FABRICATION OF Al-BASED COMPOSITES REINFORCED WITH Al_2O_3-TiB_2 CERAMIC COMPOSITE PARTICULATES USING VORTEX-CASTING METHOD

M.R. Roshan[a], R. Taherzadeh Mousavian[b,*], H. Ebrahimkhani[b], A. Mosleh[c]

[a] Department of Materials Science and Engineering, School of Engineering, Shiraz University, Shiraz, Iran
[b] Department of Metallurgy, Zanjan Branch, Islamic Azad University, Zanjan, Iran
[c] Department of Materials Science and Engineering, Tarbiat Modares University, Tehran, Iran

Abstract

Vortex casting is one of the simplest methods of producing metal matrix composites (MMCs). However, this simple method does have some drawbacks, which reduce the mechanical properties of the produced composites. In this study, we tried to modify the process of composite production before, during, and after the casting procedure. Low-cost Al_2O_3-TiB_2 ceramic composite particles, which produced after combustion synthesis, were used as reinforcement. These powders, which are thermodynamically stable with molten aluminum below 900 °C, were mixed with aluminum and magnesium powders before casting using ball milling and the mixed powders were injected into the molten metal (pure Al). This process was applied to enhance the wettability of ceramic particles with molten aluminum. After casting, warm equal channel angular pressing (ECAP) and hot extrusion processes were applied to investigate their effects on the mechanical properties of the final composites. It was revealed that both warm ECAP and hot extrusion have a strong influence on increasing the mechanical properties mainly due to decreasing the amount of porosities.

Keywords: Composites; Casting; Equal channel angular processing; Extrusion; Mechanical characterization.

1. Introduction

Today, there is increasing demand for research on finding new materials superior to the conventional materials. In these studies, aluminum based composite materials have been gaining greater attention, especially in the aviation, space, and automotive industries. These composites combine the great strength of ceramics and the ductility from the metallic matrix. Their advantage over ferrous materials is the reduction in weight, leading to lower moment inertia and fuel consumption, and better corrosion resistance [1-4].

Many techniques have been developed for producing particulate reinforced MMCs, such as powder metallurgy and squeeze casting. Stir casting (vortex technique) is generally accepted as a commercial practicable method. It's advantages lie in its simplicity, flexibility, and applicability to large volume production. This process is the most economical of all the available routes for MMC production and allows very large-sized components to be fabricated [5-11].

However, several difficulties in stir casting are of concern, which are [5-9]: (i) Chemical reactions between the reinforcement material and matrix alloy, (ii) Porosity in the cast MMC, (iii) Wettability between the two main substances, and (iv) Difficulty in achieving a uniform distribution of the reinforcement material.

By using an in situ fabrication method or the ceramic reinforcement, which are thermodynamically stable with molten metal, no reaction could take place between reinforcement and matrix. In situ fabrication has many advantages. In particular, a clean interface will be obtained between matrix and reinforcement. However, the formation of undesirable compounds in some systems is inevitable. Many authors [12-15] reported the formation of undesirable compounds (Like Al_3Ti or Al_4C_3) during in situ fabrication, leading to reduction in the mechanical properties.

Another alternative is using thermodynamically stable ceramic reinforcement. Reinforcement materials generally used to reinforce aluminum alloys include carbides (e.g. SiC and TiC), boride (TiB_2 and ZrB_2), and oxides (Al_2O_3 and SiO_2) [14, 15]. Among these reinforcing particulates, titanium diboride (TiB_2) is particularly attractive because it exhibits high elastic modulus and hardness, high melting point and good thermal stability. TiB_2 particles do not react with aluminum, thereby, avoiding the formation of brittle reaction products at the reinforcement−matrix interface [14, 15]. On the other hand, alumina does not react with aluminum. Therefore, a combination of

alumina and TiB_2 ceramic particles seems to be thermodynamically stable with molten aluminum. These materials are expensive and using cheaper reinforcement will lead to reduction in the fabrication cost for MMCs. Self-propagating high-temperature synthesis (SHS) is one of the rapidly emerging cost-effective technologies used to synthesize monolithic and composite in situ ceramics. Al_2O_3-TiB_2 ceramic composite was fabricated in our previous works [16-18] using SHS method and low cost reactants (Al, TiO_2, and acid boric). Care should be taken that during SHS, all in situ reactions take place completely, otherwise, the fabricated ceramic particles would not be thermodynamically stable due to the presence of starting reactants [16-18]. Many authors [19-23] focused on the production and properties of aluminum composites reinforced with Al_2O_3-TiB_2 ceramic composite using in situ fabrication via powder metallurgy method, while Kurtoglu [24] and Niyomwas [11] fabricated Al_2O_3-TiB_2 and TiB_2–Al_2O_3–Fe_xAl_y ceramic composites by using SHS method and used these composites as reinforcement in the molten aluminum.

