Microstructure evolution and mechanical behaviour of severely deformed pure titanium through multi directional forging

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A B S T R A C T

Multi directional forging (MDF) is one of the severe plastic deformation methods utilized for production of ultrafine grained materials with improved mechanical properties. The main goal of the current study is to enhance mechanical properties of commercial pure (CP) titanium using grain refinement by MDF method. For this purpose, after one hour annealing at 800 °C, the CP titanium was forged by MDF process up to six passes at room and 220 °C temperatures. The results of microstructure analysis by scanning electron microscope equipped with EBSD showed that average grain size of samples after six MDF passes at room and 220 °C temperatures was about 60 times finer than that of the annealed specimen. The mean grain size of the titanium is reduced from 64 μm to 1 μm after 6 passes at room temperature. Also, the tensile and shear strengths are notably improved by increasing number of MDF passes and reduction of the processing temperature. Yield tensile and shear strengths of six passes MDFed samples at room temperature were about 2.5 times greater than those of the annealed specimen. The correlation between tensile and shear strengths was studied as well. The ratio between yield tensile and shear strengths and also between ultimate tensile and shear strengths was achieved about 1.5 and 2, respectively.

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1. Introduction

It is generally accepted that severe plastic deformation (SPD) method improves mechanical properties of materials through grain refinement and fabricates ultrafine grained (UFG) and even nanostructure (NS) metals and alloys [1–6]. Among various SPD techniques, equal channel angular pressing (ECAP), high pressure torsion (HPT), friction stir processing (FSP), accumulative roll bonding (ARB), equal channel forward extrusion (ECFE), and multi directional forging (MDF) are the most prominent and fast-advancing techniques in materials engineering. All these techniques are based on imposing a large magnitude of shear strain into the material causing significant increase in dislocations density and consequently lead to substantial refinement of the microstructure. It is worthy to note that unlike the bottom-up approaches such as ball milling or inert gas condensation, the processed materials through SPD methods have no porosity, impurity, or cracks [7–10]. Accordingly, materials with higher strength to weight ratio could be obtained by SPD compared to the different conventional metal forming techniques such as forging, extrusion, and rolling. As a consequence the aforementioned methods are of high interest. The potential application of UFG and/or NS materials is in particular for the production of very light automobiles, airplanes, and machines with low energy consumption and less environmental pollution [10–14].

Among all SPD techniques, MDF has more potential to be utilized for fabrication of relatively large bulk samples with industrial application due to the simplicity and low-cost of the process and tooling. MDF is a closed forging process where a rectangular cube with different dimensions is pressed at different directions in consecutive passes. The direction of pressing alternates in sequence X→Y→Z→X … between successive passes [15–18]. There have been performed a lot of experimental work on the MDF processing of various materials including stainless steel, copper, aluminium alloys, titanium alloys, and magnesium alloys. It is reported that after several passes of MDF process, ultrafine grained materials with the grain size of less than 0.5 μm can be produced. Such significant grain refinement leads to notable increment in mechanical properties of the material [10,15–17,19,20].

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Titanium alloys have been utilized especially in military and aviation industry due to their high strength to weight ratio. Additionally, medical applications are another main utilization of titanium alloys because of their suitable mechanical properties and excellent corrosion resistance. These alloys exhibit low density despite their high melting point (refractory property). Previous studies indicated that commercial pure titanium has also excellent formability. It could be cold rolled at ambient temperature up to 90% reduction in thickness without a major cracking [21,22]. Such formability is not usual in HCP metals and may be attributed to relatively low c/a ratio leading to higher activity of non-basal slip systems and twinning. As it was mentioned above, titanium alloys have vast applications in medical devices due to their suitable biocompatibility. On the other hand, commercially pure titanium (CP-Ti) has lower strength in comparison with commonly used Ti-6Al-4V, however, it could be refined by various SPD techniques and its applicability would considerably increase by this approach [23,24]. It is worthwhile to mention that Ti-6Al-4V is used as an implant material despite the toxic effect of the released aluminum and vanadium ions in the human body. Therefore, high strength CP-Ti may be a suitable candidate to solve this problem [22,25].

In this study, commercial pure titanium - Grade 2 was processed by MDF up to six passes at the room and elevated temperature without the heat treatment procedure between the individual passes. Grain refinement mechanisms during the processing at different temperatures were analysed by scanning electron microscope (SEM) equipped with electron backscatter diffraction (EBSD) camera. Furthermore, evolution of mechanical properties of processed samples was studied by shear punch and tensile tests. Correlation between microstructure and mechanical properties was studied and discussed.

