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Influence of incremental ECAP on the microstructure and tensile behaviour of commercial purity titanium

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Abstract

Severe plastic deformation (SPD) is an effective method for producing ultrafine grained (UFG) structures in metals. UFG materials are characterized by an average grain size of <1 µm and mostly high angle grain boundaries. These materials exhibit exceptional improvements in strength, superplastic behaviour and in some cases enhanced biocompatibility. Among various SPD methods available, equal channel angular pressing (ECAP) is the most effective method for obtaining bulk UFG billets. Lately, the interest is towards industrialization of the ECAP technique to enable processing of very long or continuous billets. Incremental ECAP (I-ECAP) developed at University of Strathclyde, offers such possibility. The present work details the processing of commercial purity titanium (CP-Ti), using I-ECAP process, with the objective of improving its strength characteristics. CP-Ti billets were successfully processed for up to four passes at 300 °C using an I-ECAP die with a channel angle of 90°. Electron backscatter diffraction (EBSD) technique was used to characterize the microstructure after first and fourth pass of the process. Analysis of the first pass sample revealed heterogeneous structure with a mixture of elongated and refined equi-axed grains. Moreover, existence of $\{10\bar{1}2\}$ tensile twinning in the microstructure was also observed. Remarkable refinement was achieved after fourth pass and ultrafine-grain (UFG) structure was successfully achieved. Room temperature tensile tests carried out on unprocessed and UFG material, display the improvement in strength. The yield strength of the processed material was increased from 308 to 671 MPa and the ultimate tensile strength from 549 to 730 MPa. However, strain-hardening ability of the material was greatly reduced because of processing. Consequently, the material suffers loss in ductility, from 31.9% elongation to failure in the unprocessed form to 21.1% in UFG form. Finally, fracture morphology of the unprocessed and processed CP-Ti displays characteristics of ductile failure. It has been shown that I-ECAP is an effective method for improving strength characteristics of CP-Ti.

Keywords: Titanium; UFG structure; Incremental ECAP; Tensile behaviour; EBSD; Fractography

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1. Introduction

Titanium alloy Ti-6Al-4V is widely used in the biomedical industry for its excellent corrosion resistance and mechanical performance [1, 2]. However, research has suggested that alloying elements such as aluminium and vanadium present in the alloy can be toxic in the long term and are therefore undesirable for full bio-integration [3]. Commercially pure Titanium (CP-Ti) has superior biocompatibility and therefore it is an attractive alternative to Ti-6Al-4V. However, due to its lower strength, its usage is restricted in most load bearing implants. An effective solution for improving the mechanical strength and performance of CP-Ti is by grain refinement through severe plastic deformation (SPD).

SPD is an established technology for achieving extreme grain refinement and obtaining ultrafine grain (UFG) structure in metals, thereby significantly improving their mechanical properties [4]. The technology involves generating large plastic strain in material, without significantly changing the sample dimensions. Among the various available SPD techniques, equal channel angular pressing (ECAP) is by far the most widely used technique. It is capable of producing bulk UFG material, large enough for practical applications [5]. The technique involves passing a billet through a die that consists of two channels with equal cross-sections, intersecting at an angle (Φ) and with an optional outer corner angle (ψ), subtended by the curvature at the outer point of intersection between the two channels. As the billet passes through the intersection, it is subjected to simple shear, while retaining the original cross-sectional area. The billet is normally passed multiple times through the die, in order to produce a desired level of plastic strain. The billet is usually also rotated about its longitudinal axis between the passes, creating different ECAP routes [6]. Despite the success of the ECAP process, it is not an ideal option from the commercialization perspective. Its inability to process very long or continuous billets, due to very high force resulting from friction, is the main limitation. The incremental ECAP (I-ECAP) process [7], which has been developed after extensive FE simulations, is a possible solution. The schematic illustration of the process is shown in Fig. 1(a). Here, material pressing and deformation, take place in two separate stages as opposed to ECAP. Separating the pressing and deformation stages reduces friction substantially. This in turn lowers the force required to feed the material; thereby enabling processing of very long or continuous billets.

