X-ray diffraction peak broadening, the elastic anisotropy factor of the crystal is necessary. This paper also gives a method for the determination of this quantity in polycrystalline HEAs. The investigation of the relationship between the dislocation density and the yield strength yields the dislocation strengthening parameter in the present HEA material.

2. Experimental procedure

2.1. Materials and processing

A refractory HEA with equiatomic composition \( (\text{Ti}_{20}\text{Zr}_{20}\text{Hf}_{20}\text{Nb}_{20}\text{Ta}_{20}) \) was processed by arc melting and induction processes under argon atmosphere. Two master alloys, Nb–Ta and Ti–Zr–Hf, were first melted on a water-cooled copper plate from high purity metals (exceeding 99.9% purity) in the form of slugs and wires. Both alloys were remelted twice in order to improve homogeneity and then mixed together. A titanium getter was used prior each arc-melting fusion in order to capture the residual oxygen in the chamber. Homogenization of the alloy was then assessed by high frequency induction melting in sectorized cooled copper crucible under helium atmosphere. Finally, the homogenized refractory alloy was obtained by arc melting in the form of an ingot with 60 mm in length and approximately 10 mm in diameter.

2.2. Compression tests

Compression tests were carried out at room temperature at a strain rate of \( 1.5 \times 10^{-3} \text{ s}^{-1} \) on a cylindrical specimen with 5 mm in diameter and 7 mm in length using a 100 kN MTS testing machine (20/MH). In order to study the microstructure evolution during compression, samples were deformed up to different plastic strain values between 2 and 20%.

2.3. EBSD and TEM investigations

The grain structure of the initial sample was studied by electron backscatter diffraction (EBSD) investigations. The investigated surface was prepared by mechanical grinding using 1200 to 4000 grit SiC papers followed by a final polishing step using a 20 nm alumina oxide particle suspension (OPS) from StruersTM. EBSD investigations were carried out using a Zeiss Supra 40VP FEG scanning electron microscope (SEM). After mechanical testing, transmission electron microscopy (TEM) analyses were conducted on samples cut perpendicular to the load axis. Thin foils with 3 mm in diameter were prepared by mechanical polishing to 100 \( \mu \)m and finally thinned by the classical twin-jet electropolishing apparatus using a HF/H\(_2\)SO\(_4\) solution at \(-35^\circ\text{C}\). The TEM observations were carried out by a JEOL 2000EX operating at 200 kV.

2.4. Lattice parameter and microstructure from X-ray diffraction

The average lattice parameter for the initial and the deformed HEA samples was investigated by X-ray diffraction (XRD) using a Philips X’Pert θ–2θ powder diffractometer with CuK\(_\alpha\) radiation (wavelength: \( \lambda = 0.15418 \text{ nm} \)). XRD patterns revealed that all the studied samples have body-centered cubic (bcc) structure. The average lattice parameter was determined by extrapolating the lattice parameters obtained from the different reflections to the diffraction angle of \( 2\theta = 180^\circ \) using the Nelson–Riley method [25]. The microstructure in the specimens was studied by X-ray line profile analysis (XLPA). The X-ray line profiles were measured by a high-resolution rotating anode diffractometer (type: RA-MultiMax9, Manufacturer: Rigaku) using CuK\(_\alpha\) (wavelength, \( \lambda = 0.15406 \text{ nm} \)) radiation. Two-dimensional imaging plates detected the Debye–Scherer diffraction rings. The line profiles were determined as the intensity distribution perpendicular to the rings obtained by integrating the two dimensional intensity distribution along the rings. The diffraction patterns were evaluated by the Convolutional Multiple Whole Profile (CMWP) analysis [26,27]. In this method, the diffraction pattern is fitted by the sum of a background spline and the convolution of the instrumental pattern and the theoretical line profiles related to crystallite size and dislocations. The instrumental diffraction peaks were measured on a LaB\(_6\) standard material (SRM 660). The CMWP evaluation procedure yields the area-weighted mean crystallite size \( <\infty\text{area} >\), the dislocation density \( (\rho) \) and parameter \( q \) which describes the edge/screw character of dislocations.

