MICROSTRUCTURE AND MECHANICAL PROPERTIES OF TiB+TiC REINFORCED TITANIUM ALLOY BY ECAP

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Keywords: Titanium matrix composite, ECAP, Mechanical property, Microstructure

ABSTRACT

In this study, forged 5 vol. % (TiB+TiC) reinforced Ti6Al4V titanium matrix composites with a heterogeneous structure were successfully subjected to equal channel angular pressing (ECAP). The evolution of the microstructure and mechanical properties (strength and ductility) of (TiB+TiC)/Ti6Al4V during this thermo-mechanical processing was studied. Bc route was selected and the ECAP number varied in the range of 1-4 passes. The microstructure was much refined with the increase of ECAP pass numbers. Formation of homogenous TiB short fibers and TiC particles with some ultrafine grains and sub-grains were observed after 4 ECAP passes. Strength increased with the increase of ECAP pass numbers and saturates after 4 ECAP passes to yield strength of 1150MPa. The ductility after 4 ECAP passes was much higher compared with that after the first ECAP pass. The strengthening effect of small size reinforcements was also significant since their distribution was more homogeneous in the matrix.

1 INTRODUCTION

Equal-channel angular pressing (ECAP), also known as equal-channel angular extrusion (ECAE), is one of the most innovative severe plastic deformation (SPD) methods, which was first introduced by Segal in the 1970s and 1980s[1]. The die of ECAP has two channels of equal cross-section at a certain angle. It is considered as the most prospective candidate for practical applications [2]. The properties of materials processed by ECAP are strongly dependent on the plastic deformation behavior during pressing, which is governed mainly by die geometry and process variables. During the process of ECAP, metal billets are squeezed downwards through the channels under the pressure from the punch without changing their cross-sectional area, which introduces high strains into billets. Since the cross-sectional area of the billets is constant, this process can be repeated to achieve a high total strain in the billets. Various process variables, including the channel angle, pressing pass, route, speed and temperature [3], influence the ECAP-processed material structure. The initial microstructure, phase composition and the slip systems of the materials also play an important role in the formation of ECAP-processed structure [4, 5]. At present, most studies on ECAP are mainly limited to pure metal and its alloys with cubic structure (such as aluminum, copper, nickel, carbon steel, etc.), which have good ductility at ambient temperature. However, the plastic deformation of hexagonal close-packed (HCP) metals is much more difficult than that of cubic structures due to the lack of sufficient independent slip systems [6]. Therefore, the studies on the ECAP to titanium alloys, a kind of HCP metals, are still relatively few. Selected titanium alloys were also concentrated in commercial pure titanium, Ti-6Al-4V [7, 8], TA15 [9], etc.

Titanium matrix composites (TMCs), which have excellent mechanical properties, are promising candidate materials to be used in a lot of different fields such as aviation and space [10, 11]. Multi-phase and multi-scale reinforcements added by in-situ synthesized technique can effectively enhance the comprehensive properties of TMCs. The incorporation of high strength and high stiffness ceramic reinforcements can improve dramatically mechanical performances of particulate reinforced titanium matrix composites, which are currently being widely used in aerospace, commercial automotive engineering, and military applications. Generally, extensive studies have pointed out that TiB short fibers and TiC particles are the best reinforcements for titanium matrix composite. But the addition of high modulus and high strength reinforcements into titanium, it makes titanium matrix composite show high specific strength but low ductility at room and elevated temperature.

The ductility is related to the metal matrix behavior, and the damage is generally due to the particle fracture and interfacial debonding between the metal matrix and particles. It is vital to achieve good grain size, particle size, particle distribution and interface bonding when the composites undergo severe plastic deformation. It can eliminate the agglomeration phenomenon, and stronger adhesion at the particle interface improves load transfer, and further increases the yield strength and plasticity. Hence, it is of interest to apply ECAP process for extruding the titanium matrix composite [12].

In this study, the Ti6Al4V alloy is used as matrix of the composite for its good mechanical properties and wide applicability in the industries. The composite is manufactured from the in situ chemical reaction between Ti6Al4V and B_4C addition, and the microstructure and mechanical properties with the increase of the ECAP pass numbers were investigated. A considerable amount of work have shown that Ti6Al4V alloy can achieve a better balance between high strength and good ductility utilizing the ECAP technique.