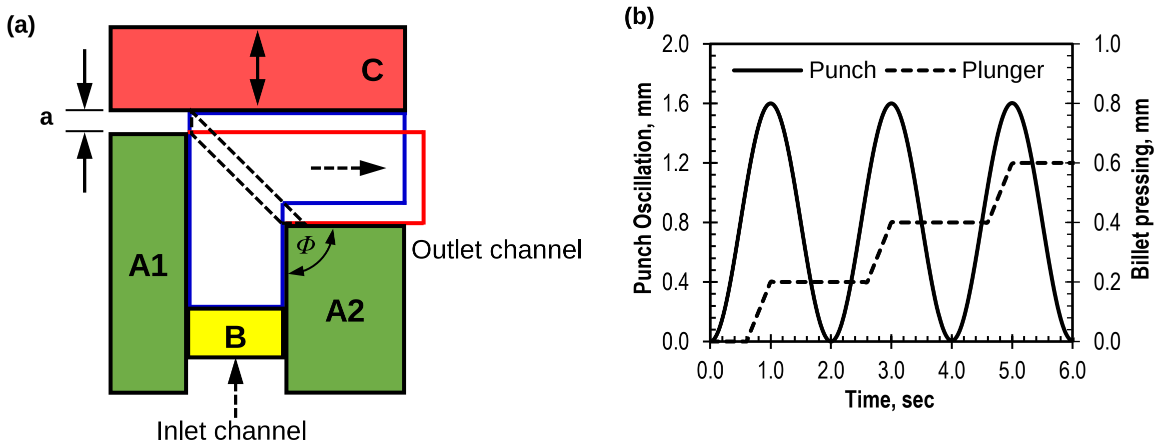

Fig. 1. (a) Schematic illustration of the I-ECAP process (A1 and A2 – Die, B – Plunger and C – Punch) and (b) Relative movement of punch and plunger during I-ECAP.

There are three main tools in I-ECAP; die – A1 A2, plunger – B and punch – C. During processing, the punch is oscillating with a certain frequency and therefore comes cyclically in contact with the billet top surface. In the material pressing stage, while the punch is retracting, the billet material is fed into the deformation zone in increments of 'a' by the plunger, which is known as pressing stroke. In the deformation stage, the punch comes down and deforms the billets. The blue colour outline of the billets in Fig. 1(a) represents the pressing stage whereas the red colour outline represents the deformation stage with the dashed outline representing the plastically deformed zone. The mode of deformation is similar to that in ECAP i.e. simple shear, provided the pressing stroke is not too large. Fig. 1(b) shows the relative movements of punch and plunger for the first three cycles of an I-ECAP process.

The aim of the study is to investigate the microstructural changes and improvement in strength after processing CP-Ti by I-ECAP. Microstructure was characterize after first and fourth pass of I-ECAP and tensile tests were performed to observe the changes in strength and ductility. Fractography of the tensile samples was also performed to investigate the fracture morphology.

2. Material and method

2.1. Material

The material used in the present study was commercial purity titanium, grade 2 (here after referred to as CP-Ti). The material was received in the form of 12.5 mm thick plate from Dynamic Metals Ltd (UK), having an average grain size of ~20 µm. The reported chemical composition (max wt. %) was 0.08% C, 0.03% N, 0.18% O, 0.015% H, 0.20% Fe and balance Ti. Square cross-section billets measuring 10 mm^2 and 120 mm in length were EDM wire-cut from the plate for I-ECAP processing.

2.2. Experimental procedure

In the present study, a double-billet variant of the I-ECAP process was employed using a die with a channel intersection angle (Φ) of 90°. This technique enables processing of two square cross-section billets simultaneously [8]. Experiments were performed at 300 °C on a 1000 kN servo-hydraulic press, at a pressing rate of 0.2 mm/cycle, punch frequency of 0.5 Hz and with a peak to peak punch amplitude of 1.6 mm. Fig. 2(a) and (b) show the I-ECAP press and the tool set, respectively. The die channel configuration led to an imposed equivalent strain of ~1.0 per pass. The billets were processed repeatedly and were subjected to a total of four passes giving a maximum strain of ~4.0. The processing route B_C (90° billet rotation between consecutive passes) was followed, as it is considered to be most effective in achieving homogenous microstructure of grains separated by high angle boundaries [9, 10].

