View metadata, citation and similar papers at core.ac.uk

brought to you by CORE

provided by University of Strathclyde Institutional Repository

**Qarni, Muhammad Jawad and Rosochowski, Andrzej and Boczkal, Sonia (2017) Warm deformation behaviour of UFG CP-Titanium produced by I-ECAP. IOP Conference Series: Materials Science and Engineering. pp. 1-6. ISSN 1757-899X (In Press) ,**

This version is available at http://strathprints.strath.ac.uk/60468/

**Strathprints** is designed to allow users to access the research output of the University of Strathclyde. Unless otherwise explicitly stated on the manuscript, Copyright © and Moral Rights for the papers on this site are retained by the individual authors and/or other copyright owners. Please check the manuscript for details of any other licences that may have been applied. You may not engage in further distribution of the material for any profitmaking activities or any commercial gain. You may freely distribute both the url (http://strathprints.strath.ac.uk/) and the content of this paper for research or private study, educational, or not-for-profit purposes without prior permission or charge.

Any correspondence concerning this service should be sent to the Strathprints administrator: strathprints@strath.ac.uk

The Strathprints institutional repository (http://strathprints.strath.ac.uk) is a digital archive of University of Strathclyde research outputs. It has been developed to disseminate open access research outputs, expose data about those outputs, and enable the management and persistent access to Strathclyde's intellectual output.

# Warm deformation behaviour of UFG CP-Titanium produced by I-ECAP

**Muhammad Jawad Qarni[1],*, Andrzej Rosochowski[2] and Sonia Boczkal[3]**

[1]Advanced Forming Research Centre (AFRC), University of Strathclyde, Inchinnan, Renfrewshire, PA4 9LJ, UK

[2]Design Manufacture and Engineering Management (DMEM), University of Strathclyde, Glasgow, G1 1XJ, UK

[3]Institute of Non-Ferrous Metals in Gliwice, Light Metals, Division, ul. Pilsudskiego 19, 32-050 Skawina, Poland

*Corresponding autor: jawad.qarni@strath.ac.uk

**Abstract**. The objective of the present study is to investigate the deformation behaviour of Ultrafine-grain (UFG) commercial purity Titanium (CP-Ti) at warm temperatures. Firstly, CP-Ti billets were processed through six passes of incremental equal channel angular pressing (I-ECAP) at 300° C using a die channel angle ($\Phi$) of 120°. Uniaxial compression tests were then performed under isothermal conditions on cylindrical samples obtained from the UFG CP-Ti billets. A series of these tests were conducted at different temperatures of 400, 500 and 600 °C and at varying strain rates of 0.01, 0.1 and 1.0 $s^{-1}$. In each test, the original height of the sample was deformed by ~50% of its original value. The true stress-strain curves obtained, revealed that the flow stress was sensitive to both temperature and strain rate. In general, the flow stress was higher for lower temperatures and higher strain rates. For tests conducted at 400 and 500 °C, the flow stress quickly reaches a peak value, beyond which it exhibits a steady state response where there is no appreciable change in flow stress with increasing strain. The 600 °C tests however shows a strain hardening behaviour. Microstructure of the sample deformed at 600 °C and 0.01 $s^{-1}$, exhibited significant grain growth.

## 1. Introduction

Owing to its high specific strength, low density, outstanding corrosion resistance and excellent bio-compatibility; Titanium and it's alloys are a material of choice in many aerospace, military, chemical and biomedical applications [1]. Ti-6Al-4V is most widely used for medical device applications such as in total replacement implants, where higher strength characteristics is generally a requirement [2]. The addition of alloying elements such as Al and V in Titanium, significantly enhance the mechanical performance, but these are considered to be toxic and therefore undesirable for full bio-integration [3]. Commercial purity Titanium (CP-Ti) is biologically more compatible but cannot satisfy the higher strength level requirements. An effective solution for improving the mechanical strength and performance of CP-Ti is by grain refinement through severe plastic deformation (SPD) processes. To date, strengthening of various metals including CP-Ti via grain refinement has been demonstrated by numerous studies [4, 5].