2 EXPERIMENTAL METHODS

The studied (TiB +TiC)/Ti6Al4V titanium matrix composite billet with a diameter of 25mm and length of 1000mm was preliminarily annealed at 600℃ for 30min and air cooled to room temperature. Then the billet was cut into specimens of 10mm×10mm×140mm for ECAP on a die-set with the channels' intersection angle of φ=120° and the ECAP die was heated to 550℃ for each pass. The cubic specimen was held for 20min to reach the temperature stabilization, while the subsequent extrusion temperature was 800℃. Route B_c 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 sample was extruded at the speed of 8mm/min, and the samples were extruded for one, two, three and four passes at the same temperature. Longitudinal sections of the samples were prepared for optical microscopy (OM) and scanning electron microscopy (SEM) analyses.

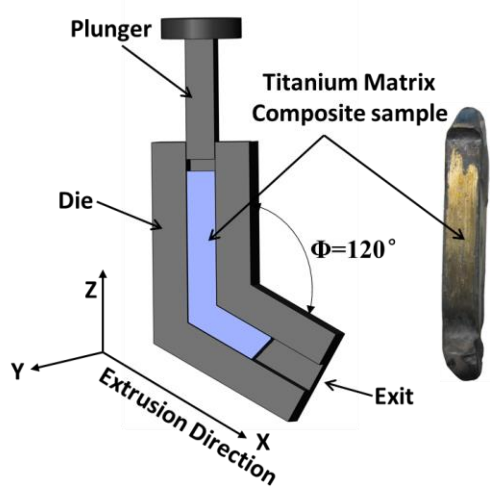

Fig.1 Schematic diagram showing the ECAP process

To observe the microstructural evolution, the deformed specimens were sectioned parallel to compression axis from one side of the deformation specimens, and the cut surface of the smaller part was prepared for metallographic examination by means of optical metallographic (OM) and scanning electron microscopy (SEM). The polished specimens were etched with a solution of 30% HNO_3+10% HF in water for 5 seconds. Tensile tests were performed at room temperature using a universal testing machine Zwick Z100/SN3A. Flat samples with a gage length of 18mm and a cross-section of 3mm×1.6mm were wire Electrical Discharge Machining (EDM) cut from the titanium matrix composite billets. A strain rate of 10^{-3}mm/s was used for the all tests. Yield strength and elongation to failure at the necking cross-section were measured.

3 RESULTS AND DISCUSSION

3.1 Microstructure and phase identification

Fig. 2 shows the microstructure of the (TiB+TiC)/Ti6Al4V titanium matrix composite billets after 1 and 4 ECAP passes at the same pressing speed. In Fig. 2a, it is observed that the microstructure of specimen shows equiaxed structure and the volume fraction of the microstructure consisted of approximately 85% equiaxed α phase, 10% long TiB fibers and large TiC particles. The size of the equiaxed α phase is about 8~10μm in diameter, TiB fibers are about ~15μm in length and ~3μm in width, TiC particles are about 15μm in diameter with an irregular shape. The surfaces of the samples are much smooth and some small metal flash exists comparing with surface after the first ECAP pass. Nevertheless, close inspections revealed that cracks appeared on the sample which was processed by 4 ECAP passes, and the microstructure showed the TiB and TiC reinforcements dispersed homogeneous in the composite. Fig.3 shows the X-ray diffraction patterns for initial and ECAP-processed specimens. It indicates that there are four kinds of phases in the composites, α-Ti, β-Ti, TiB and TiC. The results of X-ray diffraction analysis confirms that the (TiB+TiC)/Ti6Al4V titanium matrix composite could be fabricated by common casting technique using chemical reaction between titanium matrix metal and B_4C powder. There is no significant difference in the X-ray diffraction patterns.

