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Synthesis and Characterization of Aluminum Composites Materials Reinforced with TiC Nano- Particles

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Abstract

In this paper, aluminum matrix composites are successfully synthesized by reinforcing various TiC Nano-particles (273.196 nm, 194.732nm, and 149.071 nm). The green compacts of Al-TiC composites were sintered for 3 h at 500, 550, and 600^{0}C. Hardness test and wear test were carried out on the Al-TiC composites. Powders of (Al, and TiC), and composites of (Al-TiC) were characterized using scanning electron microscopy, and X-ray diffraction techniques. Different weight ratios (5%wt, 15%wt, and 25%wt TiC) and different particles size of TiC were used to study the microstructure, and mechanical properties. The results obtained reveal that the densities of sintered composites show a marginal increase with the decrease in the particle size. Al-25wt % TiC composites (particle size 149.07 nm) with 600^{0}C sintering temperature exhibited highest hardness (63.7 HV). Al-25wt % TiC composites with TiC particle size 273.196 nm exhibit the lowest wear rate (0.043 mm^3/s).

Keywords: *: Aluminum Matrix, Composites, Various Average Particles, TiC.*

1. Introduction

TiC reinforced Metal Matrix Composites (MMCs) are increasingly being used in the automobile, aircraft, cutting tools, and space industries. Worldwide attention has been focused on the processing and fabrication of MMCs because of both favourable manufacturing costs and performance [1]. MMCs offer outstanding properties such as high strength-to-weight ratio, high torsion stiffness, good corrosion resistance and versatility to the designer. Two major processing techniques that have been found suitable for these composites are powder metallurgy and solidification processing [2]. As investigated by Yang *et al.* [3], hard TiC particles help to improve the soft matrix in terms of hardness and wear resistance the improvement depends on the amount and uniformity of distribution of particles of TiC, and the strength of the particle-matrix boundary and the mechanical properties of the matrix. The Al-TiC composites occupy a unique position in the family of metal matrix composites due to their excellent wear/stiffness, strength-to-weight ratio with good mechanical properties. The most common applications of these composites are in commercial aerospace, space technology, automobile, general industrial and engineering structures. Typical examples are found in helicopter blade, automotive piston, engine block, cylinder liners, motorcycle brake disk, and valve engines [4-7]. Powder metallurgy technique is a promising technique for fabricating the composites of Al reinforced with a hard ceramic phase. TiC particle-reinforced MMCs have been developed by many researchers because of the thermodynamically stability of TiC and the hardness and low density which it imparts to the composite [8, 9]. The Al-TiC composite system has been studied by a number of researchers and has been found to possess good strength and stiffness. It has been reported that the TiC reinforced Al matrix composites exhibit higher stiffness and ductility than TiB_2 reinforced composites. This may be attributed to the stronger interfacial bonding in the Al-TiC system due to the increased tendency for nucleation of solid on the particle surfaces [8-10]. Solay and Mohan studied the effect of varying weight percent of particulate TiC (ranges from 1% to 10%) reinforcement with elemental 6061 Aluminum alloy on mechanical properties (hardness and tensile strength) of specimens processed through powder metallurgy, compaction pressure from 125 MPa to 175 MPa, the density, hardness and tensile strength value increases[12]. Kennedy and Wyatt reported that the interfacial bond strength in Al–TiC MMCs varies significantly with manufacturing method (flux-assisted casting method, powder metallurgy followed by hot isostatic pressing (PM-HIP), extruding mixed powders at slow (PM-EXTS) and fast (PM-EXTF) ram speeds.

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Conventional mechanical testing (elastic modulus, tensile, and extrusion) was performed on composite materials. Tong and Ghosh evaluated the room and elevated temperature mechanical behavior (tensile properties) of Al/TiC, high-strength Al-Si/TiC and the elevated temperature-resistant Al-Fe(-V-Si)/TiC composites [13]. Zhang *et al*. studied in situ process-Reaction synthesis used to fabricate Al/TiC composites, the phase constitute, microstructure and mechanical property of the Al/TiC composite [14]. Therefore, in the present paper, Al-TiC composites and examined the effects of sintering temperature, ratio and particles size of TiC on microstructure, density, hardness, and wear behaviour in detail.