2. Materials and method

2.1. MDF processing procedure

The ASTM Grade 2 commercial pure titanium was used in this research. The as-received material was in rolled condition. Chemical composition was analysed by GNR Italy Metallab-7580J spectrometer and is shown in Table 1. The as-received material was first cut and machined to the size of 10 × 10 × 15 mm$^3$, and afterwards all prepared samples were annealed at 800 °C for 1 h. Fig. 1 shows the used MDF setup including die, punches, and sample. The die was built from H13 tool steel and was heat treated in order to achieve appropriate hardness of 52 HRC.

Samples of the investigated material were processed by MDF up to 6 passes at room temperature (RT) and 220 °C. Molykote 1000 Paste lubricant with composition of grease, graphite, calcium fluoride and copper metal powder was applied to reduce the friction coefficient between the interfaces of die, sample and punch. The magnitude of the imposed plastic strain during one pass of MDF process was calculated according to Eq. (1) with the result of 0.47 for the aforementioned condition [26]. In this relationship, $H$ and $W$ are length and width of the sample, respectively.

$$\varepsilon = \frac{2}{\sqrt{3}} \ln \left( \frac{H}{W} \right)$$  \hspace{1cm} (1)

2.2. Microstructure and analysis of mechanical properties

The microstructure of all studied samples was investigated by EBSD in scanning electron microscope ZEISS Auriga Compact at an accelerating voltage of 15 kV and a beam current of 10 nA. Samples were cut from the bars as schematically shown in Fig. 2, and mechanically ground and polished. Finally, ion polishing was performed by Leica EM RES102. Microstructural examinations were carried out in the middle of the cross-section - green rectangle in Fig. 2. Due to the different grain size of the investigated samples, various step sizes from 50 nm to 1.5 µm with different area sizes from $30 \times 50 \mu m^2$ to $1000 \times 2000 \mu m^2$ were used in EBSD analysis. Finally, EDAX OIM TSL7 software was utilized for raw data analysis.

Shear punch test with the schematic representation shown in Fig. 3 (a) was used for all samples to study the effect of MDF on shear mechanical properties of CP-Ti. Accordingly, the used die and punch diameters were 6.25 mm and 6.2 mm, respectively. The samples were cut from the central zone of the initial and deformed samples with thickness of ~0.7 mm. All tests were conducted using Zwick/Roell Z100 universal testing machine with strain rate of $10^{-3}$ s$^{-1}$ [27]. The force $P$ was measured in term of the punch displacement $h$ for all conditions. Then, they were converted to shear stress $\tau$ versus normal displacement $d$ according to the relationships (2) and (3), in which $t$ represents sample’s thickness. In addition, tensile tests have been performed with miniature samples (Fig. 3 (b)) using Zwick/Roell Z100 universal testing machine with constant speed of $10^{-3}$ s$^{-1}$. Both shear punch and tensile tests were repeated three times for each condition.

$$\tau = \frac{P}{\pi dt}$$  \hspace{1cm} (2)

$$d = \frac{h}{t}$$  \hspace{1cm} (3)

3. Results & discussion

3.1. Microstructure characterization

3.1.1. Initial material

For the annealed condition, inverse pole figure (IPF) map, grain boundary map, histogram of grain size distribution and histogram

![Figure 1](image1.png)

Fig. 1. Multi directional forging setup including (1) Die, (2) Big punch, (3) Small punch, and (4) CP-Ti sample.
of misorientation angles resulted from the EBSD analysis are presented in Fig. 4. Accordingly, the annealed sample has equiaxed coarse grains without any twins. Low angle grain boundaries (LAGBs) with misorientation angles $2^\circ$-$5^\circ$ are highlighted by red lines, LAGBs with misorientation angles $5^\circ$-$15^\circ$ are shown with green lines and high angle grain boundaries (HAGBs) with misorientation angles $>15^\circ$ was illustrated with blue lines in Fig. 4 (b). As expected, from both EBSD maps and histogram of misorientation angles in Fig. 4 (d), it can be concluded that the most of the boundaries - 86%, are HAGBs. Average grain size calculated from Fig. 4 (c) as an area fraction is about $64\mu m$.