As mentioned, the billets synthesized using the stir casting technique have inherent problems such as the presence of porosity, agglomeration of ceramic reinforcement, and gas entrapment. Extrusion is a common secondary process used in the manufacturing of MMC materials. For particulate-reinforced MMCs in particular, extrusion has been widely used to increase the quality of the produced composites [4, 25-31]. Equal channel angular pressing (ECAP), being one of the severe plastic deformation (SPD) methods that can produce submicrometer or even nanometer-sized materials, has drawn much attention in recent years [32]. Equal channel angular pressing is an attractive process because it has the potential to produce large samples. Most investigations on ECAP have concentrated on pure metals and metallic alloys, while Al-SiC composite was subjected to severe plastic deformation through equal channel angular pressing (ECAP) in Ramu et al [4] study. An improvement in mechanical properties of the composites was observed in their study after ECAP.

The presence of oxide films on the surface of molten metal and the adsorbed contaminant on the reinforcement surface generally lead to non-wetting of the reinforcement with molten metal. The alumina oxide layer creates a resistance to reinforcement particle penetration of a molten matrix, especially when the particles are added from the top of a cast. Therefore, ceramic powders were rejected during injection and they could not enter into the molten metal [33-37]. This problem might lead to formation of a composite with unexpected chemical composition (lower amount of reinforcement). Some of the techniques to improve metal-reinforcement wettability include metallic coatings on the reinforcements, addition of reactive elements, such as magnesium, calcium, or titanium, to the melt and heat treatment of particles before addition [33-38].

Recently, researchers [39-41] have been used ball milling to mix and coat the ceramic powders with aluminum powder. In Ghahremanian et al [39] study, particulate composite powders were produced by low-energy ball milling of equal volumes of pure aluminum powder and SiC particles to simplify the incorporation of ceramic particles in the molten aluminum. It was shown in their study that a higher incorporation as well as better distribution of ceramic particles would be obtained using this method. Amirkhanlou et al [40, 41] have used a mixture of Al-SiC and Al-SiC-Mg powders milled using ball milling and then injected this mixture in the molten aluminum. It was reported in their studies that this method aid the incorporation of ceramic particles, and Mg reacts with oxide surface layer, leading to further incorporation of ceramic particles and improved mechanical properties.

In this study, ceramic composite particles that were prepared using SHS technique were milled with Al and Mg powders to simplify their incorporation in the molten aluminum. In addition, warm ECAP and hot extrusion were separately applied on as-cast billets to assess their effects on the mechanical strength and hardness of the composites.

2. Experimental procedures

Details of the experimental set-up and production processes of Al_2O_3-TiB_2 ceramic particles are reported in our previous studies [16-18]. The morphology and particle size of the ceramic particles were determined using SEM (see Fig. 1). The size of the ceramic particles was found to be less than 20 μm. A brief
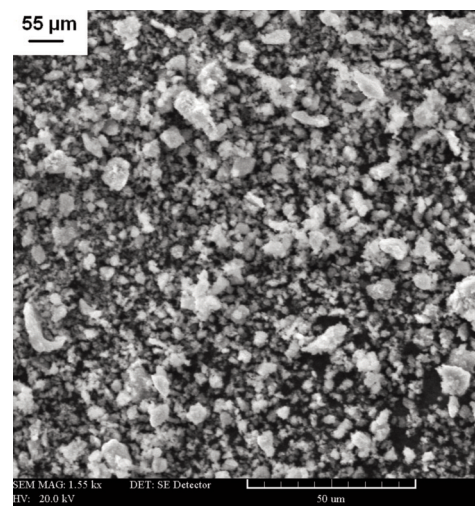

Figure 1. The morphology and particle size of Al_2O_3-TiB_2 ceramic particles.

comparison between the properties of pure Al and Al_2O_3-TiB_2 ceramic composite is listed in Table 1.