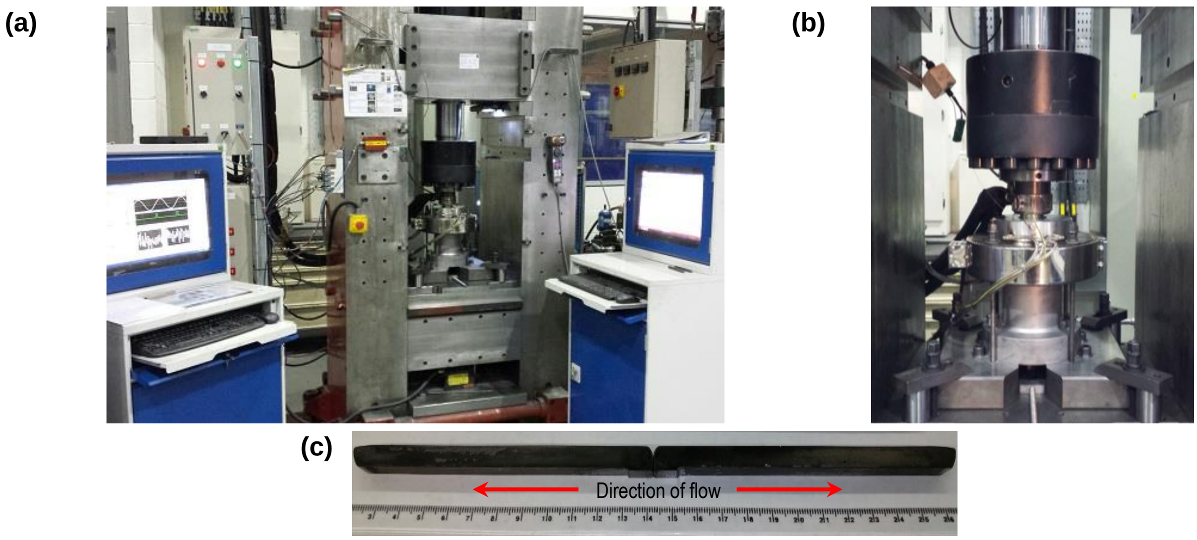

Fig. 2. (a) I-ECAP experimental facility with the 1000 kN servo hydraulic press, (b) I-ECAP tool set and (c) pair of CP-Ti billets after first pass.

2.3. Microstructure characterization and tensile testing

Electron back scatter diffraction (EBSD) was used to characterize the microstructure after the first and fourth pass of I-ECAP, parallel to the flow direction of billets. For characterization purposes, samples were cut from the middle of billet to avoid end effects. The surface of each sample was polished using standard mechanical polishing techniques and was then ion milled using Leica RES101. The SEM was performed on a FEI Inspect F50 with an EDAX TSL

EBSD detector. The sample was tilted 70° from the horizontal plane for EBSD data collection, at a 20 kV accelerating voltage and 200 mA beam current. A step size of 0.40 µm was used for the unprocessed material and 80 nm for the processed material. Analysis of the EBSD data was performed using TSL OIM software.

Tensile tests were carried out at room temperature following the ASTM E8 standard on the Zwick/Roell Z150 testing machine at $0.01s^{-1}$ strain rate. For this purpose, flat tensile samples with a 14 mm gauge length and 3 x 2 mm^2 cross-section were cut parallel to flow plane of the billets using wire EDM. Each test was repeated at least twice to ensure repeatability so the true stress-strain curve obtained represents the average of the tests. Following tensile testing, fracture surface of the samples was examined to study the fracture morphology, using the FEI's Quanta FEG 250 scanning electron microscope (SEM), operating at 20 kV and with secondary electron (SE) mode.