3.1.2. One pass of MDF

The IPF and grain boundary maps after the first pass of MDF process at RT and $220^\circ C$ are shown in Fig. 5. Histogram of grain size distribution and histogram of misorientation angles of the mentioned samples are shown in Fig. 6. According to Fig. 5 (a), a lot of deformation induced twins formed due to the application of one pass of MDF at RT. The formation of twins was activated as a result of the limited number of independent slip systems in HCP titanium, and also the restriction in movement of dislocations with a basal burgers vector when the loading is parallel to the c-axis. Formation of high volume fraction of twins with different twinning plane resulted in substantial segmentation of grains and breakdown of the original microstructure. The twins are usually in form of convex-shaped lenses, which have two interface types of coherent and incoherent boundaries. The incoherent part is the edge of the twins (lens’s corner), which cannot fit well with the parent lattice. In fact, the incoherent section with the surrounding boundary is formed by the dislocation rows. Dislocations are formed due to the high amount of strain, and they are arranged in the side borders and eliminate the mismatch between lattices of parent and twin; therefore, the energy of twin’s boundaries is reduced $[21,28-30]$. Furthermore, since the sharp front of the growing twins is eliminated by collision to the free surface, some of the twins will have a half-lenticular shape according to Fig. 5 (a). Also, most of the twins are located across the grain boundaries; because twins cannot reach from one grain to another due to the difference in the arrangement of adjacent grains; see Fig. 5 (a).

As shown in Fig. 5 (b), most of the boundaries are HAGBs, as they are twin boundaries. Twin-boundaries are HAGBs with lower energy in comparison with grain boundaries (HAGBs). Nevertheless, the fraction of boundaries with misorientation angles of $2^\circ$-$5^\circ$ is also considerable. The LAGBs were formed particularly through plastic deformation especially at the center of MDFed samples, where the shear microbands collide $[15,16]$. Dislocations are preferentially nucleated from the grain boundaries, where the plastic strain is the highest. As a result, the highest density of LAGBs with misorientation angles of $2^\circ$-$5^\circ$ was observed along former grain boundaries; see Fig. 5 (b).

It is clear from Fig. 5 (c) that the first MDF pass at $220^\circ C$ resulted in lower grain fragmentation. Only the limited number of twins was observed when compared to the sample processed at RT, however, substantial increase of LAGBs with misorientation angles of $2^\circ$-$5^\circ$ was seen, as depicts Fig. 5 (d). The plastic strain stored in dislocation was particularly represented by these boundaries; therefore, notable change of intensity of deformation mechanisms was observed with the increase of the processing temperature. Increase of processing temperature have negative effect on twinning activity, but oppositely have positive effect on slip activity, because the operating of deformation twinning restricts at high temperature, and also the critical resolved shear stress of all slip systems.
including \(<c+a>\) slip decreases with increasing temperature [28,30,31].

According to Fig. 5 (d), there is a high fraction of LAGBs after the first pass of MDF process at the 220 °C. This is proved in Fig. 6 (a)
where the histogram of misorientation angles demonstrates the high fraction of boundaries with misorientation angles of 2°—5°. In contrast, the microstructure of the sample processed at RT exhibits the highest fraction of boundaries with misorientation angle of ~65° and ~87°. This indicates the excessive formation of {1122} and {1012} twins, correspond with the IPF map; see Figs. 6(a) and 5(a). Additionally, some twins are formed within the primary twins, which are entitled secondary twins. {1122}-{1012} double twins are also observed after compression of pure titanium [32].

Nevertheless, the increase of dislocation activity during the processing at 220°C did not lead to comparable grain refinement as in sample processed at RT. The reason is twofold — (a) increase of deformation temperature leads to reduction of the imposed strain to the structure, as a result, shear bands and micro shear bands with large local strains are reduced and (b) twinning introduce to the material high fraction of cells separated by HAGBs, and could transform to grains with subsequent deformation. As a result, EBSD analyses of samples processed by one MDF pass revealed that the dislocation slip and twinning have the dominant role on the grain refinement during the MDF process at 220°C and RT, respectively, but the grain fragmentation is strongly affected by the dominant deformation process. This is also reflected in Fig. 6(b), where grain size distribution of both samples is shown. The sample processed at RT had considerable fraction of grains with size less than 6 μm and the mean grain size calculated by the area fraction was 5.4 μm. Such grain refinement comparing to the annealed condition can be mainly related to the formation of high angle grain boundaries due to the severe twinning. On the other hand, sample processed at 220°C still contained large grains and area fraction of the small ones was low. The calculated mean grain size was 28 μm.