Table 1. *The properties of pure aluminum and Al_2O_3-TiB_2 ceramic composite [42].*

Properties	Pure Al	Al_2O_3-TiB_2 ceramic composite
Hardness (Vickers)	20 Hv	23.4 GPa ± 3.2
Thermal expansion coefficient	23.1×10^{-6} $°C^{-1}$	8.6×10^{-6} $°C^{-1}$
Young's modulus	70 GPa	415 GPa
Theoretical density	2.7 g.cm^{-3}	4.12 g.cm^3

In order to investigate the possibility of reaction occurrence between the ceramic powders with molten aluminum, DSC analysis (Netzsch STA 409, Germany) was used. The random amounts of aluminum powders were mixed with Al_2O_3-TiB_2 powders and then the mixed powders were heated from 600 to 900 °C with the heating rate of 20 °C/min using pure argon atmosphere.

High-energy ball milling was used to mix the ceramic particles with Al and Mg powders with the average particle sizes of 20 µm for both metals so that the final composite contains 2 %wt ceramic and 1 %wt Mg beside remained pure aluminum. The magnesium lowers the superficial tension and the contact angle (θ) between Al and ceramic. It acts as a surfactant power that gets the oxygen [6, 34, 36]. The milling was performed in a planetary ball mill with an alumina container and balls, under an argon atmosphere. The ball to powder weight ratio was 10:1 and the rotation speed was 450 rpm. Milling was performed for 2 h as further milling might lead to a reaction between Al_2O_3 and Mg.

In order to incorporate the mixed powders into the aluminum melt, stir casting (vortex technique) was applied. 450 g of commercial pure Al was used as a matrix material. An electrical resistance furnace with a stirring assembly (a graphite impeller) was used for the dispersion of the ceramic particles into liquid aluminum. Al was melted and the temperature was maintained at 730°C. The stirrer was lowered into the melt slowly to stir the molten metal at a speed of 500 rpm. Approximately 0.3 g mixed powders was inserted into an aluminum foil by forming a packet. The packets were added every 20 s to the centre of the vortex in a continuous stream when the vortex was formed. The packet of mixture melted and the particles started to distribute in the aluminum melt. This method enabled a full and homogenous distribution of the particles in the aluminum matrix [9]. The temperature was measured by K-type thermocouple with a solid-state relay temperature

controller with a temperature accuracy of ±1 °C. The temperature of the furnace was gradually lowered until the melt reached a temperature in the range of 710 °C, while stirring was continued. The maximum duration of mixing was 15 min. Before casting, the surface of the melt was cleaned by skimming. The slurry was finally cast into a preheated steel mould in the form of a 10 mm x 80 mm cylinder using copper hollow cylinders with a wall thickness of 1 mm and height of 80 mm for the ECAP specimens. Another preheated steel mould in the form of a 27mm x 40mm cylinder was used for the extrusion specimens. Then, composite billets were extruded to 6 mm diameter bars at 500°C, through shear-faced dies, at a reduction ratio of 20:1 and a speed of 0.2 mm s^{-1}. A copper hollow cylinder with a wall thickness of 1mm was used to avoid die damage by the hard composite surface during ECAP and to prevent instabilities at the surface of the composite that may occur during ECAP. The ECAP facility had an internal angle of 90° and an angle of 20° at the outer arc of curvature at the intersection of two parts of the channel (see Fig. 2). The pressing was conducted at a constant displacement rate of 0.5 mm s^{-1} at 200 °C (<0.5 T_m of the aluminum) and the pressing force was monitored during ECAP. The copper layer was then removed by machining to make samples for mechanical behavior studies.

Tensile specimens were prepared from the as cast and as formed composites. All of the tensile tests were performed at room temperature using an Instron type-testing machine operating at a constant rate of crosshead displacement, with an initial strain rate of 2×10^{-3} s^{-1}. The 0.2% proof strength (interpreted as the measurable yield stress), ultimate tensile strength
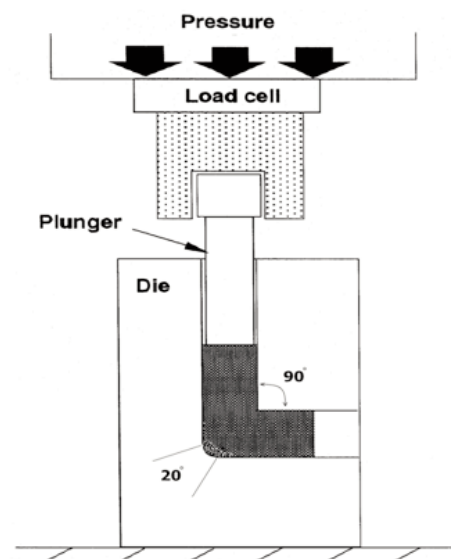