3. Results and discussion

3.1. As-cast microstructure

A detailed characterization for the as-cast microstructure of this refractory alloy has been given elsewhere [28]. Fig. 1 is a typical inverse pole figure of the initial microstructure. There is no preferential crystallographic orientation of the grains (see the inset showing the standard stereographic triangle). The grain shape is irregular and the grain size distribution is broad, ranging between 10 and 350 \( \mu \)m. As it was revealed in a previous study [28], the size and morphology of grains as well as the chemical composition depend on the cooling rate during HEA processing. Indeed, it was shown that in the upper part of the ingot (lower cooling rate) the grains have a dendritic structure and the dendrite arms are mostly of Ta and Nb enriched while microsegregations consisting of Ti, Zr and Hf rich zones are found in the interdendritic zones.

3.2. Mechanical behavior

Typical engineering stress versus engineering strain and deduced true stress versus true strain compression curves for the as-processed HEA are shown in Fig. 2. In accordance with the work by Senkov et al. [21], the engineering stress versus engineering strain plot display strong linear hardening behavior. The yield strength is about 890 MPa. It should be noticed that when the true stress is plotted against true strain, the observed hardening is reduced.

![Fig. 1. EBSD inverse pole figure map showing the microstructure in the initial (undeformed) material.](image-url)
3.3. Microstructure of the compressed samples from X-ray line profile analysis

The average lattice constants determined for the initial and the deformed samples are listed in Table 1. All lattice constants are in the range between 0.3397 and 0.3414 nm. For the compressed samples the lattice parameter remains unchanged within the experimental error. The small difference between the lattice constants of the initial and compressed specimens can be explained by the slight variation of the chemical composition in the as-cast specimen, as revealed by coupled energy and wavelength dispersive spectrometry (EDS/WDS) in a previous paper [28].

The average crystallite size and dislocation density were determined by XLP A in samples compressed up to true plastic strains of 3, 10 and 20%. As an example, Fig. 3 shows the X-ray diffraction pattern for the sample compressed up to 20%. Reflection 222 was omitted from the pattern due to its weak intensity. The dependence of the peak breadths on the diffraction order (i.e. on the indices hkl) can be visualized by plotting the full width at half maximum (FWHM) as a function of the modulus of the diffraction vector, g (classical Williamson–Hall plot) [29]. FWHM and g can be calculated as:

\[
\text{FWHM} = \frac{\Delta(2\theta) \cos \theta}{\lambda} \text{ and } g = \frac{2 \sin \theta}{\lambda},
\]

where \( \theta \) is the Bragg angle of the peak, \( \Delta(2\theta) \) is the breadth of the line profiles in radians and \( \lambda \) is the wavelength of X-rays. The classical Williamson–Hall plot for the sample compressed up to 20% is shown in Fig. 4a. The non-monotonous variation of FWHM as a function of \( g \) for plastically deformed metallic materials is usually caused by the anisotropic strain field of dislocations, and this phenomenon is referred to as strain anisotropy [29]. The contrast effect of dislocations on line broadening can be taken into account by the average contrast (or orientation) factor of dislocations [29]. For a polycrystalline material containing dislocations the FWHM values fit to a smooth curve, when they are plotted as a function of \( g^2 \tau_{\text{hkl}} \), where \( \tau_{\text{hkl}} \) is the average contrast factor given as:

\[
\tau_{\text{hkl}} = \tau_{000} \left( 1 - q \frac{h^2 + k^2 + l^2}{h^2 + k^2 + l^2} \right).
\]

In Eq. (2) hkl are the indices of reflections and \( \tau_{000} \) is the contrast factor for reflection h00. Fig. 4b shows the modified Williamson–Hall plot for the sample compressed up to 20%. The datum points follow a smooth curve if \( q = 2.4 \) is selected. It is noted that the fitting of the measured diffraction pattern by the CMWP procedure also gives the value of \( q \) which was the same within the experimental error as the value obtained by the modified Williamson–Hall plot. The values of \( q \) for all deformed samples determined by CMWP fitting are listed in Table 1.