3.1.3. Three passes of MDF
Fig. 7 depicts the IPF and grain boundary maps after the third pass of MDF at both processing temperatures - RT and 220°C. Corresponding histogram of grain size distribution and misorientation angle distribution for the aforementioned samples are represented in Fig. 8. Increased number of MDF passes resulted in the finer microstructure of the investigated material - compare Figs. 5 and 7. The main reason of this observation could be associated to the 90° sample rotation between subsequent passes and more pronounced intersection of twins and dislocation bands. Still, higher MDF temperature leads to a considerably suppressed grain refinement. In case of the sample processed at RT, grain refinement rate through twinning was severely suppressed with increasing number of MDF passes. Activation energy for twinning is strongly dependant on the grain size. Volume fraction of twins in titanium at constant strain is significantly influenced by grain size, according to Eq. (4) [33]:

![Fig. 6.](image1)
![Fig. 7.](image2)
\[ V_v = \frac{4\pi\beta k^2 d}{\sqrt{d}} (\frac{k}{d} + \tau_0(e)) \] (4)

Where, \( V_v \) is the volume fraction of twins, \( \beta \) is the material constant (scaling factor), \( k \) is the Hall-Petch constant and \( \tau \) is the frictional stress. It is needed to note that a linear relationship can be drawn between volume fraction of twins and grain size, if \( \frac{k}{\tau_0(e)} \geq \tau_0 \) [33]. Therefore, probability for nucleation of new twins and consequently the volume fraction of new twins was significantly reduced by the first MDF pass and continuously decreased with the following ones. However, twinning was still an important refinement mechanism especially with rotation of strain with subsequent passes. Grains, which were oriented unfavourable for twinning during the first pass or remained coarse enough, were subjected to twinning during the next passes. Consequently, sample processed at RT exhibited considerably refined microstructure after three MDF passes, in which the twins and parents could not be easily distinguished from each other. On the other hand, much coarse grain structure observed in sample processed at 220°C allowed observation of distinctive twins even after three MDF passes; see Fig. 7 (c).

Fig. 7 (b) indicates significant refinement through the HAGBs; nevertheless, the microstructure exhibits also a lot of LAGBs, which are particularly visible as a high peak in Fig. 8 (a). The aforementioned LAGBs were formed particularly by deformation induced during second and third passes. As mentioned, the decrease of grain size results in reduced activity of twinning and slip mechanism became more pronounced, as a result, higher fraction of \( 2 - 5^\circ \) LAGBs was observed in sample processed by three MDF passes compared to the sample processed by one pass, cf. Figs. 6 (a) and 8 (a). On the other hand, the fraction of \( 2 - 5^\circ \) LAGBs measured in the samples processed at 220°C was high after both processing steps. However, one could notice that fraction of \( 2 - 5^\circ \) LAGBs was considerably higher in the sample processed by three passes at RT compared to the one processed at 220°C. This could be explained by higher tendency to work hardening and recovery at room and elevated temperatures, respectively. Also, these low angle boundaries may be fabricated by the extension of central deformation zone of MDF sample by addition of pass numbers. Still, the peak in misorientation angles histogram is within low angle boundaries zone and is an indicator of capability to transform LAGBs to HAGBs as grain refinement process during subsequent passes.

As can be observed in Figs. 7 and 8, the grain size distribution after three passes is not still homogenous and the grains have elongated shape, regardless to the processing temperature. The microstructure of both samples (processed at RT and 220°C) was significantly refined and is more uniform compared to the samples processed by one pass, irrespective of the processing temperature. Average grain size was 1.4 μm and 4.1 μm for RT and 220°C samples, respectively. The highest area fraction of grains with diameter of ~0.5 μm was measured in both studied conditions. However, much larger grains were still observed in both investigated samples. Especially in the sample processed at 220°C, large grains of diameter up to 20 μm were still recognized. Therefore, grain refinement process and homogenization of the microstructure have not been completed even after three passes of MDF and further processing was needed.

3.1.4. Six passes of MDF

The results of EBSD analysis conducted on samples processed by six MDF passes at RT and 220°C are displayed in Figs. 9 and 10. In sample processed at RT, almost homogenous UFG structure was achieved, see Fig. 9 (a) and (b). The lamellar structure formed by excessive twinning during the first pass has been almost completely suppressed. Only few former twins could be recognized in the sample processed at RT. Such refinement and homogenization resulted from massive intersection of twins and shear microbands after each pass of MDF process and continuous transformation of LAGBs to HAGBs. Microstructure observation also pointed out that applying higher number of MDF passes reduces gradually the materials’ anisotropy. Mechanical properties measurements, which will be discussed in the next section, indicated that the mechanical properties of titanium saturate in the sixth pass. It means that the grain refinement reached its maximum. Further processing with the same conditions does not lead to higher dislocation density and consequently lower grain size. In case of the sample processed at 220°C, the final step of the processing did not lead to complete homogenization of the microstructure and few larger grains is still present, see Fig. 9 (c). However, it is obvious that the microstructure of high temperature sixth pass titanium particularly consists of equiaxed ultrafine grains. Therefore, it could be concluded that RT as a processing temperature is more effective for processing of pure titanium by MDF.