3. Results and discussion.

Fig. 2(c) shows the appearance of billets after the first pass of processing through the I-ECAP die. The process uniformly deformed billets and the macroscopic appearance of the processed billets was smooth, with no visible signs of defects or cracks. The following sections include the discussion on the microstructural aspects of the processed material and its tensile behaviour.

3.1. Microstructure after processing

EBSD technique was used to observe the evolution of grain refinement caused by I-ECAP processing. Fig. 3 shows the inverse pole figure (IPF) maps representing the microstructure along the flow plane of the samples after (a) first and (b) fourth pass of I-ECAP. The I-ECAP die configuration induces a shear strain of ~1.0 per pass, so these IPF maps correspond to a total strain of ~1.0 and ~4.0, respectively. The flow direction (FD) and normal direction (ND) are shown in Fig. 3(a) for the scanned samples. The colour code: red for (0001), green for $(2\bar{1}\bar{1}0)$ and blue for $(10\bar{1}0)$ as shown in the standard stereographic triangle (inset in Fig. 3(a)), corresponds to the crystallographic orientation of each grain. The colour variation within the grains qualitatively represents internal misorientations. Fig. 4(a) and (b) display the corresponding grain size histogram after first and fourth pass, respectively. It is important to highlight again, that the samples for EBSD analysis were taken from the centre region of the processed billets to avoid end defects.

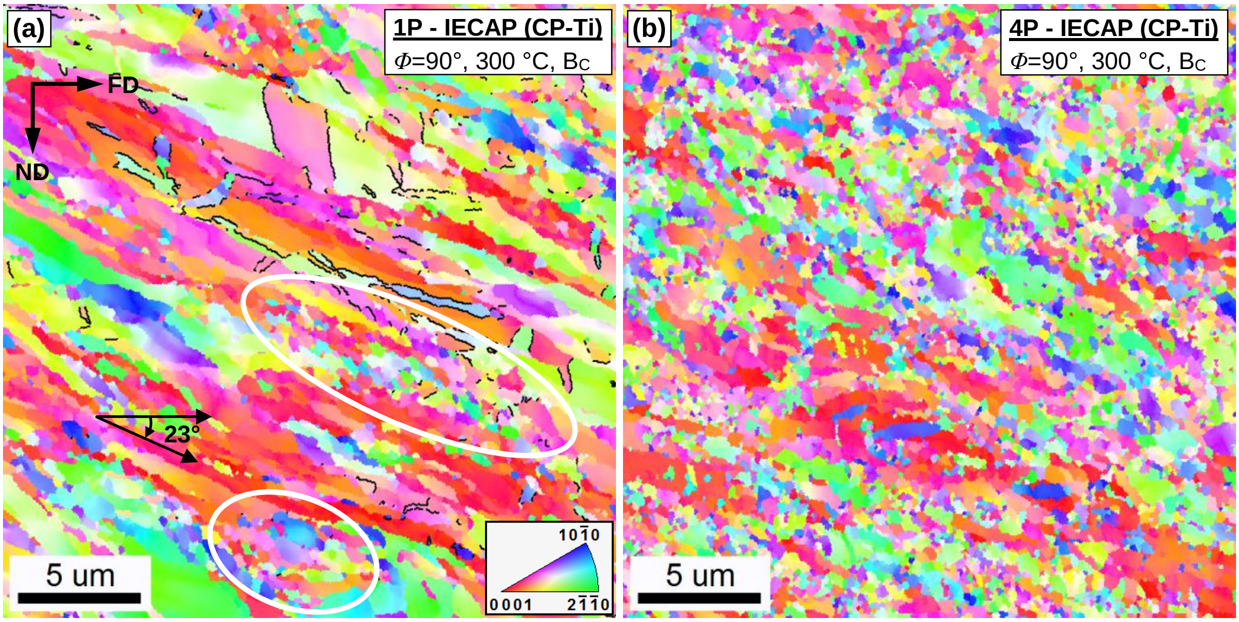

Fig. 3. EBSD based inverse pole figure (IPF) maps after first (a) first and (b) fourth pass of I-ECAP. The black lines in (a) represent the $\{10\bar{1}2\}$ tensile twin boundaries.