The comparison of the experimentally obtained \( q \) with the theoretical values calculated for pure edge and screw dislocations enables the determination of the edge/screw character of the dislocation structure. The theoretical values of \( q \) for pure edge and screw dislocations depend on the anisotropic elastic constants of the studied material. The dependence of parameter \( q \) on the elastic constants of bcc polycrystals can be given using the ratio of the elastic constant \( c_{12}/c_{44} \) and the elastic anisotropy factor \( A = \frac{c_{44} - c_{i j}}{c_{44}} \) [4]. Fig. 5 shows parameter \( q \) for edge and screw dislocations in bcc crystals as a function of \( A \) for three different usual values of \( c_{12}/c_{44} \) (0.5, 1 and 2). The data were taken from Ref. [29]. As the single crystal anisotropic elastic constants are not known for the present HEA composition, therefore the exact value of parameter \( q \) for pure edge and screw dislocations cannot be determined. In addition, in the CMWP evaluation of the diffraction patterns, one fitting parameter is \( \tau_{000} b^2 \rho \), where \( b \) is the modulus of the Burgers vector of dislocations. Assuming the usual Burgers vector in bcc structure ([100][111]), \( b \) equals 0.2944 nm in the present HEA material. Since \( \tau_{000} \) depends on the anisotropic elastic constants, without the knowledge of their values the dislocation density cannot be determined.

For a polycrystalline material the single crystal elastic anisotropy factor can be determined by depth-sensing indentation technique, as shown by Vlassak and Nix [30]. Then, this factor may be used for the estimation of the edge/screw character of dislocations from the experimental value of parameter \( q \) and for the determination of the dislocation

Table 1

<table>
<thead>
<tr>
<th>Sample</th>
<th>Lattice constant [nm]</th>
<th>Area-weighted mean crystallite size [nm]</th>
<th>( \rho ) [10^{14} \text{ m}^{-2}]</th>
<th>( q )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial</td>
<td>0.3414 ± 0.0005</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>Compressed, 3%</td>
<td>0.3401 ± 0.0002</td>
<td>151 ± 16</td>
<td>2.0 ± 0.5</td>
<td>1.0 ± 0.1</td>
</tr>
<tr>
<td>Compressed, 10%</td>
<td>0.3399 ± 0.0002</td>
<td>123 ± 14</td>
<td>10 ± 2</td>
<td>2.1 ± 0.1</td>
</tr>
<tr>
<td>Compressed, 20%</td>
<td>0.3397 ± 0.0003</td>
<td>39 ± 5</td>
<td>15 ± 2</td>
<td>2.4 ± 0.1</td>
</tr>
</tbody>
</table>
density from CMWP fitting. The analysis of a load–penetration depth curve obtained by depth-sensing indentation provides the indentation modulus defined as:

\[ M = \frac{E}{1-\nu^2} . \]

where \( E \) and \( \nu \) are the average Young's modulus and Poisson's ratio. If the size of the indentation is much smaller than the grain size, the value of \( M \) depends on the orientation of the indented grain due to the elastic anisotropy of the crystal. Vlassak and Nix [30] have shown that the indentation modulus \( M_{hkl} \) measured on the \((hkl)\) surface by a Berkovich indenter can be expressed as the product of the isotropic indentation modulus, \( M_{iso} \), a correction factor \( \beta_{hkl} \) due to the elastic anisotropy, and another factor \( \alpha_{hkl} \) owing to the anisotropic (triangular) shape of the Berkovich punch:

\[ M_{hkl} = \alpha_{hkl} \beta_{hkl} M_{iso} , \]