**Accepted for publication in IOP Conference Series: Materials Science Engineering
Vol XX, 2017: The 7th International conference on Nanomaterials by Severe Plastic Deformation (NanoSPD 2017) 2 – 7 July 2017, Sydney - Australia

Equal channel angular pressing (ECAP) is by far the most promising SPD technique available. It is capable of producing bulk UFG materials, large enough for practical applications [6]. Developed by Segal et al. [7], the technique involves pressing a billet through a die consisting of two channels with equal cross-sections, intersecting at an angle ($\Phi$). As the billet passes through the intersection, it is subjected to simple shear, while retaining the original cross-sectional area. The same billet can therefore be passed through the die multiple times, in order to impart desired level of plastic strain. The billet is usually rotated along its longitudinal axis between the passes, creating different ECAP routes [8]. ECAP has been widely applied on CP-Ti for the purposes of refining the grain structure and subsequently improving its strength. Research has shown that grain refinement in CP-Ti, not only improves its yield and tensile strength [9-12] but also improves fatigue behaviour and corrosion resistance considerably [13, 14].

CP-Ti has a hexagonal closed packed (HCP) structure at room temperature. Compared to face-centred cube (FCC) and body-centred cube (BCC) metals, it is regarded as ‘hard-to-deform’ because of its poor formability. The inferior deformability, is mainly due to the limited slip systems available in HCP structure. In order to improve the formability in CP-Ti, it is necessary to carry deformation at elevated temperatures [15]. However, use of excessively high temperatures can cause increased recovery, leading to significant grain growth. This will result in considerable loss of hardness and strength, especially if the deformation process is carried at very slow strain rates. Deforming CP-Ti in the warm temperature range is therefore desirable, not only in terms of retaining the strength characteristics of UFG but also in terms of reducing the forming process energy requirements. Finding a good balance between formability and loss in strength is essential by developing a deeper understanding into the warm temperature range deformation behaviour of UFG CP-Ti. It is generally accepted that compression flow stress data can be used to simulate the deformation behaviour during metal forming processes. The effective use of accurate flow stress data in simulations combined with the knowledge of grain growth at different temperatures and strain rates, will greatly aid in establishing the optimal processing regimes of UFG CP-Ti.

The present study is therefore concerned with studying the warm temperature deformation behaviour and subsequent grain growth in UFG CP-Ti during compression. Firstly, the incremental equal channel angular pressing (I-ECAP) process, which is a recognized SPD processing technique [16] was employed to obtained UFG structure in CP-Ti. Uniaxial compression tests were then performed on cylindrical samples obtained from the processed billets. To obtain a comprehensive flow stress dataset, a series of these tests were conducted for a temperature range of 400 to 600 °C and for varying strain rates. Moreover, by using the electron backscatter diffraction (EBSD) technique, the microstructure was analysed to observe grain growth following compression tests.

## 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. Billets were cut such that the longitudinal axis of the billet was parallel to the rolling direction of the plate.

### *2.2. I-ECAP experimental procedure*

Despite the success of ECAP process, it is not an ideal option from the viewpoint of commercialization. Its inability to process very long or continuous billets, due to very high contact pressure, is the main limitation. I-ECAP process developed by Rosochowski and Olejnik [17], is a possible solution. The schematic illustration of the process is shown in Figure 1(a). Here, material pressing and deformation, takes place in two separate stages as opposed to ECAP [18]. By separating the pressing and deformation stages, reduces friction substantially. This is turn lowers the force required to press the material; thereby enabling processing of very long or continuous billets.

There are three main tools in I-ECAP: (1) die, (2) plunger and (3) punch; denoted by A, B and C respectively. During processing, punch tool (a working die) is oscillating with a certain frequency and therefore comes cyclically in contact with the billet top surface. In material pressing stage, while the punch tool is retracting, the billet material is pressed into the deformation zone in increments of 'a' by the plunger tool known as pressing stroke (see Figure 1(a)). In the deformation stage, the punch comes down and deforms the billets. The blue colour outline of the billets in Figure 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.

In the present study, the double-billet variant of the I-ECAP process, with a channel intersection angle ($\Phi$) of 120° was used. This technique enables processing two square cross-section billets simultaneously, the two billets are marked 1 and 2 in Figure 1(a). A detailed description of the I-ECAP apparatus used in the study can be found elsewhere [19].Experiments were performed at 300 °C, on a 1000 KN servo-hydraulic press shown in Figure 1(b), at a pressing rate of 0.2 mm/cycle (0.1 mm/s), punch oscillatory movement of 0.5 Hz and with a peak to peak amplitude of 1.6 mm. The die configuration lead to an imposed strain of ~0.67 per pass through the die. The billets were processed repeatedly and were subjected to six passes, leading to an induced total strain of ~4.02. Processing route $B_C$ was followed, as it is considered to be the most effective in achieving homogenous microstructure of grains separated by high angle boundaries [20, 21].