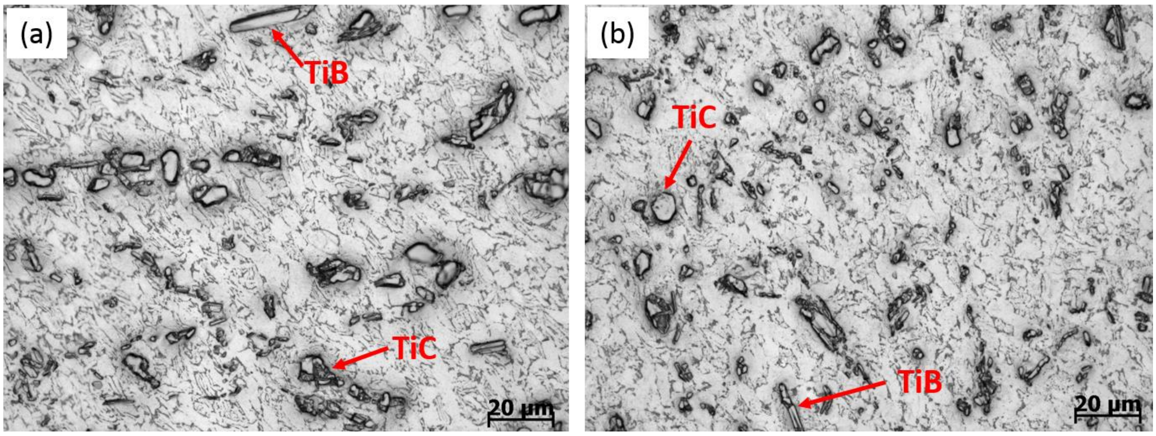

Fig.2 Optical micrograph of the ECAP processed titanium matrix composite billets
(a) 1 pass (b) 4 passes

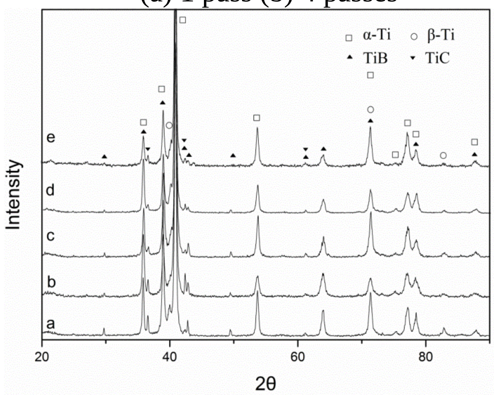

Fig.3 X-ray diffraction patterns (a) Initial material, (b) 1 pass (c) 2 passes (d) 3 passes (e) 4 passes

SEM micrographs (Fig.4) show the microscopic structure of the ECAP processed

(TiB+TiC)/Ti6Al4V titanium matrix composite at higher magnification. The initial ECAP processed TiC large particles and long TiB fibers that are severely fragmented and agglomerated. Distinct α grains were still visible. However, all the grains were severely elongated, as seen in Fig.2. The evolution of the grain size was determined by quantitative metallographic methods and it varied around 20μm when ECAP process started, and about 14μm when the ECAP process finished, i.e. after four passes of ECAP. The evolution of the reinforcements was investigated by measuring the aspect ratio and particle length by using image analysis software. It was found that the aspect ratio of TiB fibers and TiC particles decreased as the ECAP passes number increased in all specimens. Simultaneously, the length of TiB fibers decreased heavily when the billets were deformed by ECAP, which was around 12μm in length when the ECAP process finished. The size of TiC particles was also reduced with the increase of ECAP numbers, refined and near-spherical TiC particles were obtained at when the ECAP process finished.

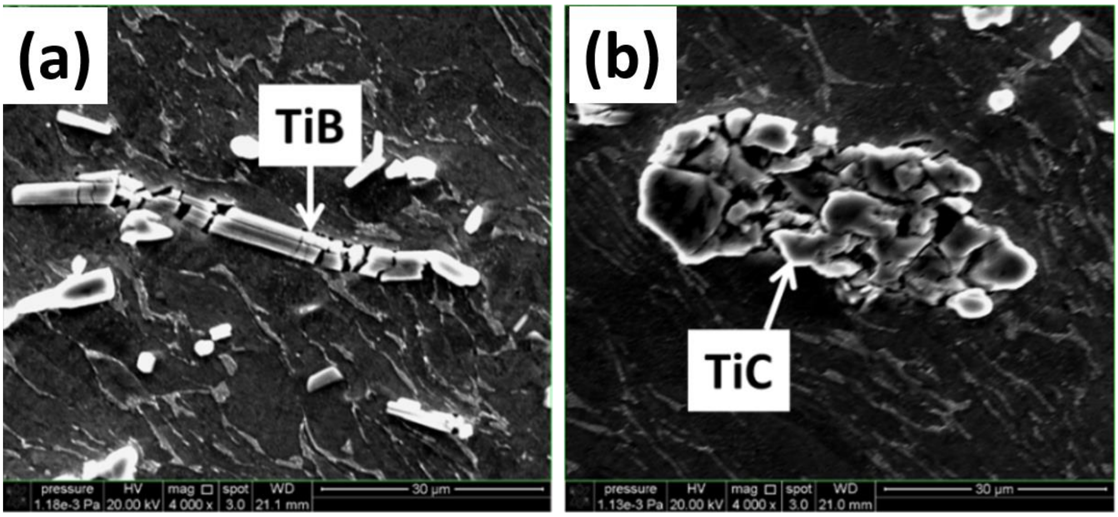

Fig.4 The evolution of the reinforcements of the ECAP extruded composite
(a) Fractured TiB fiber (b) Broken TiC particles

