Characterization of the Microstructure, Local Macro-Texture and Residual Stress Field of Commercially Pure Titanium Grade 2 Prepared by CONFORM ECAP

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Abstract: The paper investigated the residual strain and stress distribution, microstructure, and macro-texture along the transverse direction of commercially pure titanium grade 2 samples prepared by the CONFORM ECAP technique. This method belongs to the severe plastic deformation methods; hence, it could be assumed that residual stress fields would be present in the work-pieces. Residual stresses cannot be directly measured; thus, neutron diffraction measurements, Electron back-scatter diffraction (EBSD) investigations, and local X-ray macro-texture measurements were performed in different regions of the sample to determine the data for the residual stress calculation. The calculation was based on the modified Kröner model. Neutron diffraction strain scans and residual stress calculations revealed that symmetrical residual strain and stress gradients with compression character were present in the axial and hoop direction after one and two passes. Asymmetric distribution of the residual strains and stresses remained after the third pass of the CONFORM ECAP. EBSD investigations showed that after the first pass, significant grain refinement occurred; however, further passes did not cause any dramatic grain refinement. X-ray texture measurements revealed that local macro-texture was dependent on the number of passes of the CONFORM ECAP and on the investigated area in the samples.

Keywords: titanium; CONFORM; ECAP; residual; strain; stress; EBSD; texture

1. Introduction

Titanium and its alloys have a wide range of applications due to possessing high strength, low density, excellent corrosion resistivity, and nontoxicity [1]. For many years, the materials have been playing an important role in medicine, where a large number of implants are titanium-based. In biomedical application, the desired mechanical properties are usually achieved through alloying. However, some of the alloying elements are believed to be toxic to the human body, which limits the elements usage as biocompatible materials. Thus, the scientific community is constantly seeking...
for new approaches, which can lead to the enhancement of the mechanical properties without harmful additives.

One of the possible approaches is the processing of the material using severe plastic deformation (SPD). It has been shown that SPD processes can significantly improve the mechanical properties of materials through grain refinement and the introduction of many lattice defects [2]. The equal-channel angular pressing (ECAP) method, where the billet is pressed through two intersecting channels, belongs to the most popular method. The main advantage of this method is that the processed sample retains the initial cross-section in contrast to other industrial processing methods, for example, rolling or extrusion. In the last two decades, many studies have appeared on the topic of ECAP processed titanium alloys. All investigations agreed that the ECAP method led to an ultra-fine grained (UFG) microstructure and enhanced strength [3–8].

Despite the many benefits of the ECAP method, it cannot be used on an industrial scale because of the non-continuous manner of the process. This problem was overcome by the introduction of the CONFORM ECAP (C-ECAP) method [9], where the work-piece was driven forward in a curved die by the friction forces until it reached the abutment and was forced to turn an angle by shear as in a regular ECAP process. A comparative study by Medvedev et al. between conventional the ECAP and C-ECAP methods reported that after the application of C-ECAP, commercially pure (CP) titanium (Ti) grade 4 exhibited finer grains and a higher yield and ultimate tensile strength than after the application of the conventional ECAP [10]. Nevertheless, it should be noted that the same study reported heavily distorted grains and structure after the C-ECAP, whilst there were almost defect-free grains after the conventional ECAP. This fact gives rise to the supposition that the C-ECAP treatment increases the probability of the existence of a higher residual stress field and an inhomogeneous strain distribution in the processed material [11,12].

The residual stresses in many cases can negatively influence the quality and behavior of engineering components. In some extreme situations, the existence of the stresses may lead to significant distortion or deformation of the parts or in the worst cases, even to fracture. Thus, understanding the stresses origin may allow the lengthening of the components service life. The residual stresses can be successfully studied using diffraction-based methods [13,14]. Neutron and hard X-ray diffraction measurements are preferentially used for this purpose, since they allow the studying of bulk samples, due to the large penetration depth of neutrons and the high-energy X-ray photons in the majority of the materials [14,15]. However, only a few studies of this kind were performed on materials processed by the ECAP or C-ECAP methods [16], and limited data are available for the ECAP processed Ti.

The objective of the present paper was the investigation of the residual strain using neutron diffraction in CP Ti grade 2 samples processed by the C-ECAP method. Electron back-scatter diffraction (EBSD) studies and local X-ray texture measurements were also performed to elucidate the microscopic background responsible for the stress field. The influence of the number of C-ECAP passes on the residual strain field distribution was also discussed in detail.