According to Fig. 9 (b) and (d), there are high number of HAGBs at the boundaries map of pure titanium after sixth passes of MDF at both RT and 220°C. Also, as expected, higher amount of LAGBs is transformed to HAGBs after sixth passes in comparison with microstructure after three passes. The results shown in Fig. 10 confirm this observation, in which the simultaneous decrease of peak in LAGBs zone and increase of peak in HAGBs zone are evident; it is another proof of low angle boundaries transformation to high angle ones by addition of MDF pass number.
One of the momentous mechanisms for fabrication of ultrafine grained and nanostructure materials through various severe plastic deformation methods such as MDF process is grain’s segmentation which is caused by two types of dislocations boundaries (sub-boundaries) [25]:

- Geometrically necessary boundaries (GNBs), extended boundaries of planar dislocations.
- Incidental dislocation boundaries (IDBs), formed by statistical trapping of dislocations.

IDBs nucleate usually in dislocations sources or they could be the direct result of cross slip or other processes [25]. On the other hand, when high tension gradient is imposed to the metal structure using a SPD process, another group of dislocations entitled as geometrical dislocations can be found in locations with high amount of tension gradient such as grain boundaries and/or they can be formed by activation of different slip systems [34,35]. Accordingly, the grain segmentation mechanism is based on the considerable increment of dislocations density resulted from the severe shear strains of the MDF process. Therefore, application of severe plastic deformation by MDF leads to the formation of both IDBs and GNBs, and consequently cell blocks and cells are formed by IDBs and GNBs, respectively. Increasing plastic strain by increasing MDF pass number results in increase of the misorientation angle of both boundary types; hence, low angle boundaries or sub-boundaries are formed. It is generally accepted that the rate of misorientation angle increment is higher for the GNBs as compared to the IDBs. Thus, GNBs are transformed to the high angle boundaries by increasing strain, while the misorientation angles of IDBs are usually less than 3°. However, as shown in EBSD analysis, the division of grains is mainly caused by twinning in case of pure titanium, especially in the initial pass at room temperature [35–38].

The other mechanism which enables production of ultrafine grained and nanostructure materials through various SPD methods is dynamic recovery (DRV) [9]. As it will be discussed in the following, DRV can be occurred during MDF process at RT due to the temperature increment during the process. As known, tendency of dislocations to form low energy cellular structure is high for the majority of pure metals, even at very low temperatures. The dynamic recovery is more pronounced in metals with high stacking fault energy (SFE), because the main mechanism involved in the dynamic recovery is the heat-activated cross slip [21]. SFE of pure titanium with hcp structure can be considered high. SFE for basal slip planes and prismatic ones are 300 and 150 mJ/m², respectively. Higher value of SFE for basal planes stems from the fact that these planes are denser and have high atomic density [9,39]. The
temperature increase during the SPD process can be estimated from the following relation [9,40]:

\[ \Delta T = \left( \frac{\beta}{\rho C_v} \right) \int_0^r \sigma \epsilon \ \text{d}r \]  

(5)

Accordingly, \( \beta \) is the efficiency of converting mechanical work to heat, which is about 0.9, \( \rho \) is the density of pure titanium, which is about 4520 kg/m\(^3\), \( C_v \) is the special heat capacity of titanium, which is equal to 528 J/kg.K, and \( r \) is the mean effective stress component for material deformation. For instance, temperature rise of 173 °C has been reported for one cycle of ARB process at the ambient temperature with the effective strain of 0.5 and as a result homologous temperature of about 0.26 was obtained. Also in this work, the amount of temperature increase after one pass of MDF process with the effective strain of about 0.5 was calculated by Eq. (5). For this purpose, relation between the stresses implied by the hydraulic press as a function of the imposed strain for the titanium specimen was measured (Fig. 11); see Eq. (6). Consequently, the temperature rise during the SPD process can be estimated from the following relation [9,40]:

\[ \sigma = 2 \times 10^{10} \epsilon^3 - 2 \times 10^{10} \epsilon^2 + 7 \times 10^9 \epsilon - 4 \times 10^7 \]  

(6)

At high number of MDF passes and processing temperatures, continuous dynamic recrystallization (CDRX) can also act as an effective grain refinement mechanism along with the other mechanisms during MDF of Ti. CDRX can be continuously active at whole structure due to the high SFE of pure titanium. Also, the high amount of energy stored by the intensive deformation may decrease the recrystallization temperature. Therefore, the one of the most probable formation mechanism of new grains could be CDRX or DRX. Also, the geometric dynamic recrystallization can be active as the other mechanism of grain refinement of pure titanium at high temperature and pass number, in which, high angle scalloped boundaries are introduced and segmented the grains [9,28,42–44].

