The effect of severe plastic deformation on the microstructure and mechanical properties of (TiB+TiC) reinforced titanium matrix composites

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ABSTRACT. Equal-channel angular pressing (ECAP) was successfully applied to in-situ synthesized particulate reinforced titanium matrix composites. The results prove that the ultrafine-grained structure can be effectively obtained in the titanium matrix composite by ECAP at different temperatures and passes. Formation of homogenous TiB and TiC particles with ultrafine grains and sub-grains were obtained after 4 ECAP passes, which indicates that the reinforcement-induced ultrafine grains. The lamellar α phases are also twisted obviously due to the shear stress, which can reduce the average thickness of lamellar α phases to less than 1μm. After ECAP at relatively low temperature, dislocation pile-ups, dislocation tangling and plenty of cell structures with size of about 500nm can be observed in the matrix. While many newly formed ultrafine grains with clear grain boundaries and size of 100~500 nanometers have formed. The tensile test results show that the yield strength is improved by ECAP pressing and saturate after 4 ECAP passes to a value of 1150MPa, and the ductility is much higher compared with the first ECAP pass.

Keywords: Titanium Matrix Composite, Microstructure, Severe Plastic Deformation, Mechanical Property

1. Introduction
There is considerable current interest in controlling of materials structures by severe plastic deformation (SPD) [1-4]. An important attractive point of this method is grain refinement to the sub-micro scale, or even the nanometer scale that can be attained in bulk billets, in a cost effective manner and for different metal and alloys [2]. Among a few known methods of SPD, equal channel angular pressing (ECAP) is presently considered as the most popular approach for industrial applications [3,4]. As high strains are known to introduce larges misorientations within subdivided grains, it has been expected that multi-pass ECAP should lead to ultra-fine grained structures, usually in the range from a few microns to 0.2 micro, provide a reasonable compromise between high strength and satisfactory ductility that is especially attractive for industrial applications. However, realization of ECAP still remains imperfective [5, 6]. Despite of extensive activity in the ECAP, majority of papers published in the journal. This approach was restricted on hard deformed materials. It is difficult to use the method in titanium alloys and still there are few reports on the application of this method to particulate reinforced titanium matrix composites [7, 8].
The incorporation of high strength and high stiffness ceramic reinforcements can improve dramatically mechanical performances of particulate reinforced titanium matrix composites [9], which are currently being widely used in aerospace, commercial automotive engineering, and military applications [10]. Meantime, this material exhibits poor plasticity due to the microstructural heterogeneity. The plasticity is related to the metal matrix behaviour, and the damage is generally due to the particle breaking and interfacial debonding between the metal matrix and particles [11, 12]. Achieving good grain size, particle size, particle distribution and interface bonding is vital if the composites experience severe plastic deformation. It can eliminate the agglomeration phenomenon, and stronger adhesion at the particle interface improves load transfer, further increasing the yield strength and plasticity [13].

Our studies were devoted to developing a process for fabricating titanium matrix composite bars with ultrafine grain structure. The aims focused on the changes in the microstructure and mechanical properties of deformed composites in the process of their production by severe plastic deformation and subsequent heat treatment. Further investigation of the influence of deformation degree applied on the morphology and distribution of TiB short fibers and TiC particles, and to evaluate the deep processing and property improvement of particulate reinforced titanium matrix composite.

2. Experimental material and procedures
(TiB +TiC)/Ti6Al4V titanium matrix composite billet with a diameter of 25mm and length of 1000mm was preliminarily annealed at 600°C for 30min and air cooled to room temperature. Then the billet was cut into specimens of 10mm × 10mm × 140mm for equal channel angular pressing (ECAP) on a die-set with the channels’ intersection angle of φ=120° and at a temperature of 550°C, while the subsequent extrusion temperature was 800°C. Route Bc is turned by 90° about its longitudinal axis after every pass, and number of passes n=4. After every ECAP pass, the specimen should be removed quickly from the dies and water quenched to keep the high temperature microstructure. Fig.1 shows the schematic illustration of the ECAP process. The appearance of the extruded specimens in intermediate stages is presented in Fig.2.

![Fig.1 Schematic diagram of ECAP process](image1.png)

![Fig.2 Appearance of extruded specimens](image2.png)

The microstructure was studied by using optical microscopy, scanning electron microscopy and transmission electron microscopes (TEM). All observed surfaces were parallel to the extrusion axis. Tensile tests were performed at room temperature in air using Zwick Z100/SN3A. Flat samples with a gage length of 18 mm and a cross-section of 3 mm × 1.6 mm were cut by wire Electrical Discharge Machining (EDM) from the titanium matrix composite billets. A strain rate of 10⁻³ mm/s was used for the all tests.