2. Materials and Methods

CP Ti grade 2 was studied in this work. The chemical composition of the samples can be found in Table 1.

| Element | Fe  | O    | C    | H    | N    | Ti    |
|---------|-----|------|------|------|------|-------|
| Content | 0.046| 0.12 | 0.023| 0.0026| 0.0076| Rest  |

Work-pieces in the form of 2 m long cylindrical rods with a diameter of 10 mm were subjected to the C-ECAP process as described in reference [9] using COMTES FHT a.s. company’s specially designed machine, as in reference [17]. Specimens passed 1×, 2×, and 3× through the C-ECAP
machine on a route denoted as “A” during the multiple C-ECAP processes described in reference [18]. Then, 80 mm long samples were cut from the rods after the mechanical treatments.

2.1. Neutron Diffraction

Neutron diffraction (ND) measurements were carried out on the samples after the C-ECAP treatment at the Nuclear Research Institute, Řež, Czech Republic, on the HK4 beamline. The beamline HK4 is equipped with a strain scanner SPN-100 diffractometer supported by a 2D position sensitive detector (PSD) and a Si (111) monochromator. The complete description of the instrument can be found in References [19,20].

The diffracted neutron wavelength was 2.13 Å and square shaped slits with 3 mm side length were used. The center of the 2D PSD was set to a fixed value $2\theta_D = 55^\circ$ during the experiments. This $2\theta_D$ angle allowed the observation of reflections $(10\overline{1}1)$ and $(0002)$, and the nominal gauge volume (NGV) was $\sim 32.96 \text{ mm}^3$.

Samples were placed on the xyz stage, which allowed both the motion in three perpendicular directions, i.e., x, y, and z, as well as the rotation by an arbitrary angle.

Samples were scanned using ND, where the sampled gauge volume was moved in the coordinate system of the sample. The scanning was performed along a straight line by 1 mm shifts, which intersected the center of the circular cross-section. This line lay in the transverse direction in the conventional ECAP notation. Three scans were performed along the same line on each sample:

1. Diffraction vector $\vec{q}_R$ was parallel with the radial direction of the sample;
2. Diffraction vector $\vec{q}_H$ was parallel with the hoop direction;
3. Diffraction vector $\vec{q}_A$ was parallel with the axial direction.

The scheme of the scanning is drawn in Figure 1.

![Figure 1. Scheme of the ND scans.](image-url)
The differential form of Bragg’s Equation (1) was used for calculation of the lattice strain $\varepsilon_{hkl}$ of the particular crystallographic plains as follows.

$$\varepsilon_{hkl} = \frac{(d_{hkl} - d_{hkl}^0)}{d_{hkl}^0} = - \cot^2 \theta_{hkl}^B \left( \theta_{hkl}^B - \theta_{hkl}^B \right)$$  (1)

$d_{hkl}^0$ and $\theta_{hkl}^B$ are the interplanar distance of the $hkl$ planes of the stress-free reference and the corresponding Bragg angle. Furthermore, $d_{hkl}$ and $\theta_{hkl}^B$ are the interplanar distance of the $hkl$ planes under load and the corresponding Bragg angle.

The ND scanning started at a distance of $-3$ mm and ended at distance of $+3$ mm from the center of the samples. Omission of the measurement points at the edges of the specimen (positions $\pm4$ mm) was given by the fact that at these points the NGV was not completely filled with material, which resulted in anomalous $\theta_{hkl}^B$ shifts, and consequently in spurious strains [21].

2.2. EBSD

Specimens for the microstructural analysis with the EBSD technique were cut from the samples used at the ND measurements. Samples were prepared by standard metallographic grinding on SiC papers (from 320 to 1200 grit), followed by three-step vibratory polishing for 24 h (Alumina 0.3 $\mu$m, Alumina 0.05 $\mu$m, and Colloidal Silica 0.04 $\mu$m—each for eight hours). Finally, the specimen surfaces were ion-beam polished on a Leica EM RES102 system (Leica Mikrosysteme, Wetzlar, Germany). The investigated surfaces were perpendicular to the axial direction.

Microstructure analysis based on the EBSD technique was performed using a scanning electron microscope FEI Quanta 200 FX (Thermo Fisher Scientific, Brno, Czech Republic). Working distance was 13 mm with accelerating voltage 15 kV. The scan was conducted with a 0.1 $\mu$m step size.

The scanned areas (left and right periphery, and the central part) were chosen with the aim to characterize the microstructure in the ND irradiated sample volume.