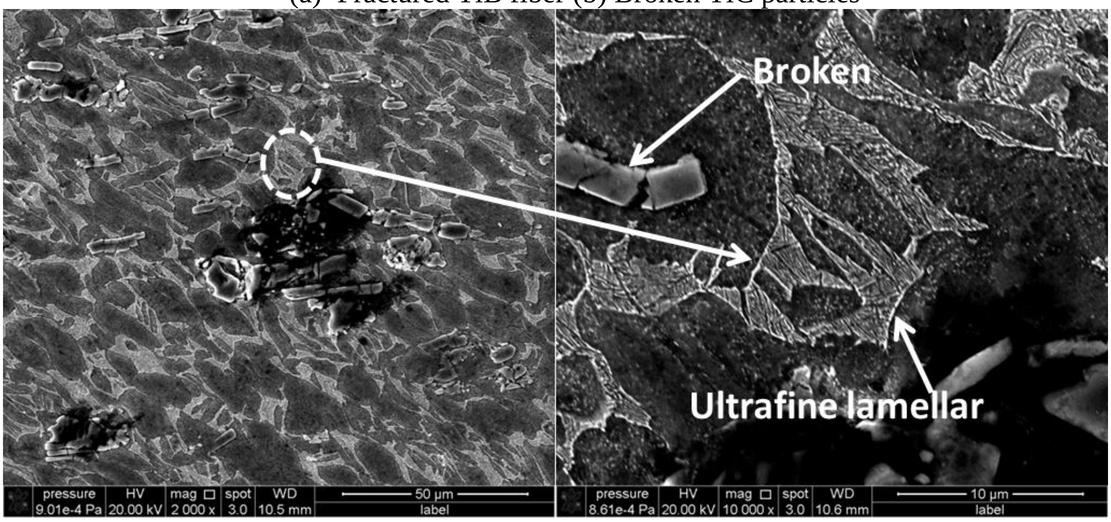

Fig.5 SEM image of the ECAP pressed sample after one pass

As for the matrix, Fig.5 illustrates SEM micrograph of the matrix of ECAP processed TiB+TiC/Ti6Al4V composite. The primary equiaxed α phases were elongated in an angle, lamellar secondary α phase generated a twisting phenomenon due to the bear of the shear deformation of die angle of 120°. Meanwhile, lots of nearly acicular α phases with thickness of about 0.6μm were observed in the transformed β phase, which could be fractured easily in the following high strains, result in much more ultrafine grains in nanoscale. The breakage of ultrafine lamellar can be explained as a result of the following reasons: first, the shearing action which is generated in the matrix by ECAP can directly break the lamellar phase. Second, the compressive stresses generated during ECAP can cause the crushing of the lamellar phase.

3.2 Mechanical property

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 in Fig. 6. The influence of the ECAP pass numbers on the mechanical property could be seen clearly. The ductility was evident increased with ECAP pass numbers through the 120° ECAP dies. It is improved to the biggest value (4.5%) in the four ECAP passes. The improvement was attributed to the refinement of grains after four ECAP passes. The tensile strength of the examined samples was about 1160 MPa after one ECAP pass, which decreased slightly to about 1150MPa after two and four passes. The reason of the improvement is attributing to the phase boundaries increasing resulted from the refinement of reinforcement and the grain size after one ECAP pass number. The effect of work hardening behavior gradually becomes obvious after three ECAP passes, while the tensile strength decreases slightly after four passes. The decrease of strength and the increase of ductility could be the interaction of refinement and working hardening. It is concluded that the ECAP processing provides an effective procedure for improving the ductility of particulate reinforced titanium matrix composite. The ECAP processed (TiB+TiC)/Ti6Al4V composite billets after the second and the fourth pass exhibited higher elongation but similar strength compared with the ECAP processed sample after the first pass.