2. Experimental Procedure

The aluminum powder used was of 99.9% purity (purchased from Fluka) and average particle size of less than 47.5 µm. Different average particle sizes (273.196 nm, 194.732 nm, 149.071 nm) of TiC (99.9% purity, purchased from Fluka) were prepared by ball milling. Mixtures Al powder with different average particle sizes and different compositions (5%, 15%, 25%wt TiC) were prepared. Mixtures were subjected to normal mixing for 2 h at the speed of 20 rpm then compacted into pellets by 750 MPa applied for 3 min. 0.5g of the powder mixture was pressed into 1 pellet in a steel mold of 10mm internal diameter. Zinc stearate was used as a mould wall lubricant. The green compacts of Al-TiC composites were sintered by tube furnace under protective argon gas atmosphere for 3 h at 500, 550, and 600^0C. SEM technique was used for microstructure examination of the raw powders, and Al-TiC composites. The hardness test of the sample was done by using micro hardness Vickers, the micro-Vickers hardness was measured at 5 points from the along the surface of the samples. Dry sliding wear tests were performed by a pin-on-disc apparatus. Sliding velocity was 150 rpm with abrasive paper under a constant load. The abrasive paper was replaced with a new one after every test. Each test was repeated three times and the average value was taken.

2.1. *Characterization*

Aluminum and titanium carbide powders were used as a raw material. Morphology and physical characteristics of the powder were identified with scanning electron microscopy (SEM) and X-ray diffraction (XRD) techniques.

2.2. SEM Images Analyses

Morphology and microstructure of the initial (as received) powder particles used are given in Figures 1 (a) and (b).

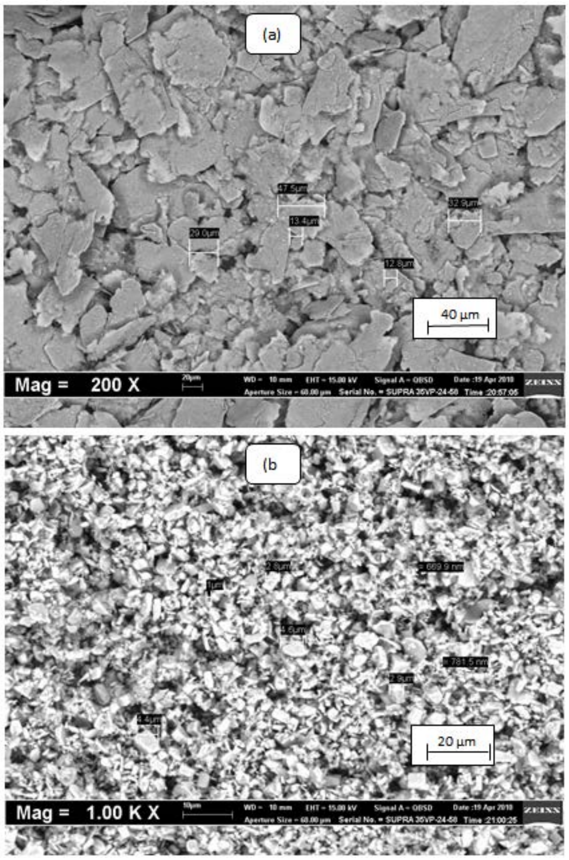

Figure 1. SEM images for (a) as received Al powder (b) received TiC powder

From SEM image in Figure 1, the size distribution of aluminium particles mostly lies in the range of 29.0-47.5 µm with some fine particles in the range of 12.8-13.4 µm. It also shows that the Al particle is in a flaky shape. The particle size distribution of as received TiC powder exhibits that most (~75%) of the particles are in the range of 781.5 nm to 2.9 µm. Some TiC particles are agglomerated due to their fineness and are in the range of 4.4 to 4.6 µm. The image shows that the TiC particle is in an angular shape with sharp edges. The titanium carbide powder was milled for different duration (5h, 10h, and 15h). SEM images of the TiC powders after different milling time (5, 10 and 15h) are shown in Figure 2. (a, b and c), respectively.