2.3. X-ray Texture

The X-ray PANalytical XPert MRD (PANanalytical, Almelo, The Netherlands) diffractometer using CuK$\alpha$ radiation with polycapillary optics in the primary beam was employed for acquiring the $(0002)$, $(1010)$, and $(1011)$ pole figures. The orientation distribution functions (ODF) and the full pole figures were determined using MTEX 4.4 Free and open source software toolbox for MatLab (MathWorks, Natick, MA, USA) [22].

2.4. Calculation of the Residual Stresses

The calculation of the residual stresses in the three perpendicular directions (axial, hoop, and radial) was based on the results acquired by the EBSD, X-ray texture, and ND measurements. The calculation was done using the software IsoDEC version from 9/21/2016 (NIST Center for Neutron Research, Gaithersburg, MD, USA) as in reference [23], where the computation was based on the model proposed by Kröner [24] and modified by Bollenrath [25] and Behnken [26]. Owing to the assumption of grain-interactions, the model allowed for the use of specific grain shapes to calculate the right diffraction elastic constants. Moreover, the crystallographic preferred orientation (texture) of the sample given by the orientation distribution function (ODF) was also included in the model.

3. Results and Discussion

3.1. Neutron Diffraction and Residual Strain

The residual strains were calculated using Equation (1), where $\theta_{hkl}^B$ angles were determined from the neutron diffraction measurements. The average of the measured $\theta_{hkl}^B$ angles between points $-3$ and $3$ in the axial direction of the initial sample were taken as the stress-free reference $(\theta_{hkl}^B)$, because the
angle variation through the sample was small in contrast to the hoop and radial directions. The residual strains in the radial, hoop, and axial directions are shown for each sample in Figure 2a–c. The sample after the first C-ECAP pass was denoted as C1, then after the second pass as C2, and after the third pass as C3.

**Figure 2.** Residual strain distribution in the axial (a), hoop (b), and radial (c) direction of the samples after the first, second, and third C-ECAP pass.

### 3.1.1. Axial Direction

It was obvious that in the axial direction, after the first pass, the peripheral regions were almost strain-free whilst in the center of the sample, large strain with compression character was present. This behavior resulted in a large strain gradient, where the strain changed from \(\sim -17 \mu \varepsilon\) to \(\sim -600 \mu \varepsilon\) (on the left side of Figure 2a-C1) and from \(\sim -600 \mu \varepsilon\) to \(\sim -110 \mu \varepsilon\) (on the right side of Figure 2a-C1). Furthermore, from a comparison of this result with other directions and numbers of passes (Figure 2a–c) it was obvious that such a large gradient was unique for this case.

After the second pass (Figure 2a-C2), the compression strain increased in the periphery, whereas in the center part it had slightly decreased. Consequently, the strain gradient was less pronounced. In general, the shape of the strain distribution through the samples in the axial direction, after the first and second pass, was a “V” form.

After the third pass (Figure 2a-C3), the compression strain reached a lower value in the center of the sample and at the same time the strain had increased in the periphery. The shape of the overall strain distribution lost the “V” form, and it took on a rather wave-like shape with the lowest compression strain in the center of the sample. The reason was that during the third C-ECAP pass,
the alignment of the work-piece, as a consequence of the previous severe deformation, was not precise. Consequently, the stress distribution during the processing was uneven, which caused the asymmetric residual strains with wave-like form in the axial direction.

In summary, the first pass introduced a large strain gradient in the axial direction toward the center of the sample. The increasing number of passes (2x-3x passes) increased the compression strain in the peripheral regions, whilst decreasing the compression strain in the center part.

3.1.2. Hoop Direction

In the hoop direction, all of the values showed a compression strain character (Figure 2b). It was obvious that after the first and second pass, the strain distribution followed the same shape through the sample (Figure 2b-C1, C2). The difference was in the higher values of the compression strain after the second pass. After the third pass (Figure 2b-C3), the C-ECAP process resulted in a decrement of the compression strain (increasing the interplanar distance) on the left side of the sample, whilst on the right side of the sample, the interplanar distance decreased (compression character was more pronounced) with respect to the previous pass.