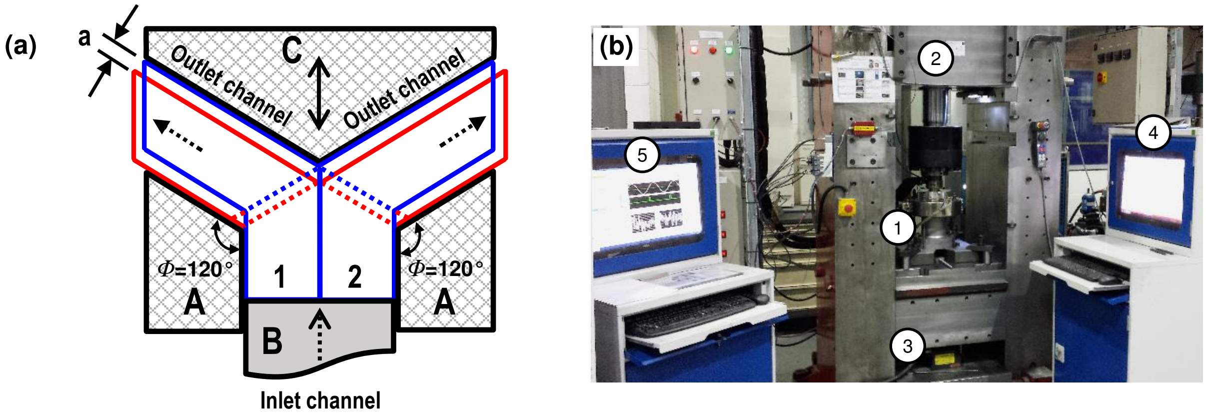


**Figure 1.** (a) Schematic illustration of double-billet variant of I-ECAP process (A=Die, B=Plunger and C=Punch) and (b) the I-ECAP experimental rig (1=I-ECAP die, 2=1000 KN servo-hydraulic press, 3=servo motor coupled to screw jack for material pressing, 4=Zwick Controller for controlling the press and 5=LabVIEW application for data acquisition)

### *2.3. Compression testing*

For investigating the warm deformation behaviour of CP-Ti, uniaxial compression tests were performed following the ASTM E209-00 on the Zwick/Roell HA250 servo hydraulic testing machine with a 250 kN load cell. Compression samples, measuring ∅8 mm in diameter and 8 mm in height, were precision turned on a lathe machine, from the sixth pass processed billets such that the compression axis was parallel to longitudinal axis of the billet. To avoid end effects, samples were taken from the central region only, after cutting away the head and tail region of the billets.

Compressions tests were conducted at constant strain rate and under isothermal conditions. A series of these tests were conducted, at three deformation temperatures of 400, 500 and 600 °C and at three different strain rates of 0.01, 0.1 and 1.0 $s^{-1}$. A minimum of two tests were performed at each test conditions to ensure repeatability of results. Temperature was monitored using two calibrated N-Type thermocouples, which was located near the deformation zone. When the temperature value was stable by ±2 °C, compression test was initiated and the height of cylindrical sample was deformed by ~50% in a single loading step, achieving a true strain of ~0.80. After the test, each sample was immediately quenched in cold water to arrest the microstructure and to stop any grain growth.