3. Another section of your paper

3.1. Original microstructure
The microstructure of the as-received material, which had been produced via hot forging followed by annealing, was observed to be equiaxed structure with a volume fraction of approximately 85% equiaxed α phase (Fig.3). On closer examination, it was found that α phases is about ~10μm in diameter. Additionally, SEM analysis of the surface of the material after mechanical polishing revealed that the material had a distribution of reinforcements in the matrix. TiB fibers and TiC particles in these image are platelet and spherical in shape, respectively. All reinforcements are homogenously distributed. In quantitative analysis, there are about 10% long TiB fibers and large TiC particles. TiB fibers are about 20 mm in length and width of ~5 mm, TiC particles are about 15 mm in diameter with an irregular shape, suggesting that the reinforcements are long and large distributed in the metal matrix.

Fig.3 SEM image of original forged samples

3.2. Microstructure of the composite processed by ECAP

Fig.4 shows the appearance of the billets after pressing through one and four passes of ECAP at the same pressing speeds, which showed that the surface of the titanium matrix composite samples were smooth and there was some small metal flash after pressing four passes.

Fig.4 Appearance of the ECAP extruded billet and corresponding microstructure

Nevertheless, close inspection of the sample processed by four passes revealed the presence of cracking in this sample, and the microstructure of the samples processed by four passes showed that the reinforcements dispersed in a homogeneous manner in the composite. After one pass of ECAP, the matrix exhibited a near-fully equiaxed microstructure, and the α phases were elongated in the direction of extruding with average grain size of about 20mm in length, suggesting that the α phases were elongated to be platelet in shape. Meanwhile, TiB fibers in titanium matrix composite showed good alignment along the extrusion direction, and the TiC large particles disperse in the specimens with
diameter of about 14 mm. As the ECAP pass number increased, more homogenous structure was
formed after four pass of ECAP. Elongated α phases were refined and rearranged significantly in
the metal matrix, with the estimated grain size of about ~14 mm in diameter, which was a little larger
than the original microstructure due to the grain growth at a relative high extruding temperature. The TiB
fibers and TiC particle were smaller and distributed more homogenous comparing with the one pass of
ECAP.

At higher magnification, SEM micrographs in Fig. 5(a) and Fig. 5(b) show the microscopic structure
of (TiB+TiC)/Ti6Al4V titanium matrix composite in ECAP billets after one pass and four passes,
respectively. After ECAP, the initial TiC large particles and long TiB fibers that are severely
fragmented and agglomerated TiC have been observed. Distinct α grains were still visible. However,
all the grains were highly elongated, as seen in Fig. 4. The evolution of the grain size was determined
by quantitative metallographic methods and it varied around 20 μm at the beginning ECAP process,
and around 14 μm at the end of ECAP process, i.e. after four passes of ECAP. The evolution of the
reinforcements were investigated by measuring the aspect ratio and particles length by using image
analysis software. It was found that the aspect ratio of TiB fibers and TiC particles in all specimens
decreased with the number of ECAP passes increasing. Simultaneously, the length of TiB fibers
decreased heavily when the billets were deformed by ECAP, which was around 12 μm in length at the
last of ECAP process. The size of TiC particles were also reduced with the increase of ECAP numbers,
refined and near-spherical TiC particles were obtained at the end of the ECAP process.

![Fig.5 The evolution of the reinforcements of the ECAP extruded composite](image)

(a) one pass of ECAP, (b) four passes of ECAP

3.3. Mechanical properties

Mechanical properties of ECAP extruded (TiB+TiC)/Ti6Al4V composite billets were investigated in
the tensile tests at room temperature. The deformation curves after one pass, two passes and four
passes of ECAP were plotted as the strain versus the strength (Fig. 6).

![Fig.6 Changes of tensile properties of ECAPed (TiB+TiC)/Ti6Al4V composite billets](image)
The increase of the ductility was evident, and the ductility increased with the numbers of passes through the 120° ECAP dies. The reason of the improvement was attribute to the refinement of grains after four ECAP passes. In regarded to the tensile strength of the examined samples, it was about 1160 MPa after one pass of ECAP, which decreased slightly to about 1150 MPa after two and four passes. The decrease of strength and the increase of ductility could be the interaction of refinement and working hardening. It can be concluded that equal channel angular pressing provides an effective procedure for improving the ductility of particulate titanium matrix composite. The ECAPed (TiB+TiC)/Ti6Al4V composite billets after the second and the fourth pass exhibited higher elongation but similar strength compared with the sample ECAP processed after the first pass.

4. Discussion

The experimental results show that the mechanical properties of TiB+TiC/Ti6Al4V composite depend essentially on its microstructural state, including the small grain size attained after ECAP and the homogeneity of the reinforcements. Since ECAP processing involves the rapid imposition of an exceptionally high strain, the initial microstructural change associated with ECAP is the introduction of very high density of dislocations, and the high pressure involved in the pressing operation may lead to the breaking and fragmentation of internal reinforcements. The observed ductility increase of the four passes of ECAP, in comparison with the one pass of ECAP, is caused by the smaller grain size, and also by the presence of the optimized reinforcements in the matrix. Such changes in microstructure of the composite lead to a vibration of the multiplication, annihilation and reversion of dislocations.