3.1.3. Radial Direction

The residual strain distributions through the samples in the radial direction after the first and second pass showed a very homogenous nature with the compression character (Figure 2c-C1, C2). After the first pass, the values were distributed around $-200 \mu \varepsilon$. Only at the right peripheral region of the sample was the strain closer to the stress-free reference. After the second pass, the residual strain values were distributed around $-400 \mu \varepsilon$; however, at the left peripheral region, the compression strain had increased. The third pass resulted in an inhomogeneous residual strain distribution compared to the previous passes (Figure 2c-C3). In the right peripheral region, the compression strain slightly decreased to a level similar to that after the first pass, whilst in the left peripheral region, the strain distribution showed alternating behavior.

In summary, the C-ECAP process resulted in a relatively symmetric distribution of the residual strains in all directions after the first and second pass. The largest strain gradient was in the axial direction (Figure 2a), whilst in the radial direction (Figure 2b), the strain was homogenously distributed. This symmetry was interrupted by the application of the third C-ECAP treatment on the material.

3.2. Microstructure: EBSD Results

The observed grain orientation map, pole figures, grain size distribution, and fractions of the low- and high-angle grain boundaries (LAGB and HAGB) of the initial state, after 1, 2, and 3 C-ECAP passes are shown in Figures 4–7. Samples after the C-ECAP treatment were studied in three different regions across the diameter, schematically shown in Figure 3. In this paper, the notation used at strain scanning by neutron diffraction to describe the cylindrical sample reference system that was used instead of the conventional axis notation used in material engineering (normal—ND, transverse—TD, and rolling direction—RD). In the pole figures, the vertical axis represented the hoop direction (Hoop), the horizontal axis represented the radial direction (Radial), and the normal of the pole figure plane represented the axial (Axial) direction.
Figure 3. Scheme of the investigated regions with the Electron back-scatter diffraction (EBSD) method.

Figure 4. Grain orientation maps, local micro-textures, grain size distributions, and the low- and high-angle grain boundaries (HAGB and LAGB) fractions of the initial state in the periphery (a) and in the center of the sample (b).
In the initial state, the average grain sizes were in the µm domain (5–7 µm) and the grain shape was approximately equiaxed in all the regions (Figure 4a,b). In peripheral region (a), the (0001) basal planes were parallel to the axial direction, forming four distinct maxima, which were between the hoop and radial directions. The (10̅10) prismatic planes formed several maxima in the peripheral region: one perpendicular to the axial direction and then two which were ∼60° inclined from the axial direction of the sample. This so-called partial fiber texture is typical for extruded hexagonal materials [27,28]. In the central region (b), the texture was completely different. The (0001) basal planes formed two distinct maxima which were ∼40° inclined from the axial direction. Furthermore, in the peripheral region, the fraction of the HAGB was less than in the center of the sample, whilst the fraction of the LAGB did not depend on the measured region.

Figure 5. Grain orientation maps, local micro-textures, grain size distributions, and the HAGB and LAGB fractions of the sample after the first C-ECAP pass in the periphery (a,c) and in the center of the sample (b).

After the first C-ECAP pass, significant grain refinement occurred in all the three regions, where the maximum grain size distribution was at 0.8 µm. However, other local maxima could be found at 2.7–3.7 µm (Figure 5a), 2.5–3.5 µm (Figure 5b), and 2 µm (Figure 5c). The microstructure
was formed by a mixture of elongated and equiaxed grains. The dark areas in the EBSD maps were places with a confidence index (CI) < 0.1. Most probably, the higher dislocation density caused poor diffraction patterns in these areas. The local micro-texture in the center (b) and in the right peripheral region (c) was the same, whilst in the left peripheral region (a) it was slightly different. In positions (b) and (c), the (0001) basal planes formed three distinct maxima: along the hoop axis and inclined by 45°–55° from the axial direction, and then two on the radial axis perpendicular to the axial direction. The first mentioned texture component was also observed in other magnesium alloys processed by classical ECAP [29]. In the left peripheral region (c), there were two distinct maxima near the edge of the (0001) pole figure, between the hoop and the radial direction. However, these maxima were blurred, compared to the sharp maxima in positions (b) and (c), and part of the maxima area included even a 45°–60° inclination from the axial direction. The maxima in the (10T0) pole figures in position (a) and (b) had the same location, whilst in position (c), there was no sharp and unique maximum. The (10T1) pole figures showed that these planes had an almost homogenous distribution; however, in position (a), there was a local minimum in the surroundings of the axial direction. In addition, after the first C-ECAP pass, the sample exhibited a higher fraction of HAGBs in all the three regions than that in the initial state, which is typical for materials processed by ECAP [30,31].