where \( M_{iso} \) is the indentation modulus measured on a randomly oriented polycrystalline aggregate consisting of crystals which are much smaller than the indent (i.e. this is the average indentation modulus). The dependence of factor \( \alpha_{hkl} \) on the orientation of the triangular indent on the surfaces \((001)\), \((101)\) and \((111)\) is plotted in Fig. 8 of Ref. [30]. For the surface \((001)\) this factor equals 1.058 independently of the indent orientation. For surfaces \((101)\) and \((111)\) the value of factor \( \alpha_{hkl} \) varies with the angle between one of the sides of the indenter and directions \([10\bar{1}]\) and \([1\bar{1}2]\), respectively. In the present experiments \( \alpha_{101} \) and \( \alpha_{111} \) are 1.057 and 1.051, respectively.

For grains with cubic crystal structure the correction factor \( \beta_{hkl} \) is a function of the Poisson's ratio in the cube directions \((\nu_{100})\) and the anisotropy factor \( (A) \) [30]. \( \beta_{hkl} \) can be given by the following formula [30]:

\[ \beta_{hkl} = a + c(A-A_0)B , \]

where \( a, c, A_0 \) and \( B \) depend on \( \nu_{100} \). The parameters in Eq. (5) for \( \beta_{101} \) and \( \beta_{111} \) are listed in Table 1 of Ref. [30] for six different values of \( \nu_{100} \), namely for 0.2, 0.25, 0.3, 0.35, 0.4 and 0.45. Due to the very large average grain size of the present HEA material (several hundreds of microns as shown by the EBSD images in Figs. 1 and 6), \( M_{iso} \) cannot be determined. However, \( M_{iso} \) can be eliminated by expressing the ratios of \( M_{001}, M_{101} \) and \( M_{111} \) using Eq. (4):

\[ \frac{M_{001}}{M_{101}} = \frac{\alpha_{001} \beta_{001}}{\alpha_{101} \beta_{101}} \quad \text{and} \quad \frac{M_{001}}{M_{111}} = \frac{\alpha_{001} \beta_{001}}{\alpha_{111} \beta_{111}} . \]

The left sides in the formulas of Eq. (6) can be determined experimentally from the indentation moduli measured on surfaces \((001)\), \((101)\) and \((111)\). The right sides can be calculated as a function of \( A \) and \( \nu_{100} \) (see Eq. (5)), therefore, if the ratio of the experimentally determined indentation moduli and the theoretically calculated ratio of the correction factors are made equal, the values of \( A \) and \( \nu_{100} \) can be obtained.

In the present experiment the indentation moduli were determined on the area shown in Fig. 6 using a UMIS nanoindentation device with a Berkovich indenter and applying a maximum load of 100 mN. The
The following values were obtained for the indentation moduli: $M_{001} = 88$ GPa, $M_{101} = 97$ and $M_{111} = 99$ GPa. Figs. 7 and 8 show the calculated ratio of the correction factors ($\alpha_{hkil}$/$\beta_{hkil}$) for the surface pairs 001–101 and 001–111 as a function of the anisotropy factor $A$, respectively, for six different values of $\nu_{100}$ between 0.2 and 0.45. The intersection of each curve with the experimental ratio of the indentation moduli gives a possible value of $A$ for a given value of $\nu_{100}$. Then, for 001–101 and 001–111 surface pairs the functions of the anisotropy factor $A$ versus the Poisson ratio $\nu_{100}$ are plotted in Fig. 9. From the coincidence of the two curves for 001–101 and 001–111, the values of 2.3 ± 0.2 and 0.25 ± 0.05 are obtained for $A$ and $\nu_{100}$, respectively. This anisotropy factor was used for the estimation of the theoretical values of parameter $q$ for pure screw and edge dislocations. According to the vertical dashed line in Fig. 5 the values of $q$ for pure screw and edge dislocations are 2.65 ± 0.05 and 1.10 ± 0.50, respectively.