### *2.4. Microstructure*

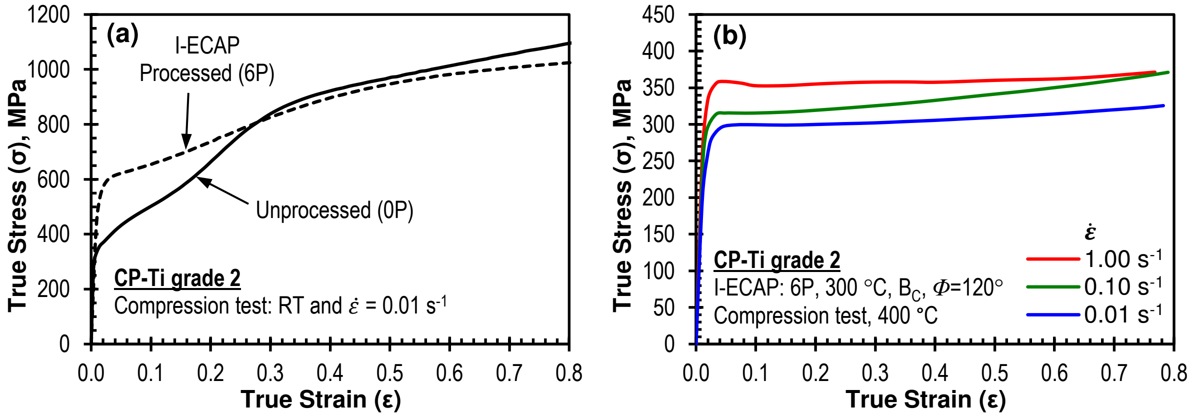
The deformed compression samples were cut along the compression axis from the centre. The surface of each sample was polished using standard mechanical polishing techniques and was then ion milled on Leica RES101 as a final step. The microstructure was characterized by scanning electron microscopy (SEM) using electron backscatter diffraction (EBSD) technique. The SEM used was a FEI Inspect F50 with an EDAX TSL EBSD detector. The sample was tilted 70° from the horizontal for EBSD data collection, at a 20 kV accelerating voltage, 200 mA beam current and with a 80 nm step size.

## 3. Results and discussion

### *3.1. Flow stress behaviour*

Figure 2, shows the true stress and true strain curves obtained from the compression testing, representing the flow stress and deformation behaviour of CP-Ti studied in the present work at various temperature and strain rate conditions. Room temperature compression tests at 0.01s$^{-1}$ were also performed on unprocessed (0P) and for severely deformed (6P) material, to observe the increase in strength and strain hardening behaviour following I-ECAP processing. Figure 2(a) shows this comparison, here the solid line represents the processed material displays a much higher yield strength compared to the unprocessed material. The results for the unprocessed material is consistent with the findings by Gray [22]. He concluded that the rate of strain hardening increases with increase in grain size due to pronounced increase in deformation twinning.

Figure 2(b-d), shows the flow stress behaviour of UFG CP-Ti at warm deformations temperatures of (b) 400, (c) 500 and (d) 600 °C. As expected, the flow stress increases with decreasing temperature and increasing strain rate. At same temperature, the flow stress decreases with decreasing strain rate. It is evident from these graphs that compared to the room temperature result, the flow stress at warm temperatures for all strain rates is considerably lower. These figures also displays low levels of strain hardening behaviour of UFG CP-Ti at warm deformation temperatures. For test temperatures of 400 and 500 °C, the flow stress quickly reaches a peak value near ~0.02 true strain, beyond which it exhibits a steady state response and where there is no appreciable strain hardening. Except for the test conducted at 500 °C and 0.10 s$^{-1}$, which actually displays a flow softening behaviour. Flow stress obtained at 600 °C for all strain rates, shows slightly increasing levels of strain hardening, compared to at 400 and 500 °C. However, here the flow stress has been dropped by half compared to 500 °C.