![Figure 6](image_url). Grain orientation maps, local micro-textures, grain size distributions, and the HAGB and LAGB fractions of the sample after the second C-ECAP pass in the periphery (a,c) and in the center of the sample (b).
According to grain size distributions of the sample after the second C-ECAP pass (Figure 6a–c), the maxima of the distributions were at the same position as they had been in the case of the sample after the first pass. On the other hand, there were no other notable local maxima, indicating a more homogeneous microstructure, although, the grain orientation maps showed some larger grains. From the (0001) pole figures, the maxima of the main texture component were blurred and \( \sim 40^\circ - 75^\circ \) inclined from the axial direction in all the three regions. If we compared the (10\(\overline{1}0\)) pole figures in positions (a), (b), and (c), it could be noticed that the distribution of these planes was very similar. Owing to these features, the sample after the second C-ECAP pass exhibited the same microstructure in positions (a), (b), and (c). The only difference between the investigated regions was in the fraction of the HAGBs, which was, in respect to level and distribution, the same as in the sample after one C-ECAP pass.

In Figure 7a–c the microstructural characteristics of the left peripheral, center, and right peripheral regions of the sample after the third C-ECAP pass are shown, respectively. From the grain size distributions, it was obvious that further grain refinement had occurred. Maxima were around 0.5 \( \mu \text{m} \).
in all the three regions and the grains were equiaxed. In the grain orientation maps, large dark areas with CI < 0.1 appeared, indicating high dislocation densities. The maxima of the (0001) and (10\bar{1}0) pole figures were nearly at the same position in all the three investigated regions. Nevertheless, they were slightly different compared to the sample after two C-ECAP passes. The (0001) planes in position (a), (b), and (c) formed blurred maxima, inclined 55°–85° from the axial direction. The maxima of the (10\bar{1}0) pole figure were in the axial directions. In addition, the fraction of the HAGBs had increased in all the regions (a), (b), and (c), compared to the previous cases.

In summary, the sample after the first C-ECAP pass exhibited a heterogeneous local micro-texture. The microstructure contained a mixture of elongated and equiaxed grains. However, there was a slight difference in the grain size distribution between the central region and the peripheral regions. Nevertheless, the fraction of the HAGBs had globally increased compared to the initial state. After the second C-ECAP pass, the sample exhibited a relatively homogenous microstructure from the point of view of local micro-texture and grain shape. There was no difference between the investigated regions. The fraction of the HAGBs remained at the same level as it was after one C-ECAP pass. The third C-ECAP pass resulted in the most homogeneous microstructure and it further increased the fraction of the HAGBs. In addition, the character of the distribution of the HAGBs remained the same, i.e., it increased from left to right through the sample. Black areas on the grain orientation maps revealed the inhomogeneous dislocation density at particular regions of the samples after the first and third pass.

CP Ti primarily deforms by ⟨a⟩ slip on basal (0002), prismatic (10\bar{1}0), or pyramidal (10\bar{1}1) planes and by twinning on particular planes [32–34]. Moreover, it was shown that twinning in CP Ti played an important role as one of the main deformation modes during conventional ECAP treatment at elevated temperatures [35–38]. However, twins were not observed in our samples in the cross-section, which did not necessarily mean that they were not present. Many conditions can influence the twin activity and density in hcp materials. In reference [38], the authors showed that the maximum twin density was achieved at 623K ECAP deformation temperature of CP Ti, whilst at 473 K, the twin density was almost one-third of the maximum density. At an 873 K pressing temperature, the twin density was 10 times lower and the twin width was below 0.1 \mu m.

In reference [39], Palán et al. simulated the conditions which prevailed in the C-ECAP machine. The authors results indicated that the temperature, the strain rate, and the velocity distribution were very inhomogeneous in the die. Thus, for example, the temperature in the die could reach 800–850 K where the twin density and width reached the minimum according to reference [38]. Our EBSD observations were conducted at a 0.1 \mu m step size, which was approximately the width of the expected twins at around 800–850 K deformation temperature. Hence, the EBSD measurement evaluation software would not recognize the twins, rather it would report an error for the measured pixel instead. Furthermore, the initial grain size could also influence the twinning activity as it was reported in reference [40] for the Mg alloys and for Ti [41] (including the strain rate). Our starting material had an average grain size of 5–7 \mu m, whilst in the above-mentioned experiments where twinning was reported, the average grain size of the starting materials was several times larger than ours. Thus, the relatively small initial grain size could probably have suppressed the formation of larger twins, which could be observed by our experimental conditions.