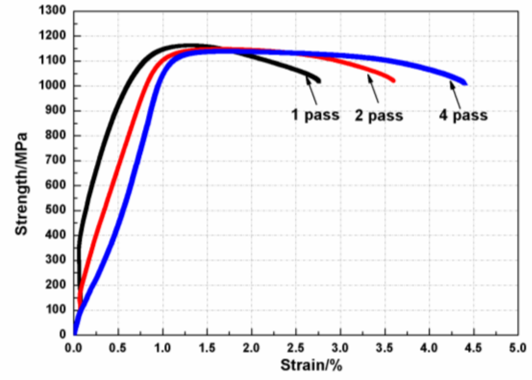

Fig.6 Tensile test curves for the ECAP processed billets for different passes

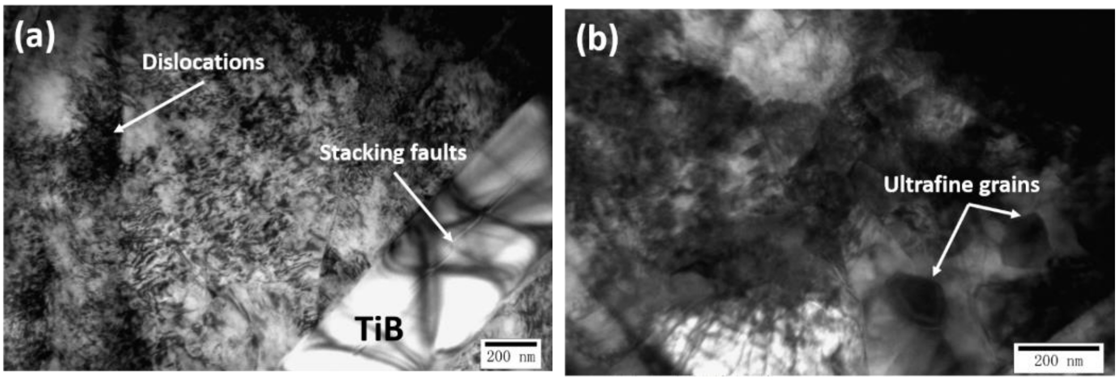

Fig.7 TEM micrographs of TiB+TiC/Ti6Al4V composite billets after (a) one pass of ECAP, (b) four passes of ECAP

From above results, it is clear that the mechanical behavior of different ECAP passes differs from each other. The difference in the ductility can be attributed to their difference between reinforcements and slight variations in the strength. Although their ECAP procedure is quite similar, resulting in the similar microstructure of matrix for each pass, this shows the effectiveness of reinforcements in achieving smaller grain sizes in titanium matrix composite. Fig. 7 shows the TEM micrographs of (TiB+TiC)/Ti6Al4V composite billets. According to the equivalent strain accumulating formula[6, 14], the values of accumulating strain increases with the increase of the ECAP pass numbers, which is better to refine the grain size and reinforcements, even improving the mechanical properties. However, that is not accordance 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 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 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.

Meanwhile, in the ECAP process, some long TiB fibers (with high aspect ratio) near fracture surface fractured. This indicates that the TiB fibers are play a load bearing role in the room temperature tensile tests. The fracture mechanism was quite typical for titanium composite at room temperature tensile test. Some large TiC particles severely break with an angle of about 60° along the tensile direction. According to the study by Bowen, et al [15], large shear strain was drawn in the ECAP process. After four ECAP passes, the size of TiB fibers and TiC particles decreased heavily, and interfacial debonding is easy to take place in the two ends of the reinforcement, which is due to a stress concentration in the end of the reinforcement with the increase of the shear strain. Furthermore, lots of cavities will immediately generate around the TiB fibers and TiC particles. It could be one of the dominant reasons for the failure of the sample which was extruded in the next ECAP pass. Thus, the reinforcements' load-bearing effect would be less dominant than the dislocation density effect for the refinement of the reinforcement dispersion strengthening.

5 CONCLUSIONS

(1). ECAP of TiB fibers and TiC particles reinforced titanium matrix composite was successfully carried out up to 4 ECAP passes. 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). Significant breaking of reinforcements was a result of ECAP procedures. After 4 ECAP passes, the TiC particle size decreased from ~15 μm to ~5μm, and the average length of TiB fibers decreased from ~15 μm to 5μm. Breaking of the reinforcements was observed to be a result of brittle fracture in the reinforcements by the severe plastic deformation during ECAP.