Figure 2. *The schematic of ECAP facility used after casting.*

(UTS) and ductility (% elongation to failure) were measured and averaged over 3 test samples. Vickers hardness was measured on the matrix of the composites using 100 g load and loading time of 15 seconds. The values reported are the average of at least five readings. The density of the composite samples was measured using Archimedes' principle. Distilled water was used as the immersion fluid. Theoretical density was calculated by rule of mixture and compared with the measured densities. Microstructural characterizations were done by using scanning electron microscope (SEM equipped with EDS, CAMSCAN-MV2300 Model, Oxford). Fracture surfaces were studied under SEM to find out the mechanism of fracture for the samples.

3. Results and discussion

The result of DSC analysis is shown in Fig. 3. This curve indicates that no chemical reaction was taken place between aluminum and ceramic powders as no exothermic peak was revealed after melting the aluminum, confirming that the Al_2O_3-TiB_2 ceramic composite powders are thermodynamically stable with aluminum below 900 °C.
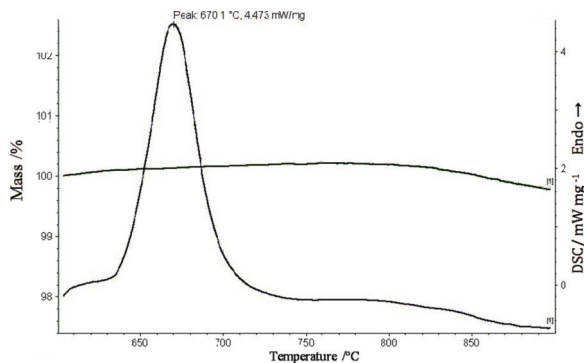
Figure 3. *The results of thermal analysis for the mixture of Al and Al_2O_3-TiB_2 powders.*

Fig. 4 shows a typical microstructure of the mixed powders (ceramic and metallic particles) after milling for 2 h. The purpose of powder mixing before casting was to simplify the incorporation of ceramic particles into the molten aluminum. A good mechanical locking could be observed because of intensive collision with balls during milling. It was observed in our experiments that no especial change would be observed in incorporation of ceramic particles if ceramic and metallic powders have low interfacial areas after mixing.

After applying warm ECAP and hot extrusion, densities of all the as cast and as formed samples are obtained and given in Table 2. As given in this Table, warm ECAP and hot extrusion processes significantly
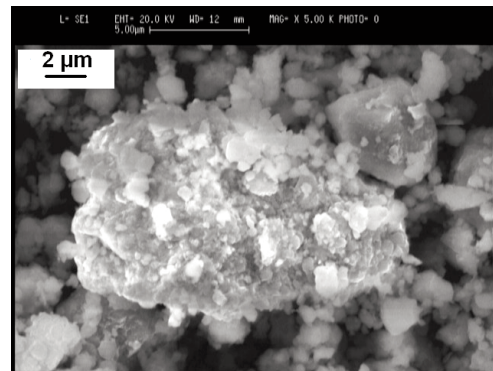
Figure 4. *A typical microstructure of ceramic reinforcement mixed with Al and Mg powders after milling for 2 h.*

decrease the amounts of porosities after casting. It seems that both warm ECAP and hot extrusion have almost the same effects on reducing the amounts of porosities. The decreased porosity of composite during warm ECAP and hot extrusion is due to the compressive forces generated by the interaction of the composite billet with the container and die, resulting in the flow of the aluminum into the voids under the applied shear forces [4, 27-31].

Table 2. *The relative density of the samples after casting, warm ECAP, and hot extrusion.*

Sample	Relative density (%)
As cast sample	95.2
As ECAPed sample	98.1
As extruded sample	98.9

The microstructure of the sample after casting is shown in Fig. 5. The Al dendrites solidify first during solidification of the composite, and the particles are rejected by the solid−liquid interface, and hence are segregated to the inter-dendritic region, leading to agglomeration of ceramic particles and non-uniform distribution. In addition, the formation of small pores after casting and solidification is clearly observed in the matrix and beside the ceramic particles. The line scan (EDAX analysis) indicated the presence of Mg, Ti, O, and matrix material around a ceramic particle. Ti, O, and Mg contents decrease whereas aluminium increases as one move from ceramic to the matrix.