3.3. X-ray Texture Measurements

The results of the X-ray texture measurements of the samples after one, two, and three passes in the three different regions of the sample (left peripheral-(a), center-(b), and right peripheral-(c)) are shown in Figures 8–10. The axis notation was identical to the axis notation at the EBSD measurements.
Figure 8. (0002), (10\overline{1}0), and (10\overline{1}1) pole figures after the first C-ECAP pass in the periphery (a,c), and in the central region (b) of the sample.

From the simulated (0002) pole figures at the peripheral regions (Figure 8a,c) after the first C-ECAP pass, the (0002) basal planes parallel to the axial direction formed maxima on the edge of the (0002) pole figures. These components would be indicated as “parallel” components in the following text. Moreover, (0002) planes formed other maxima inclined at \(\sim 50^\circ - 60^\circ\) from the axial direction and distributed between the hoop and radial axis. However, the arrangement was in the opposite sense and it was rotated to hoop axis by \(\sim 120^\circ - 130^\circ\) around the axial direction. This texture component would be noted as the “50°–60°” component. The texture component “50°–60°” was also observed in our EBSD investigation (Figure 5). The same texture component was seen in the (0002) pole figure in the central region (Figure 8b), but it lay on the hoop axis. In this region, this was the only notable maximum, and parallel components were not present. The only notable maxima in the (10\overline{1}0) pole figures were inclined at \(\sim 25^\circ - 35^\circ\) from the axial direction; however, the overall distribution of the maxima formed a ring around the axial direction. The position arrangement of the maxima around the axial direction was in the opposite sense, as it was present at the (0002) pole figures. The maxima formed by (10\overline{1}0) were in an axial direction independently from the measured region; however, the random distribution of the planes inclined by \(35^\circ - 55^\circ\) from the axial direction forming a ring in the peripheral regions. The most intense maxima of the (0002) and (10\overline{1}1) pole figures were in the central region (Figure 8b) and at the same time, the least intense maximum of the (10\overline{1}0) pole figure was also found here.
Figure 9. (0002), (10\bar{1}0), and (10\bar{1}1) pole figures after the second C-ECAP pass in the periphery (a,c) and in the central region (b) of the sample.

The result of texture measurements on the sample after the second C-ECAP pass was visible in Figure 9a–c. In the peripheral regions ((a) and (c)), the simulated (0002) pole figures showed that “50°–60°” components were almost exactly at the same position as they were in the sample after the first pass, whilst “parallel” components were slightly rotated. In the central region (Figure 9b), the “50°–60°” component was rotated counterclockwise to the hoop axis by ~20° around the axial direction, with respect to the position of the maximum after the first pass, and its intensity increased as well. The inclination of the maxima from the axial direction in the (10\bar{1}0) pole figures was the same as they were in the first pass, but they were rotated clockwise by ~25°–35° around the axial direction compared to previous positions. The maxima in the (10\bar{1}1) pole figure were still exactly in the axial direction; however, higher density regions inclined by ~35°–50° from the axial direction appeared in the peripheral regions ((b) and (c)).
Figure 10. (0002), (10\bar{1}0), and (10\bar{1}1) pole figures after the third C-ECAP pass in the periphery (a,c) and in the central region (b) of the sample.

Figure 10a–c revealed further texture evolution after the third C-ECAP pass. A much weaker intensity, than in the previous pass, could be seen in the (0002) pole figure in the position of the “50°–60°” texture component in the left peripheral region (a), though it was still present. The opposite could be observed in the right peripheral region (c) where the “50°–60°” texture component had gained intensity compared to the previous pass. The basal component exhibited a much stronger intensity in the left peripheral region (a), whilst in the right peripheral region (c), the intensity maintained the same level. The most intensive “50°–60°” texture component could be observed in the central region (b), and its position did not change in comparison to the previous pass. The position of maxima in the (10\bar{1}0) pole figures in peripheral regions ((a) and (c)) coincided with the positions after the first C-ECAP pass. However, the maxima intensity was stronger, especially in the left peripheral region (a), where the intensity reached the maximum of the scale. In the same region (a), the (10\bar{1}1) planes formed three distinct notable maxima which were inclined by \(\sim 30°–40°\) from the axial direction. On the other side of the sample (Figure 10c), except the clear maxima in the axial direction, the distribution of the (10\bar{1}1) planes inclined by \(\sim 30°–40°\) from the axial direction formed blurred regions with a higher intensity in the (10\bar{1}1) pole figure (c). The same held for the (10\bar{1}1) pole figure in the central region (b); however, three regions could be spotted with more clear edges.