(3). ECAP processed (TiB+TiC)/Ti6Al4V composites exhibit higher elongation but similar strength compared with the sample after the first ECAP pass. The enhancement of ductility is dominated by the decreasing of reinforcements' size, and the homogeneity of the reinforcements' distribution through four passes.

ACKNOWLEDGEMENTS

The authors are grateful to the National Nature Science Foundation of China (Grant No: 51371114, 51501112, 51504151, U1602274), the fund of the State Key Laboratory of Solidification Processing in NWPU (No. SKLSP201620) and the supported by the 111 Project (Grant No. B16032).

REFERENCES

[1]. Segal V M. Materials processing by simple shear, *Materials Science and Engineering A*, **197**, 1995, , pp. 157~164 (https://doi.org/10.1016/0921-5093(95)09705-8)

[2]. S.L. Semiatin, D.P. Delo, E.B. Shell, The effect of materials properties and tooling design on deformation and fracture during equal channel angular extrusion. *Acta Materialia*, **48**, 2000, pp. 1841-1851 (doi:10.1016/S1359-6454(00)00019-7)

[3]. V.V. Stolyarov, Y.T. Zhu, T.C. Lowe, R.Z. Valiev, Microstructure and properties of pure Ti processed by ECAP and cold extrusion, *Materials Science and Engineering A*, **303**, 2001, pp. 82–89 (http://doi.org/10.1016/S0921-5093(00)01884-0)

[4]. T. G. Langdon, M. Furukawa, et al. Using equal-channel angular pressing for refining grain size, **4**, *JOM*, 2000, pp. 30-33 (https://search.proquest.com/docview/232553913?accountid=13818)

[5]. T. C. Lowe, Producing nanoscale microstructures through severe plastic deformation, *JOM*, **4**, 2000, pp. 27-28. (https://search.proquest.com/docview/232553544?accountid=13818)

[6]. D.H. Shin, I. Kim, J. Kum, Y.S. Kim, S.L. Semiatin, Microstructure development during equal-channel angular pressing of titanium, *Acta Materialia*, **51**, 2003, pp. 983–996 (http://doi.org/10.1016/S1359-6454(02)00501-3)

[7]. G.G. Yapici, I. Karaman , Z.P. Luo, H. Rack, Microstructure and mechanical properties of severely deformed powder processed Ti-6Al-4V using equal channel angular extrusion, Scripta Materialia, **49(10)**, 2003, pp. 1021–1027. (http://doi.org/10.1016/S1359-6462(03)00484-6)

[8]. Y.G. Ko, C.S. Lee, D.H. Shin, S.L. Semiatin, Low temperature superplasticity of ultrafine grained Ti6Al4V processed by equal channel angular pressing, *Metallurgical and materials Transactions A*, **37(2),** 2006, pp. 381-391 (doi: 10.1007/s11661-006-0008-z)

[9]. Y. Zhao, H. Guo, Z. Shi, Z. Yao, Y. Zhang, Microstructure evolution of TA15 titanium alloy subjected to equal channel angular pressing and subsequent annealing at various temperatures, J *Journal of Materials Processing Technology*., **211**, 2011, pp. 1364–1371 (http://doi.org/10.1016/j.jmatprotec.2011.03.004)

[10]. W.F. Ding, B.A. Zhao, J.H. Xu, C.Y. Yang, Y.C. Fu, H.H. Su, Grinding behavior and surface appearance of (TiCp + TiBw)/Ti-6Al-4V titanium matrix composites. *Chinese Journal of Aeronautics* **27(5)**, 2014, pp. 1334–1342 (http://doi.org/10.1016/j.cja.2014.08.006)

[11]. L.J. Huang, L. Geng, H.X. Peng. In situ (TiB_w + TiC_p)/Ti6Al4V composites with a network reinforcement distribution, *Materials Science and Engineering A*, **527** 2010, pp. 6723–6727 (http://doi.org/10.1016/j.msea.2010.07.025)

[12]. Y.F. Han, J.X. Li, et al, Effect of ECAP numbers on microstructure and properties of titanium matrix composite, *Materials & Design*, 75, 2015, pp. 113-119 (doi:10.1016/j.matdes.2015.03.018)

[13]. J. R. Bowen, A. Gholinia, S.M. Roberts, P.B. Prangnell, Analysis of the billet deformation behaviour in equal channel angular extrusion. *Materials Science and Engineering: A*, **287**, 2000, pp. 87-99 (doi:10.1016/S0921-5093(00)00834-0)