The microstructures of the as-formed composites after warm ECAP and hot extrusion are shown in Fig. 6. It can be understood from Fig. 6a that hot extrusion affects the distribution of ceramic particles at a high degree. During the deformation of the composite in a die, the non-deformable ceramic particles tend to fragment with the softer matrix being forced into the voids created by the fracture event. Fig. 6b indicates that no considerable change would occur after warm
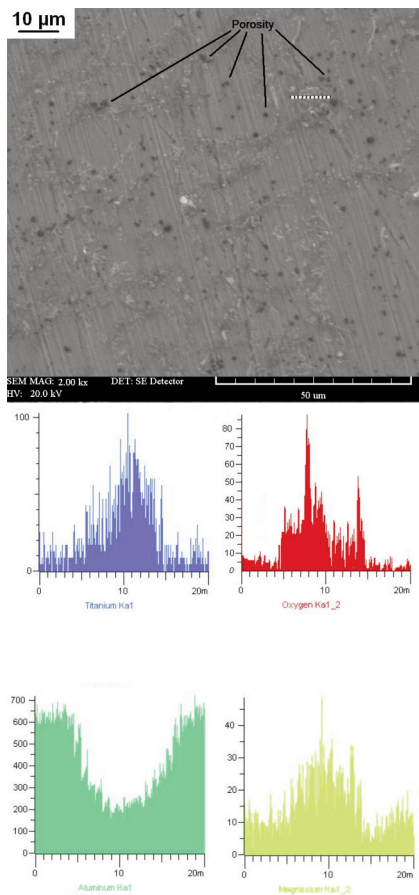
Figure 5. *The SEM/EDAX analysis of composite after casting.*

Figure 6. *The SEM microstructure of composite after hot extrusion (a) and warm ECAP (b).*

ECAP as regards distribution and fragmentation of ceramic particles in respect of hot extrusion. However, very low amount of porosities could be observed after both forming processes.

The mechanical behavior affected by the porosity formation in the stir casting of metal matrix composites is highly focused on tensile properties. Porosity tends to decrease the mechanical properties of MMCs. Porosity formation, which obviously depends on the processing and microstructure, significantly affects the yield strength (YS), the ultimate tensile strength (σ UTS), and the ductility (percentage elongation) of the MMCs. The mechanical properties results of the as cast and as formed composites indicate that warm ECAP and hot extrusion have different influences on the properties of the produced composites. Fig. 7 shows the results of tensile and microhardness tests. Some important points can be understood from this figure. First, due to immediate failure, very low values of tensile strength and ductility were obtained for the as cast sample. The observed low ductility in the as-cast samples can be explained by the heterogeneity in particle distribution and mainly by the high porosity content. During the
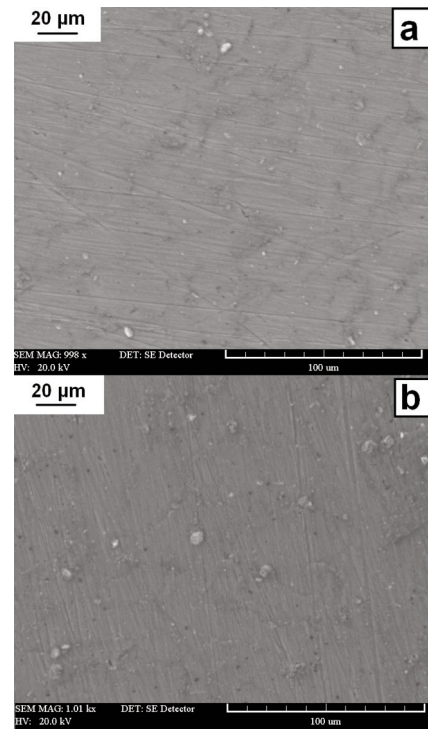
tensile test of the unreinforced aluminum matrix, plastic deformation is considerable but the presence of ceramic clusters exerts constraints on the plastic flow within the ductile matrix and, consequently, high levels of stress concentration and triaxial stresses appear in the composite. As is tabulated in the density measurements, the relative density of the as cast sample is lower than that of the as-ECAPed and extruded sample. The presence of pores is also very effective on the concentration of stresses, leading to a lower ductility and strength.