The above-discussed results can help to illustrate the preferred orientation in the different regions of the samples. It could be observed, that the C-ECAP mechanical treatment resulted in different textures in the different regions. However, the “50°–60°” component was present in all the regions, the rotation with respect to the radial axis was dependent on the region and the number of passes. The theoretical shear plane normal was inclined by 45° from the extrusion-axial direction...
due to the C-ECAP design. If we assumed, for example, after the first pass in the central region, that the main deformation mode was a dislocation slip in the basal (0002) planes in direction $\langle 11\bar{2}0 \rangle$, then theoretically the “50–60” component in the (0002) pole figure should be inclined exactly by 45° from the axial direction and fully located on the hoop axis. However, in our results, the maximum in the (0002) basal pole figure appeared at a $\sim$55° inclination. This deviation may be explained citing the results of Prangnell et al. [42], where the authors showed with the help of finite element modelling that the filling of the outer corner of the die in conventional ECAP was dependent on whether the friction between the material and the die wall was present. The present outer curvature of the die corner could theoretically change the real shear plane, which resulted in a slightly different observed macro-texture from the theoretically predicted one of the material. The material flow in the die was also simulated in reference [43], where it turned out that its distribution was highly dependent on the value of the friction coefficient. Furthermore, as discussed at the end of Section 3.2, different conditions prevailed in different parts of the C-ECAP machine. In reference [39] (Figure 2), it was shown that in the corners and the adjacent areas, the strain rate and velocity distribution was practically zero. This may be additional evidence that the real shear plane can be different from the predicted one (Figure 11).

![Figure 11. Simulated strain rate, as in reference [39].](image)

In summary, it can be concluded that the activity of the particular slip system depended on the number of passes and the relative position within the specimen. After the first pass in the central region, the basal slip system $\langle 11\bar{2}0 \rangle$ (0002) was the dominant slip mode. However, in the peripheral regions, the prismatic slip system $\langle 11\bar{2}0 \rangle$ (10T0) and pyramidal slip system of the first order $\langle 11\bar{2}0 \rangle$ (10T1) were activated, which could be deduced from the positions of the maxima of the measured pole figures. In the first case, i.e., the basal slip in the central region after the first pass (Figure 8b), the maximum of the “50–60” component in the (0002) pole figure lay fully on the hoop axis and was inclined by 55° from the axial direction. Thus, the (0002) basal planes were parallel with the mentioned other shear plane and preferably oriented for shear deformation in the vertical direction on the shear plane. In the second case, i.e., the prismatic and pyramidal first order slip in the periphery (Figure 8a,c), the maxima of the “50–60” component of the (0002) pole figure was rotated by $\sim$120° from the hoop axis on each side. The maxima of the (10T0) pole figures were inclined by $\sim$30° from the axial direction and rotated by $\sim$60° from the hoop axis. This arrangement of texture maxima allowed slipping $(a)$ dislocations in the prismatic and in pyramidal planes [44] in the direction towards the center of the work-piece. This was due to the fact that in peripheral regions, the shear direction had a normal component to the edge of the die beside the vertical one. Consequently, the stress fields of the different types of dislocations (basal, prismatic, and pyramidal) differently affected the residual strain distribution in particular regions. Obviously, a very large amount of basal dislocations was generated.
during the first pass owing to the initial texture in the central region (Figure 4b). This resulted in a large compression residual strain in the axial direction (Figure 2a–C1). After the second pass, the prismatic and pyramidal dislocations also appeared beside the basal dislocations due to the rotated “50°–60°” component. This coexistence of basal, prismatic, and pyramidal dislocations in the central region could be observed after the third pass as well. In the periphery, only the prismatic and pyramidal dislocations were active after the first and second pass. They generated a stress field, which acted in the hoop direction mainly after the first C-ECAP pass (Figure 2b). However, after the second pass, they acted in the axial direction as well (Figure 2a,b). It could be assumed, that the grain size influenced the dislocations effects in the different directions. After the third pass, they were only active in the right periphery as can be seen from the maxima of the (0002) and (10T0) pole figures (Figure 10a,c).