Fig. 7 shows the TEM micrographs of (TiB+TiC)/Ti6Al4V composite billets. According to the equivalent strain accumulating formula, the values of accumulating strain increases with the increasing of the ECAP pass numbers, which is better to the refinement of grain size and reinforcements, even improving the mechanical properties. However, that does not accord with the strength results. The strength has a slight decrease with the increase ECAP pass numbers. It is observed that the highest strength obtained in the first ECAP pass, and high dislocations are in a high density and tangling together in the matrix (Fig.7a). Some stacking faults are found in the long TiB fibers. As known the low value of dislocation density in the composite before ECAP, it could be known that the dislocation multiplication, which results in dislocation density increasing rapidly in ECAP. As a result, the velocity value of dislocation annihilation is lower than that of dislocation multiplication, and the reversion of dislocations is not evident. Therefore, the strength performs the best in the first ECAP pass number.

Fig.7 TEM micrographs of TiB+TiC/Ti6Al4V composite billets after (a) one pass of ECAP, (b) four passes of ECAP

Strength characteristics of the titanium matrix composites, measured after two and four passes of ECAP, practically coincide, and their values are slight lower than that for one pass of ECAP. The deformation strain increases to a high value, the dislocation multiplication and annihilation attain a dynamic equilibrium. Therefore, the strength has a slight changes with the increase of ECAP pass numbers. Tendency towards increase of ductility was also observed, which can be associated with accumulation of high dislocation density in an ultrafine grain (~200nm) and presence of a large
number of low-angle subgrain boundaries (Fig. 7b), which is explained by retention of grain and subgrain sizes. The dislocation in grains migrated to grain boundaries and the interface of TiB and TiC reinforcements. Dislocation density among grain boundaries reached a high value, which formed dislocation cell or wall and then gradually formed subgrain structure. It should be noted that there is a tendency towards enhancement of ductility after four passes. This can be explained with increase of a fraction of high-angle boundaries in the ultrafine grains structure, which are capable of grain boundary sliding during plastic deformation.

5. Conclusions
(1) During ECAP processing, bands of elongated α phases are refined and rearranged significantly. The TiB fibers and TiC particles are smaller and distributed more homogenous in the metal matrix. Ultrafine grains, with relatively homogeneous reinforcement and microstructure, in grain size of about 200nm, can be obtained after four passes of ECAP.
(2) The strength of ECAPed (TiB+TiC)/Ti6Al4V composite billets are closely related to the number of pass. As the following ECAP passes are applied, the value of increase becomes smaller. The enhancement of ductility is dominated by the decreasing of reinforcements’ size, and the homogeneity of the reinforcements’ distribution through four passes.
(3) With the increasing of the ECAP pass numbers, the small size reinforcements tend to acquire high dislocation density surrounding the TiB fibers and TiC particles, which forms dislocation cell or wall and then gradually forms ultrafine grain structure.

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References
[1] Valiev R 2004 Nature Materials. 3 511-6.
[2] Shankar M R, Rao B C, Lee S, Chandrasekar S, King A H and Compton W D 2006 Acta. Mater 54 3691-700.
[3] Azushima A, Kopp R, Korhonen A, Yang D Y, Micari F, Lahoti G D, Groche P, Yanagimoto J, Tsuji N, Rosochowski A and Yanagida A 2008 Circ. Ann. Manuf. Techn 57 716-35.
[4] Valiev R Z, Estrin Y, Horita Z and Langdon T G 2006 JOM 58 33-9.
[5] Zhao X, Yang X, Liu X, Wang X and Langdon T G 2010 Mater. Sci. Eng. A 527 6335-39.
[6] Shin D H, Kim I, Kim J, Kim Y S and Semiatin S L 2003 Acta. Mater 51 983-96.
[7] Han Y, Li J, Huang G, Lv Y, Shao X, Lu W and Zhang D 2015 Mater. Des 75 113-9.
[8] Wang L, Wang X, Zhang L C and Lu W 2015 Mater. Sci. Eng. A 645: 99-108.
[9] Geng K, Lu W, Yang Z and Zhang D 2003 Mater. Lett 57 4054-7.
[10] Lu W, Zhang D, Zhang X, Wu R and Sakata T 2001 J. Alloys. Compounds 327 240-7.
[11] Gorsse S, Le Petitcorps Y, Matar S and Matar S 2003 Mater. Sci. Eng. A 340 80-7.
[12] Ranganath S. J. Mater. Sci 1997, 32 1-16.
[13] Tjong S C and Mai Y W. Compos Sci. Technol, 2008, 68 583-601.
[14] Zhao X, Fu W, Yang X and Langdon T G 2008 Scripta Mater 59 542-5.
[15] Zhao X, Yang X, Liu X, Wang C T and Huang Y 2014 Mater. Sci. Eng. A 607 482-9.
[16] Shin D H, Kim I, Kim J, Kim Y S and Semiatin S L 2003 Acta. Mater 51 983-96.
[17] Sergueeva A V, Stolyarov V V, Valiev R Z and Mukherjee A K 2001 Scripta Mater 45 747-52.
[18] Arab M S, Mahallawy N E, Shehata F and Agwa M A. Mater. Des 2014; 64 280-6.