Of course, as mentioned earlier, it is highly possible that several mechanisms act together. For instance, high angle grain boundaries get closer to each other by the increment of applied strain; hence, their distance is reduced and microstructure is refined [44]. Also, the distance between low angle and high angle boundaries are reduced considerably for the sample after six MDF passes as compared to the samples after 1 and 3 MDF passes. According to the grain size distribution of sixth pass titanium at both RT and 220 °C (Fig. 10(b)), the fraction of grains with sizes higher than 1 μm is reduced, and most of them have size below 1 μm. The calculated mean grain size using area fraction method for sixth pass MDFed titanium at RT and 220 °C are 1 μm and 1.2 μm, respectively, which correspond to ~60 times reduction as compared to the annealed sample. The achieved mean grain size for the room temperature sixth pass titanium shows further finer grains in comparison with the elevated temperature sixth pass one. It is also obvious that the difference between mean grain sizes of 6 passes MDFed titanium at ambient and high temperature is reduced and the microstructure becomes almost homogenous. The effect of MDF pass number and temperature on the average grain size of the specimens calculated by area fraction methods is summarized in Table 2.

By considering the previous investigations regarding SPD processing of CP-Ti [31,40,45–47], it should be noted that the achieved grain refinement by MDF of pure titanium is almost similar to that achieved by ECAP process, but is inferior to the HPT method. The mean grain size achieved in various studies concerning ECAP processing of CP-Ti was reported to be about 860, 830, 700 and 560 nm, achieved after 4 passes at 300 °C, 4 passes at RT, 3 passes at 400 °C and 8 passes at 450 °C, respectively [31,40,45,47]. The negligible difference between the mean grain size obtained after the final pass of MDF in current research and those obtained after the final pass of ECAP in the literature can be attributed to the different imposed strain to CP-Ti in mentioned processes. Zhilyaev et al. [46] also reported that five full revolution of HPT leads to the formation of ultrafine grained CP-Ti with the mean grain size of about 200 nm as a result of imposing very high strain to the specimens during HPT process.

3.2. Mechanical properties

3.2.1. Tensile properties

The engineering tensile stress-strain curves of all investigated conditions are shown in Fig. 12. Generally, the results imply that tensile strength is considerably improved by increasing pass number. In addition, the magnitudes of tensile yield and ultimate tensile strengths for various MDF conditions are presented in Fig. 12. It was observed that the both yield and ultimate strengths of samples processed at RT are higher than those processed at 220 °C. It should be noted that the tensile and yield strengths of samples processed by six passes at both RT and 220 °C become closer to each other due to the homogenization of structure and formation of the ultrafine grained structure in both samples. This indicates that the tendency of strength enhancement has a decreasing rate with

<table>
<thead>
<tr>
<th>MDF temperature</th>
<th>Initial material</th>
<th>1 pass</th>
<th>3 passes</th>
<th>6 passes</th>
</tr>
</thead>
<tbody>
<tr>
<td>MDFed at RT</td>
<td>64 μm</td>
<td>5.4 μm</td>
<td>1.4 μm</td>
<td>1 μm</td>
</tr>
<tr>
<td>MDFed at 220 °C</td>
<td>28 μm</td>
<td>4.1 μm</td>
<td>1.2 μm</td>
<td></td>
</tr>
</tbody>
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Fig. 11. Dependency of pressing stress and imposed strain during the first pass of MDF process.
number of MDF passes. In addition, the difference between yield and ultimate strengths of the sixth pass specimens is reduced, regardless of the processing temperature. The results show that yield strength of the annealed pure titanium is enhanced about 2.7 times. Also, the ultimate strength of titanium is improved about ~2 times for the aforementioned condition. It is found that tensile yield strength (TYS) is enhanced with higher rate as compared to the ultimate tensile strength (UTS). This implies that tensile work hardening capacity \(H_T\) is reduced according to the following relation due to the grain refinement during the MDF process [48].

\[
H_T^e = \frac{UTS - TYS}{TYS} = \frac{UTS}{TYS} - 1
\]  

(7)

According to the previous studies, the ultimate tensile strength of about 980, 950 and 710–800 MPa and tensile yield strength of about 800, 790 and 600–650 MPa were obtained after the final pass of ARB, HPT and ECAP processes, respectively [25,28,47,49–51]. On the other hand, the maximum UTS and TYS of about 811 and 620 MPa were achieved after six MDF passes in current research, respectively. Therefore, it can be deduced that tensile strength of UFG CP-Ti processed by MDF in this research was approximately in the same range of UFG CP-Ti produced by other SPD processes.