3.4. Residual Stress Calculation Results

The residual stress was calculated using the method described in Section 2. EBSD and X-ray texture measurements were conducted in the three regions (left and right peripheral regions, and the central region) of the samples; hence, the calculation was provided for each of the three investigated regions and passes with separate ODFs. The grain shapes for particular regions and passes were determined from our EBSD results in the cross section and from reference [39] in the longitudinal section of the samples. The grain size and shape did not depend significantly on the region in the sample for particular passes, as seen in Section 3.2. Thus, it could be assumed that the same statement was valid as well for the axial direction. Points –3, –2 belonged to the left peripheral region, –1, 1, 1 to the central region, and 2, 3 to the right peripheral region. The results of the calculation are seen in Figure 12a–c.

![Figure 12](image-url)  
**Figure 12.** Residual stress distribution in the axial (a), hoop (b), and the radial (c) direction of the samples after the first, second, and the third C-ECAP pass.
It could be seen from the stress distributions (Figure 12a–c), that the distributions followed almost the same shape as the strain distributions from Section 3.1 (Figure 2a–c). In the center of the sample C1 in the axial direction (Figure 12a), a relatively large, $-63$ MPa, stress prevailed, as was expected from the strain measurements. In the left peripheral region, the C1 sample exhibited $+14$ MPa tensile stress, whilst the right peripheral region was practically stress-free in the axial direction in contrast to the measured compression strain (Figure 2a). It was interesting to see the change of sign between the strain and the stress at points $-3$, $-2$, and 2 mm. At points $-3$, $-2$, and 2 mm, the strain was $-17$, $-39$, and $-82 \mu \varepsilon$, respectively, but the stress was $+14$, $+14$, and $+2$ MPa. The same behavior was spotted at point $-3$ in the C2 sample (Figure 12a) and at points 2 and 3 mm in the sample C3 in the radial direction (Figure 12c). Sample C2 exhibited a very symmetric distribution of axial stresses around the center of the sample with the maximum of compression stress at $-37$ MPa. The compression stress increased from the edges towards the center of the sample C2. The C3 sample showed a large, $-63$ MPa, stress at the right peripheral point $-3$ mm. In the central region ($-1$, 0, and 1 mm) and in the right peripheral region (1 and 2 mm) the residual compression stress decreased.

In the hoop direction (Figure 12b), samples C1 and C2 showed a very symmetric distribution of stresses around the center; however, it was in opposite progress than it was in the axial direction. These samples had the lowest value of compression stress in the center and it increased towards the edges. Sample C1 seemed to be stress-free in the center. Sample C3 showed a linearly increasing compression stress from the left to the right edge in the hoop direction.

The residual compression stresses did not show any gradient in the radial direction for samples C1 and C2, though at point $-3$ mm it was slightly increased (Figure 12c), and they were distributed at around $-18$ MPa. Additionally, a very inhomogeneous distribution could be seen at sample C3. Its maximum compression stress was at point $-1$ mm. From this point, the stress decreased towards the right edge of the sample until its sign changed between points 1 and 2 mm. The right edge of the sample exhibited tensile stress at about $+20$ MPa.

4. Conclusions

Neutron diffraction measurements revealed the residual strain distributions of the samples after one, two, and three passes of the C-ECAP machine. Symmetric strain gradients were present in the case of samples after first and second pass in the axial and hoop directions. In the radial direction, there was no gradient and also the strain distribution was constant through the sample. The third pass broke the gradient symmetry in the axial and hoop direction. Moreover, in the radial direction, the constant strain distribution was interrupted as well.

EBSD observations confirmed the grain refinement effect of multiple pass C-ECAP treatment; however, after the second pass, the grain refinement was not as significant as after the first pass. Deformation twins were not observed, though twins with nano-sized width may have been present. Thus, it is most likely that a dislocation slip was responsible for the deformation of the Ti during the C-ECAP treatment. This concept was supported by the experimental pole figures from the EBSD measurements and the local X-ray macro-texture measurements. In addition, the macro-texture of the samples was dependent on the investigated region, which could indicate the activation and combination of different slip systems as the function of the position in the work-piece.

The calculation of the residual stress distributions showed that compression residual stresses prevailed in the specimens after the C-ECAP process and they followed the shape of the distributions of the residual strains.

Author Contributions: K.M. and G.N. conceived and designed the experiments; J.P. and M.D. prepared the C-ECAPed materials; C.H. performed the neutron diffraction experiments; G.N. prepared the samples for EBSD and X-ray texture measurements; K.H. studied microstructure of the samples using EBSD; P.C. performed the X-ray texture measurements; G.N. and K.M. analyzed the data; G.N. wrote the paper.

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