Second, after hot extrusion, the values of percent elongations are significantly higher than those of after warm ECAP. Very good agreements are recognized between the hardness values and the yield stresses of the samples. The as-ECAPed sample shows the highest amounts of hardness and yield stress. This is because of the fact that warm ECAP was done at 200 °C, lower than half the melting point of the aluminum. In contrast to hot extrusion process, no recovery could occur for this sample, and due to work hardening, a higher hardness and yield stress were obtained. Although, as-ECAPed sample was subjected to work hardening, a higher value of ductility was obtained for this sample rather than as-cast sample, meaning the importance of porosity.

Tensile fracture surfaces are helpful in elucidating microstructural effects on the ductility and fracture properties of the composites. Fig. 8 exhibits the fracture surface of the as-cast sample. The white areas
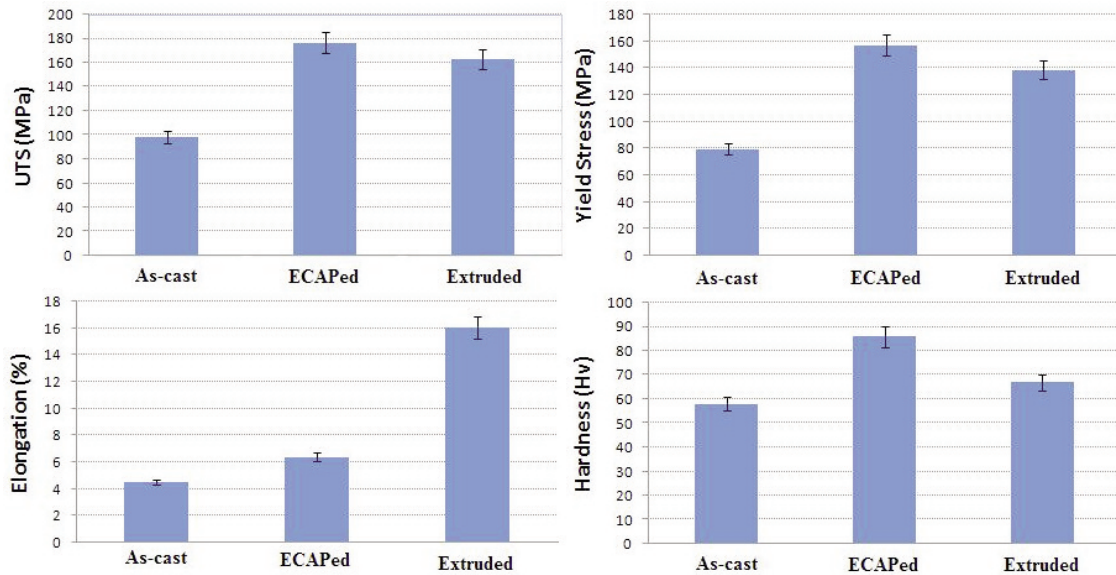
Figure 7. The mechanical properties (tensile and hardness test results) of the as cast and as formed samples.

(brittle area) are related to the agglomerated ceramic particles. A very low value of ductility is obtained for this sample. Particle fracture and porosities are more responsible for the fracture of the as cast sample. This fracture surface revealed the agglomeration of ceramic particles, which caused local stress concentrations in the composite and led to crack formation. When particle fracture occurs, microvoids are nucleated and then by growth and coalescence of these voids, crack propagation occurs.

Fig. 9a shows the fracture surface of the as ECAPed sample. This sample has a higher ductility in comparison with that of the as cast sample (see Fig. 7). This sample has a low amount of porosity. Particle fracture might be responsible for the fracturing of this sample. It seems that ECAP process could not highly break the agglomerated particles as large white areas could be observed in Fig. 9a. The extruded sample has the highest ductility. Although there is no significant difference in amounts of porosity of the as-ECAPed and extruded samples (see Table 2), however, it could be