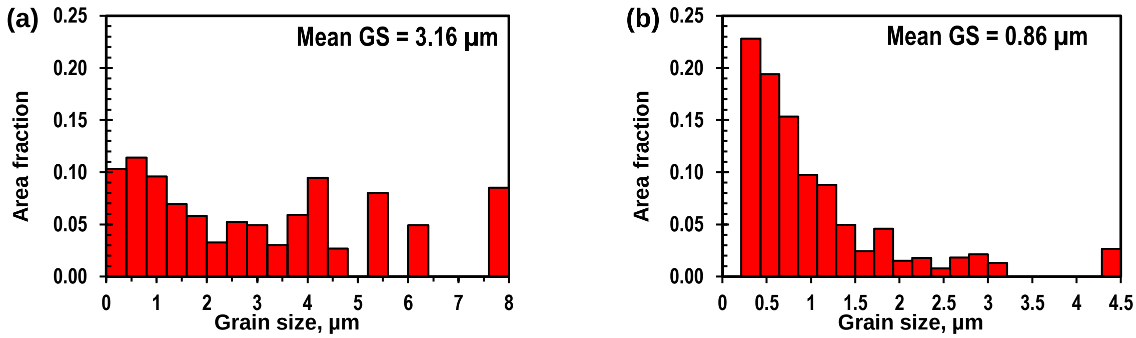

Fig. 4. Corresponding grain size histograms after (a) first pass and (b) fourth pass of I-ECAP.

The IPF map of the sample subjected to first pass of I-ECAP in Fig. 3(a) shows the grain refinement in action. Most of the grains are elongated and forming a banded style microstructure due to shearing process. The tilt of the metal flow is at a 23° angle, which is consistent with first pass processing using ECAP. Large clusters of very fine grains (<1 um) are also seen in the microstructure after first pass (marked with white circles). According to the grain size histogram, these submicron grains occupy ~30% of the total scanned area. Studies have confirmed the presence of twinning during ECAP of CP-Ti and it was reported that twinning plays a key role as a mechanism of grain refinement [11, 12]. Analysis of the microstructure revealed the presence of $\{10\bar{1}2\}$ tensile twins, boundaries of which are shown as black lines in Fig. 3(a). This was also confirmed by the misorientation histograms (not shown here), which showed a peak around 85°, generally associated with $\{10\bar{1}2\}$ twins. The microstructure therefore is heterogeneous with a mixture of twins, elongated and fine grains. The average grain size after first pass is 3.16 µm, a reduction of nearly 85% compared to the unprocessed material. Fig. 3(b) shows the microstructure after four passes of I-ECAP. It is evident that remarkable grain refinement has been achieved and the average grain size is 0.86 µm. The heterogeneity has been completely lost and the microstructure is homogenous with mostly equi-axed grains. The percentage of high angle grain boundaries (misorientation greater than 15°) is 79% of the total grain boundary fraction. The texture strength has also weakened significantly as seen by the random distribution of colours in the IPF map.

3.2. Tensile properties

Fig. 5(a) shows the true stress-strain curves obtained from tensile testing. Overall, it is apparent that the strength has increased considerably due to the formation of UFG structure in the material. In general, four passes of I-ECAP processing led to a significant increase in the values of yield strength (σ_Y) and ultimate tensile strength (σ_{UTS}) in CP-Ti. Table 1 shows the summary of tensile properties for the unprocessed and processed material. For the processed material, the yield strength σ_Y increased from 308 to 671 MPa and the ultimate tensile strength σ_{UTS} increased from 549 to 730 MPa; this corresponds to an increase of 118% and 33%, respectively.