Modified Hall-Petch relation shows that LAGBs and HAGBs have significant contribution to the strengthening of materials [52,53]. The strengthening by LAGBs is also related to dislocation strengthening (Taylor equation) [52]. It can be found that the contribution of LAGBs to material strengthening is increased by increasing the volume fraction and/or misorientation angle of LAGBs (up to 15°), and by simultaneous decreasing the space between boundaries. Furthermore, the contribution to strengthening due to HAGBs is increased by increasing the volume fraction of HAGBs and decreasing the space between boundaries [52,53]. Additionally, the contribution of twins in strengthening by fragmentation of grains is also considerable. The twin’s boundaries misorientation angles are high, and can act as grain boundaries; hence, twins can reduce the grain size of material by production of excess boundaries [21,28]. Summarily, it can be concluded that the main strengthening contributions of pure titanium processed by MDF process are related to increase in volume fraction of both HAGBs and LAGBs and decrease in the space between boundaries.

In contrast to the strength, there is a considerable decrease in

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Fig. 12. Engineering tensile stress-strain curves of pure titanium MDFed at (a) RT and (b) 220 °C. Evolution of (c) tensile yield strength and (d) ultimate tensile strength for the various MDF pass numbers calculated from (a) and (b).
the elongation by increasing in the MDF passes up to three passes, while the elongation increases slightly after the third pass of MDF, regardless of the processing temperature. The reduction of ductility in initial passes of MDF can be mainly attributed to the work hardening. On the other hand, the reason of slight enhancement of ductility in subsequent passes of MDF may lies in activation of CDRX for the formation of UFG structure [16,25]. Also according to Fig. 12(a and b), it is obvious that the elongation of samples MDF processed at 220 °C is reasonably higher than that of processed at RT.

3.2.2. Shear properties

Shear strength tests were carried out on the pure titanium before and after the MDF process up to six passes at both ambient and high temperatures, and the results are presented in Fig. 13. Accordingly, application of MDF process and addition of number of passes lead to the improvement of shear strength of pure titanium. Also, the curve of flow shear strength and its work-hardening is similar to the tensile strength. It is observed that shear yield and ultimate shear strengths (SYS and USS) of deformed titanium at RT are higher than those of the processed at 220 °C. Moreover, improvement of shear strength compared to tensile strength was less significant for samples after higher number of MDF passes. It can be found that the shear yield strength of annealed pure titanium after six passes of MDF process at RT and 220 °C is increased by about 134% and 116%, respectively. The higher shear yield strength of pure titanium at RT is mainly related to its finer grains as compared to the higher temperature sample. Additionally, about 52% and 45% improvement in the ultimate shear strength of the annealed pure titanium is observed by employing six passes of MDF at RT and 220 °C, respectively. Interestingly, the values of shear yield and ultimate shear strengths become closer to each other by increasing of the number of MDF passes, similarly to tensile strengths. It can be explained by the decrease of the grain size difference between samples processed at RT and 220 °C with increasing pass number. According to the above findings, SYS is increased further as compared to the USS (the increment percentage ratio is about 2.6 times for the final pass of deformed titanium at both temperatures). This condition suggests that the yielding caused by shear stress is more delayed in comparison with instability at shear in the MDF processed pure titanium, leading to the decrease of shear work-hardening capacity; see Eq. (8) [48]. Reduction of shear work-hardening capacity like tensile work-hardening capacity is caused by the grain refinement through the MDF process. If the grain size would be coarser, it can maintain more space for dislocations; hence, shear work-hardening capacity of material will be increased [48].