The comparison of the theoretically calculated and the experimentally obtained $q$ values (see Table 1) suggests that with increasing strain the dislocation character became more screw. This can be explained by the reduced mobility of screw dislocations compared to edge dislocations in bcc structures. This difficulty in motion of screw dislocations is due to the core properties of screw dislocations, dissociated into a non-planar configuration while edge dislocations are split into partials only in their glide planes [31]. As a consequence, during plastic deformation edge dislocation segments can annihilate more easily than screw ones thereby the remaining dislocations have more screw character. It should be noted that the value of $C_{100}$ also depends on the anisotropy factor, as shown in [29]. For $A = 2.3$ ± 0.2 and $0.5 < c_{12}/c_{44} < 2$, the values of $C_{100}$ are $0.305 ± 0.015$ and $0.260 ± 0.045$ in the cases of screw and edge dislocations, respectively [29]. Since $C_{100}$ only slightly depends on the edge/screw character of dislocations, the average of the values obtained for pure screw and edge cases (0.28) was used in the CMWP fitting procedure for the determination of the crystallite size and the dislocation density.

The area-weighted mean crystallite size and the dislocation density determined from the diffraction peak profile analysis for the compressed samples are listed in Table 1. For the initial material the diffraction peaks were as narrow as the instrumental profiles ($\Delta(2\theta) = 0.02^\circ$), therefore these lines were not evaluated for the microstructure. It is noted that the peak breadths obtained for the initial (undeformed) sample reflect the diffraction line broadening caused by the distortion of the lattice due to different-sized atoms in the present HEA. Therefore, with the application of the instrumental correction this distortion effect was eliminated during the evaluation of line profiles. In the sample compressed plastically at 3%, the crystallite size and the dislocation density were 151 nm and $2.0 \times 10^{14}$ m$^{-2}$, respectively. It is noted that in plastically deformed metallic materials the crystallite size obtained by X-ray line profile analysis is usually smaller than the grain size determined by electron microscopy. This difference can be explained by the fact the crystallite is equivalent to the volume scattering X-rays coherently and dislocation patterns inside the grains may break the coherency of X-rays. The present investigation shows that the crystallite size decreased while the dislocation density increased with increasing strain during compression of the present HEA material, and their values reached about 123 nm and $10 \times 10^{14}$ m$^{-2}$, respectively, at the plastic strain of 10%. After compression up to plastic strain of 20% the crystallite size was reduced to 39 nm, while the dislocation density increased to $15 \times 10^{14}$ m$^{-2}$. Similar high dislocation density in conventional alloys can be obtained only by severe plastic deformation at an equivalent strain of about 100% [32].

### 3.4. TEM investigation of the deformation substructure

Fig. 10 shows TEM micrographs for samples deformed at different plastic strains of 1% (Fig. 10a), 3% (Fig. 10b) and 10% (Fig. 10c). It can be seen that for small strains the deformation localizes within bands that delineate defect free domains inside grains. Both the number of bands and the dislocation density inside the bands seem to increase with increasing plastic strain. The Burgers vector and the edge/screw character of dislocations within the bands have been studied in details.
by TEM [24]. Careful inspection of the TEM images using the extinction rule for dislocations showed that the Burgers vector is of \(a/2\langle 111\rangle\) type and the dislocations at higher strains are mainly of screw character, which is in line with XLPA results described above. Although TEM investigates much smaller volume than that in XLPA, the images reflect relatively well the order of magnitude of the average dislocation density determined by the latter method. For instance, the average dislocation spacing at a strain of 10% is 32 nm determined as the inverse square-root of the dislocation density obtained by XLPA. This value is in accordance with the visual observation of the dislocation spacing in Fig. 10c. The comparison of the TEM images obtained at different strains (see Fig. 10) reveals a high rate of the increase of dislocation density during plastic deformation which is also in accordance with the results of XLPA. This observation can be explained by the difficult annihilation of dislocations due to the high stress required for dislocation motion in HEA materials. The magnitude of attractive force between dislocations with opposite signs in HEAs is not very different from that in other bcc metals since the elastic constants and Burgers vector magnitude are similar. At the same time, the stress required for dislocation motion (i.e. the Peierls stress) is expected to reach much higher value in HEAs than in other bcc metals, therefore dislocations with opposite signs can exist closer to each other without annihilation. Due to the hindered annihilation, the dislocation density will be larger for a given strain.