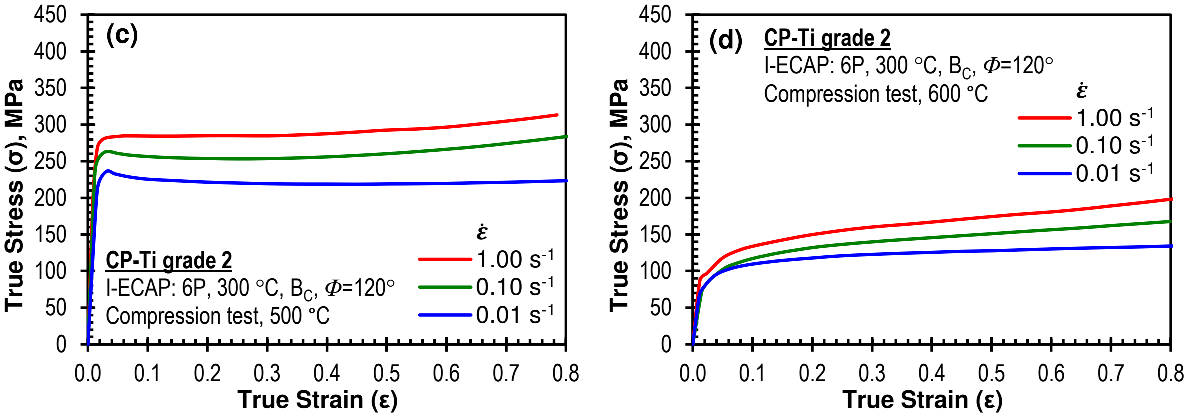


**Figure 2.** Representative true stress-true strain curves obtained from uniaxial compression testing of CP-Ti at (a) room temperature testing of both unprocessed (0P) and processed material (6P) and in the warm temperature range testing of processed material (6P) at (b) 400 °C (c) 500 °C and (d) 600 °C

### 3.2. Deformed microstructure

Figure 3(a and b) shows the colour inverse pole figure (IPF) maps obtained from the SEM based EBSD analysis, shows the microstructure after 50% compression at (a) 400 °C and 1.0 s$^{-1}$ and (b) 600 °C and 0.01 s$^{-1}$. These two extremes conditions have been selected to shows the extent of grain growth. The average grain size shown in Figure 3(a) is 0.85 μm, and it dramatically increases to ~4.0 μm as shown in Figure 3(b). The IPF map in Figure 3(b), also shows the presence of some fraction of twins within the matrix of large grains. Analysis of the grain misorientation histogram (not shown here), shows a noticeable peak around 65° and a significant peak at 85°, this strongly suggests existence of $\{11\bar{2}2\}$ and $\{10\bar{1}2\}$ twins.

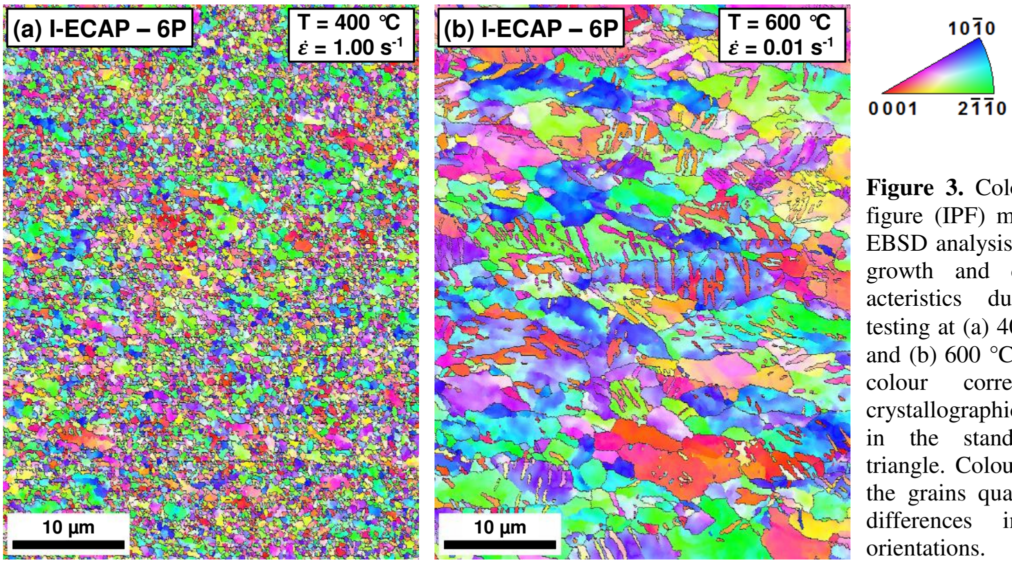


**Figure 3.** Coloured inverse pole figure (IPF) maps obtained from EBSD analysis showing the grain growth and deformation characteristics during compression testing at (a) 400 °C, $\dot{\varepsilon}$ = 1.00 s$^{-1}$ and (b) 600 °C, $\dot{\varepsilon}$ = 0.01 s$^{-1}$. The colour corresponds to the crystallographic orientation shown in the standard stereographic triangle. Colour variations within the grains qualitatively represent differences in internal mis-orientations.