observed that the later sample has a much higher ductility, meaning that the breakage of the agglomerated ceramic particles is highly effective for increase of ductility. As Fig. 9b depicted, the white areas are small and separated, and as reported extrusion can highly break the agglomerated particles [25-31].
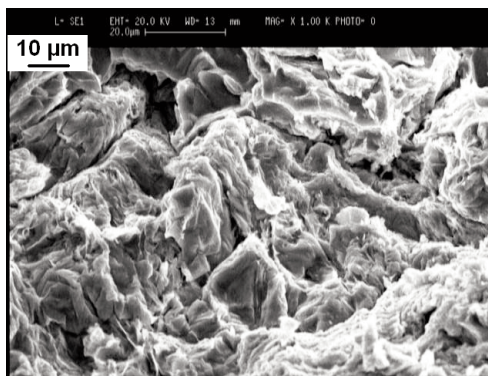
Therefore, it is concluded that warm ECAP at 200
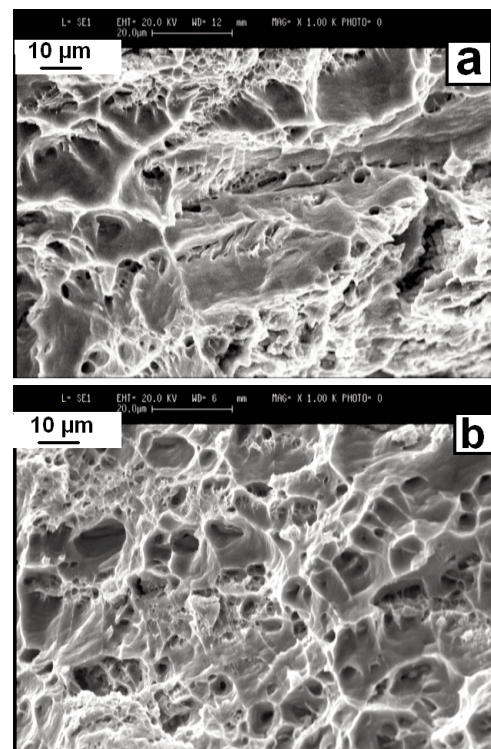

Figure 9. Fracture surfaces of as formed samples, (a) after warm ECAP (b) after hot extrusion.

Figure 8. The fracture surface of the sample after casting.

°C increases the yield stress and hardness of as cast composite. Although, the porosities were considerably eliminated after warm ECAP, however, no especial increase could be observed in ductility for this sample because of non-fragmentation of ceramic particles during ECAP. In contrast, as no agglomeration could be seen in the extruded sample, a very high amount of ductility was obtained in this sample.

4. Conclusion

In this study, Al-based composite reinforced with Al_2O_3-TiB_2 ceramic composite particles (produced using SHS) was fabricated using vortex casting method. The composites, which are produced by stir (vortex) casting, have poor mechanical properties. Therefore, warm ECAP and hot extrusion were used to improve the mechanical properties. Based on the obtained results, the following outcomes are drawn:

1. Applying Warm ECAP and, especially hot extrusion after casting leads to a substantial increase in the relative density of the samples.

2. After warm ECAP, the samples exhibit the highest hardness and yield stress, while after hot extrusion the samples show the highest ductility.

3. Although the amounts of remained porosities are almost the same after warm ECAP and hot extrusion, however, different mechanical properties are obtained after these processes, meaning that the type and temperature of deformation is highly important after casting.

References

[1] Chen Xing, Yang Chengxiao, Guan Leding, Yan Biao, Mater. Sci. Eng. A 496 (2008) 52–58.

[2] E.M. Ruiz-Navas, M.L. Delgado, B. Trindade, Composites: Part A 40 (2009) 1283–1290.

[3] G. Ramu, Ranjit Bauri, Mater. Des. 30 (2009) 3554–3559.

[4] K.M. Shorowordi, T. Laoui, A.S.M.A. Haseeb, J.P. Celis, L. Froyen, J. Mater. Process Technol. 142 (2003) 738-743.

[5] H. Abdizadeh, H.R. Baharvandi, K. Shirvani Moghaddam, Mater. Sci. Eng. A 498 (2008) 53-58.

[6] J. Hashim, L. Looney, M.S.J. Hashmi, J. Mater. Process Technol. 92-93 (1999) 1-7.

[7] S. Balasivanandha Prabu, L. Karunamoorthy, S. Kathiresan, B. Mohan, J. Mater. Process Technol. 171 (2006) 268–273.

[8] M. Kok, J. Mater. Process Technol. 161 (2005) 381–387.

[9] A. Mazahery, H. Abdizadeh, H.R. Baharvandi, Mater. Sci. Eng. A 518 (2009) 61-64.

[10] A. Onat, H. Akbulut, F. Yilmaz, J. Alloy. Compd 436 (2007) 375–382.

[11] S. Niyomwas, Int J SHS. 19 (2) (2010) 152-158.