Table 1. Tensile properties of CP-Ti in unprocessed state and after four passes of I-ECAP (0.2% σ_Y — yield strength, σ_{UTS} — ultimate tensile strength, δ_{unif} — uniform elongation, δ_{fail} — elongation to failure, ε_{fail} — true strain at failure and σ_{fail} — true stress at failure)

Material condition	Equivalent strain	0.2% σ_Y (MPa)	σ_{UTS} (MPa)	δ_{unif} (%)	δ_{fail} (%)	ε_{fail}	σ_{fail} (MPa)
Unprocessed (0P)	-	308	549	22.3	31.9	0.28	438
Processed (4P)	~4.0	671	730	8.9	21.1	0.19	543

The unprocessed material however displays much greater strain hardening compared to processed material; consequently, it displays much higher values of ductility. It is interesting to note that the UFG material exhibits higher post necking elongation. In general, elongation to failure of 21.1% after the fourth pass is still considered to be quite reasonable.

Fig. 5(b) and (c) show the morphology of the fractured tensile samples obtained using SEM from the unprocessed and fourth pass material condition, respectively. Both micrographs show that the fracture surface is covered by dimples.

This suggests that in both cases, failure occurred mainly by nucleation and growth of voids and hence, mode of fracture is ductile in nature. The unprocessed material is dominated by large size dimples with some evidence of fine size dimples as well. After four passes of I-ECAP, the dimple size has been reduced drastically and the fracture surface looked smoother compared to the initial coarse-grained material.

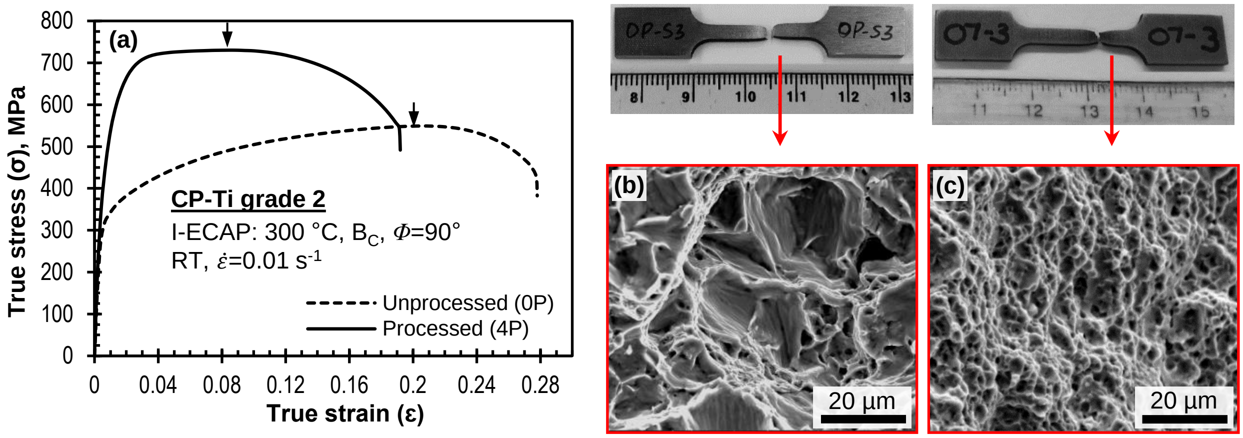

Fig. 5: (a) True stress-strain curves obtained from tensile testing, fracture morphology of (b) unprocessed (0P) and (c) processed (4P) sample.

4. Conclusions

CP-Ti grade 2 billets were processed via double-billet variant of the I-ECAP process, with the aim of improving strength characteristics of the material. The billets were subjected to a total of four passes at 300 °C following route B_C, using the die channel angle of 90°. EBSD analysis of the first pass sample revealed heterogeneous structure with a mixture of elongated and refined equi-axed grains, with some evidence of $\{10\bar{1}2\}$ tensile twins. Ultrafine-grained (UFG) structure was successfully achieved after fourth pass; as-received microstructure was refined from ~20 to ~0.86 µm. UFG CP-Ti exhibited significant improvement in the room temperature tensile strength properties. The yield strength of the material increased by 118% and the ultimate tensile strength increased by 33%. However, the ductility was reduced due to the lack of strain hardening ability exhibited by severely deformed material. Fractography of the tensile samples before and after processing revealed ductile failure.

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