3.5. Correlation between the yield strength and the dislocation density

The yield strength of the HEA samples compressed up to the strains of 3, 10 and 20% are 980, 1035 and 1050 MPa, respectively. The yield strength (\(\sigma_Y\)) of plastically deformed metallic materials is usually related to the dislocation density using the Taylor equation [33]:

\[
\sigma_Y = \sigma_0 + \alpha M^T G b \sqrt{\rho},
\]

where \(\sigma_0\) is the friction stress, \(\alpha\) is a constant describing the dislocation hardening, \(G\) is the shear modulus, \(b\) is the modulus of the Burgers vector (0.2944 nm), \(M^T\) is the Taylor factor. The value of \(M^T\) in bcc crystals varies between 2.75 and 3.06, depending on the type of slip systems populated by dislocations [34]. If the microstructure is texture-free and the glide occurs only in the most common slip system \((a/2\langle 111\rangle\langle 110\rangle)\), the Taylor factor is 3.06. In the present study, this value was selected for \(M^T\). From the compression stress–strain curves 890 MPa was obtained for \(\sigma_0\). The average shear modulus, \(G\), was determined from the average Young’s modulus \((E = 87 \text{ GPa})\) obtained from the indentation measurements and the Poisson's ratio \((\nu = 0.25)\) as \(G = \frac{E}{2(1+\nu)} = 35 \text{ GPa}\). Substituting these values into Eq. (7), the dislocation hardening parameter \(\alpha\) was found to be 0.16 ± 0.04 for the three compressed samples. This observation is in a reasonable agreement with the experimental results obtained previously for other, non-HEA bcc metallic materials such as different steels, Nb and Ta. For these materials \(\alpha\) varies between 0.17 and 0.60 [35–38]. The relatively low value of \(\alpha\) for the present HEA material is in line with the low strain hardening observed on the true stress – true strain curve. It seems that the main component in the compression flow stress is the Peierls stress and dislocation hardening has a lower contribution.

4. Conclusions

Equimolar Ti_{20}Zr_{20}Hf_{50}Nb_{20}Ta_{20} high entropy alloy with bcc structure was deformed by compression and the microstructure evolution as a function of strain was investigated by XLPA and TEM. The following conclusions were obtained:

1. In the initial sample the grain size distribution was broad, ranging between 10 and 350 \(\mu\)m. A high density of dislocations was developed during compression even at moderate plastic strains. At the highest applied strain (20%) the dislocation density was \(15 \times 10^{14} \text{ m}^{-2}\) as determined by XLPA. The dislocation density obtained by XLPA was in accordance with the dislocation spacing observed in the TEM images.

2. TEM images revealed that for small strains deformation bands were formed with high dislocation density and between the bands defect free domains were observed. Both the number of bands and the dislocation density inside the bands seem to increase with increasing plastic strain.

3. The single crystal elastic anisotropy factor was determined as 2.3 ± 0.2 by indentation technique which was required for the determination of the edge/screw character and the density of dislocations by XLPA. Using the elastic anisotropy factor, the theoretical expressions of the parameter describing the dislocation character were determined for pure edge and screw dislocations. Comparing these values with the experimentally determined parameters, it was found that the
screw character of dislocations became stronger with increasing strain. This observation was supported by TEM analysis.

4. The present HEA material has a high yield strength of about 890 MPa. The strain hardening during compression was caused by the increase of the dislocation density. The dislocation hardening parameter ($\alpha$) has a relatively low value but it is still in the range determined for other bcc non-HEA metallic materials. The Peierls stress has the main component in the flow stress and dislocation hardening has a lower contribution even for high dislocation densities.

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