[12] A.R. Kennedy, D.P. Weston, M.I. Jones, Mater. Sci. Eng. A316 (2001) 32–38.

[13] S. Sheibani, M. Fazel. Najafabadi, Mater. Des. 28 (2007) 2373–2378.

[14] S.C. Tjong, S.Q. Wu, H.G. Zhu, Compos. Sci. Technol. 59 (1999) 1341-1347.

[15] S.C. Tjong, G.S. Wang, Mater. Sci. Eng. A 386 (2004) 48–53

[16] R. Taherzadeh Mousavian, S. Sharafi, M.R. Roshan, M. H. Shariat, J. Therm Anal Calorim. 104 (3) (2011) 1063-1070.

[17] R. Taherzadeh Mousavian, S. Sharafi, M.H. Shariat, Int J Refract Met Hard Mater 29 (2011) 281-288.

[18] R. Taherzadeh Mousavian, S. Sharafi, M.H. Shariat, Iranian J. Mater. Sci. Eng. 8 (2) (2011) 1-9.

[19] H. Zhu, H. Wang, L. Ge, Wear. 264 (2008) 967–972.

[20] Z. He-Gou, W. Heng-zhi, G. Liang-qi, Chen Shi, W. Shen-qing, T Nonferr Met Soc 17(20O7) 590-594.

[21] S.C. Tjong, G.S. Wang, Mater. Sci. Eng. A 386 (2004) 48-53.

[22] Heguo Zhu, Hengzhi Wang, Liangqi Ge, Wenjuan Xu, Yunzhan Yuan, Mater. Sci. Eng. A 478 (2008) 87-92.

[23] Z.Y. Ma, S.C. Tjong, Metall Mater Trans A. 28 (1997) 1921-1942.

[24] A. Kurtoglu, M.Sc. Thesis, Middle East Technical University, Turkey (2004).

[25] U. Cocen, K. Onel, Compos Sci Technol. 62 (2002) 275-282.

[26] R. Rahmani Fard, F. Akhlaghi, J. Mater. Process. Technol. 187–188 (2007) 433–436.

[27] I. Sahin, AA. Eker, Journal of Materials Engineering and Performance, 20 (6) (2011) 1090-1096.

[28] Z. Wang, M. Song, C. Sun, D. Xiao, Y. He, Mater. Sci. Eng. A 527 (2010) 6537-6542.

[29] X.J. Wang, K. Wu, H.F. Zhang, W.X. Huang, H. Chang, W.M. Gan, M.Y. Zheng, D.L. Peng, Mater. Sci. Eng. A 465 (2007) 78-84.

[30] A.R. Kennedy, S.M. Wyatt, Compos. Sci. Technol. 60 (2000) 307-314.

[31] A. Sakthivel, R. Palaninathan, R. Velmurugan, P. Raghothama Rao, J Mater Sci 43 (2008)7047-7056.

[32] T. Liu, W. Zhang, S.D. Wu, C.B. Jiang, S.X. Li, Y.B. Xu Shenyang, Mater. Sci. Eng. A 360 (2003) 345-349.

[33] A.R. Kennedy, A.E. Karantzalis, Mater. Sci. Eng. A 264 (1999) 122–129.

[34] J. Hashim, L. Looney, M. S. J. Hashmi, J. Mater. Process. Technol. 119 (2001) 329- 335.

[35] A. Urena, E.E. Martinez, P. Rodrigo, L. Gil, Compos. Sci. Technol 64 (2004) 1843–1854.

[36] J. Hashim, L. Looney, M. S. J. Hashmi, J. Mater. Process. Technol. 119 (2001) 324- 328.

[37] T. P. D. Rajan, R. M. Pillai , B. C. Pai, J Mater Sci 33 (1998) 3491-3503.

[38] HZ. Ye, XY. Liu, J Mater Sci 39 (2004) 6153-6171.

[39] M. Ghahremanian, B. Niroumand, M. Panjepour, Met. Mater. Int 18 (1) (2012) 149-56.

[40] S. Amirkhanlou, B. Niroumand, Trans. Nonferrous Met. Soc. China 20 (2010) 788-93.

[41] S. Amirkhanlou, B. Niroumand, Materials and Design 32 (2011) 1895–902.

[42] M.A. Meyers, E.A. Olevsky, J. Ma, M. Jamet, Materials Science and Engineering A311 (2001) 83–99.