Fig. 13. Shear stress-normalized displacement curves of MDFed pure titanium at (a) RT and (b) 220 °C and their corresponding (c) shear yield strength and (d) ultimate shear strength for the various MDF pass numbers.
3.2.3. Correlation between shear and tensile strength

Fig. 14 has been represented in order to study the relationship between tensile and shear strengths of pure titanium before and after the MDF process up to six passes at both ambient and 220 °C temperatures. The general relationship between tensile and shear yield strengths, and ultimate tensile and shear strengths is shown in Eq. (9) [54].

\[ \text{TYS, UTS} = m(\text{SYS, USS} - \tau_0) \]  
(9)

In this relationship, \( \tau_0 \) which is an offset parameter could be measured using the x-axis intercept of each tension-shear curve and consequently the influence of punch compliance on SYS and USS could be modified. After subtracting \( \tau_0 \) from the SYS and USS values, the fitted lines pass through the origin of tensile and net shear strengths curves, as shown in Fig. 14. It is well known that \( \tau_0 \) is closely related to the material and design of the shear punch die such as friction, bending and etc. [55]. As illustrated in Fig. 14, the tensile data have a linear relationship with the shear data and coefficient of the equation (m), which depends on the used material, can be calculated by slopes of the trend lines. The von-Mises and Tresca yield criteria related to the state of pure shear stress demonstrate that the value of the m parameters are 1.732 and 2, respectively [54].

As can be observed in Fig. 14, the slope of relation line of tensile and shear yield strengths is about 1.5, while the corresponding slope for the ultimate tensile and shear strengths is about 2. The higher value of regression indicates that the obtained equations of lines are in good agreement with the real points, and they are reliable. The higher amount of ultimate tensile strength to shear ratio compared to that of von-Mises criterion (1.73) can be attributed to factors such as compression, bending and tension of the material in the region between the punch and die. Previous works on the two-phase titanium and two-phase β rich alloy showed that this ratio is about 2.19 and 2.76. Also, some studies reported the ratio range from 1.29 to 2.042 for different alloys. In other efforts, the relation between shear and tensile yield strength was reported in the range from 1.38 to 2.3 for different alloys [27,54–58]. Therefore, the achieved ratios in this work seem to be acceptable. Furthermore, one of the main reasons for reduced amount of ratio between yield strengths as compared to ultimate ones could be the higher reduction in value of shear work-hardening capacity in contrast to the tensile one. For example, the increment percentage ratio of SYS to USS is about 2.57 for the CP-Ti after six MDF passes, while the increment percentage ratio of TYS to UTS is about 1.7 for the CP-Ti. It means that the SYS is further getting close to USS according to Eq. (8); thus, shear work-hardening capacity is further decreased. The higher increase of SYS in comparison with TYS also leads to the slope reduction in curves of yield strengths in comparison with the ultimate ones. Also, USS is increased less than UTS, and as a result the curves slope of UTS-USS is further increased in accordance to USS. Eventually, the calculated slope of all conditions is higher than 1, meaning that yielding and instability in tension more delayed than shear ones. The instability in tension is delayed faster; because, the slope between ultimate strengths is higher.

**Fig. 14.** Dependency of tensile and net shear strengths for the MDFed pure titanium. Tensile and the net shear yield stress curve at (a) RT and (b) 220 °C. Ultimate tensile and the net shear stress curve at (c) RT and (d) 220 °C.
4. Conclusions

This work presents microstructure and mechanical properties of commercial pure titanium after the multi-directional forging process up to six passes at both room and 220 °C temperatures. The main conclusions are as follows:

- EBSD analysis showed that ultrafine grained structure is achieved by means of MDF process, and grain refinement of CP-Ti is intensified by increasing number of passes and decreasing processing temperature. Mean grain size of RT and 220 °C samples after six MDF passes was reduced from 64 μm to 1 μm and 1.2 μm, respectively, as compared to the annealed condition. The difference between the grain size of processed specimens at RT and 220 °C decreased with increasing number of MDF passes. The main reason of grain refinement is the transformation of low angle to high angle grain boundaries, and the grain refinement by twinning.

- Mechanical tests showed that both tensile and shear strengths are improved with increasing number of MDF passes. Yield tensile and shear strengths were improved after six passes of MDF process at the room temperature by the factor of about 2.7 and 2.3 and ultimate tensile and shear strengths were improved by the factor of approximate 2 and 1.5, respectively. The main reason of strength improvement at the initial stage of the process was due to the work-hardening, increment of LAGBs, and twinning, while grain refinement was the essential reason of strength improvement after higher number of MDF passes.

- The achieved tensile and shear testing results demonstrated that the slope of relationship between yield tensile and shear strengths is about 1.5 and between ultimate tensile and shear strengths is about 2, which is coincident with yielding criteria. The reduced magnitude for the ratio of yield strengths as compared to the ultimate strengths can be associated to the further reduction of shear work-hardening capacity in contrast to the tensile one.

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