## 4. Conclusion

The following conclusions are drawn from the study:

1. CP-Ti grade 2 billets were severely deformed to achieve ultrafine grain (UFG) structure, after six passes of I-ECAP processing at 300 °C using a die angle of 120° following route B$_C$.
2. Room temperature compression tests of unprocessed and I-ECAP processed material, revealed significant increase in strength due to processing. However, the unprocessed material exhibited higher levels of strain hardening compared to processed material.

3. Compression testing of UFG CP-Ti at warm temperatures of 400, 500 and 600 °C and at varying strain rates of 0.01, 0.1 and 1.0 $s^{-1}$, revealed that the flow stress was sensitive to temperature and strain rate. The flow stress increases with decreasing temperatures and increasing strain rates.
4. In compressions tests conducted at 400 and 500 °C, the flow stress quickly reaches a peak value, beyond which it exhibits a steady state response where there is no appreciable change in flow stress with increasing strain. The 600 °C tests however shows a strain hardening behaviour.
5. Microstructural observations using EBSD, exposed significant grain growth in the sample deformed at 600 °C and 0.01 $s^{-1}$. This also led to a considerable drop in flow stress during compression.

**References**

[1] Donachie MJ 2000 Titanium - A technical guide Second edition ASM International ISBN: 0-87170-686-5
[2] Geetha M, Singh AK, Asokamani R and Gogia AK 2009 *Progress in Materials Science* **54** 397-425
[3] Lugowski SJ, Smith DC, McHugh AD and Van Loon JC 1991 *Journal of Biomedical Materials Research* **25** 1443-1458
[4] Meyers MA, Mishra A and Benson DJ 2006 *Progress in Materials Science* **51** 427-556
[5] Langdon TG 2013 *Acta Materialia* **61** 7035-7059
[6] Segal VM, Dobatkin SV and Valiev R. Z. 2004 *Russian Metall* **1** 1-102
[7] Segal VM, Reznikov VI, Drobyshevskiy AE and Kopylov VI 1981 *Russ Metally* **1** 99-104
[8] Iwahashi Y, Wang J, Horita Z, Nemoto M and Langdon TG 1996 *Scripta Materialia* **35** 143-146
[9] Purcek G, Yapici GG, Karaman I and Maier HJ 2011 *Materials Science and Engineering: A* **528** 2303-2308
[10] Sergueeva AV, Stolyarov VV, Valiev RZ and Mukherjee AK 2001 *Scripta Materialia* **45** 747-752
[11] Zhao X, Yang X, Liu X, Wang CT, Huang Y and Langdon TG 2014 *Materials Science and Engineering: A* **607** 482-489
[12] Stolyarov VV, Zhu YT, Alexandrov IV, Lowe TC and Valiev RZ 2003 *Materials Science and Engineering: A* **343** 43-50
[13] Balyanov A, Kutnyakova J, Amirkhanova NA, Stolyarov VV, Valiev RZ, Liao XZ, Zhao YH, Jiang YB, Xu HF, Lowe TC and Zhu YT 2004 *Scripta Materialia* **51** 225-229
[14] Semenova IP, Salimgareeva GK, Latysh VV, Lowe T and Valiev RZ 2009 *Materials Science and Engineering: A* **503** 92-95
[15] Semiatin SL, DeLo DP, Segal VM, Goforth RE and Frey ND 1999 *Metallurgical and Materials Transactions A* **30** 1425-1435
[16] Azushima A, Kopp R, Korhonen A, Yang DY, Micari F, Lahoti GD, Groche P, Yanagimoto J, Tsuji N, Rosochowski A and Yanagida A 2008 *CIRP Annals - Manufacturing Technology* **57** 716-735
[17] Rosochowski A and Olejnik L 2007 *AIP Conference Proceedings* **907** 653-658
[18] Rosochowski A and Olejnik L 2011 *Materials Science Forum* **674** 19-28
[19] Qarni MJ, Sivaswamy G, Rosochowski A and Boczkal S 2017 *Materials & Design* **122** 385-402
[20] Oh-Ishi K, Horita Z, Nemoto M, Furukawa M and Langdon TG 1998 *Metallurgical and Materials Transactions A* **29** 2011-2013
[21] Stolyarov VV, Zhu YT, Alexandrov IV, Lowe TC and Valiev RZ 2001 *Materials Science and Engineering: A* **299** 59-67
[22] Gray G, T. 1997 *Le Journal de Physique IV* **07** 423-428