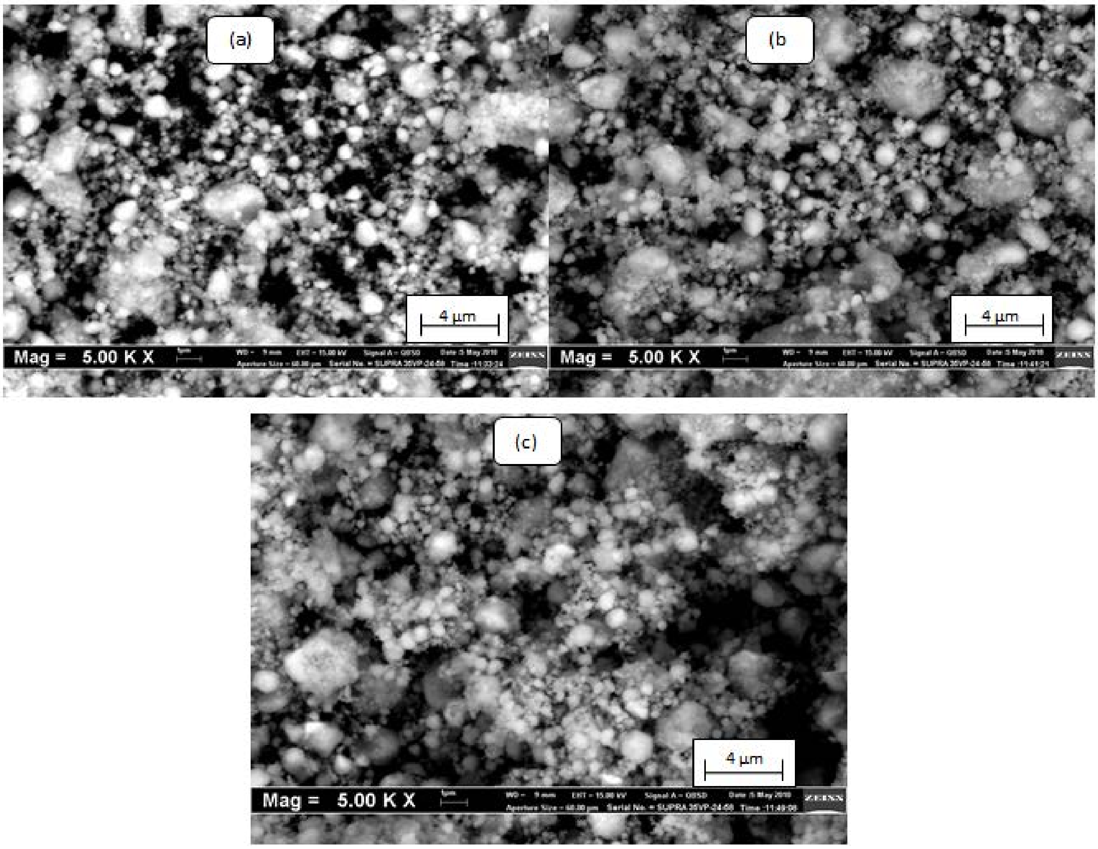

Figure 2. SEM images for TiC after milling for (a) 5 h, (b) 10 h and (c) 15 h

Figure 2 clearly reveals that large particles are in fact agglomerates of much smaller particles especially after 5 to 15 h milling time due to during the milling process; the particles are deformed, cold welded and fractured due to high energy collision [15]. Those events may change particle shape and also decrease particle size and form layered structure [16- 18]. In addition, the aluminum is generally a ductile material and it shows a highly non-linear stress-strain relationship up to the maximum strength with lower Young modulus.

3. Results and Discussion

3.1. X-Ray Diffraction and Rietveld Refinement Analyses

The aluminum and titanium carbide as received powders were characterized by X-ray diffraction. The identification of the phase of Al and TiC powders were done through the matching of diffraction patterns of powders with the International Center for Diffraction Data (ICDD). In the XRD patterns of aluminum and titanium carbide clearly a few main peaks of these powders are visible, as shown in Figure 3(a) diffraction patterns relative to Al powder exhibited peaks at 38.5^0, 45.8^0, 65.1^0, 68^0 and 62.5^0 corresponding, respectively to the (111), (200), (220), (311) and (222) reflections of F.C.C, and as shown in Figure 3(b) diffraction patterns relative to TiC powder exhibt peaks at 48.5^0, 54.8^0, 65^0, 78.2^0 and 82.4^0 corresponding, respectively to the (111), (200), (220), (311) and (222). The peaks in these patterns gave a good match with the references patterns (ICDD No. 3828). Figures 3 (a) and (b) show the diffraction pattern of Al and TiC powders respectively. From Figures 3 (a) and (b), all the starting powders were pure and so no impurities in these starting powders were visible.

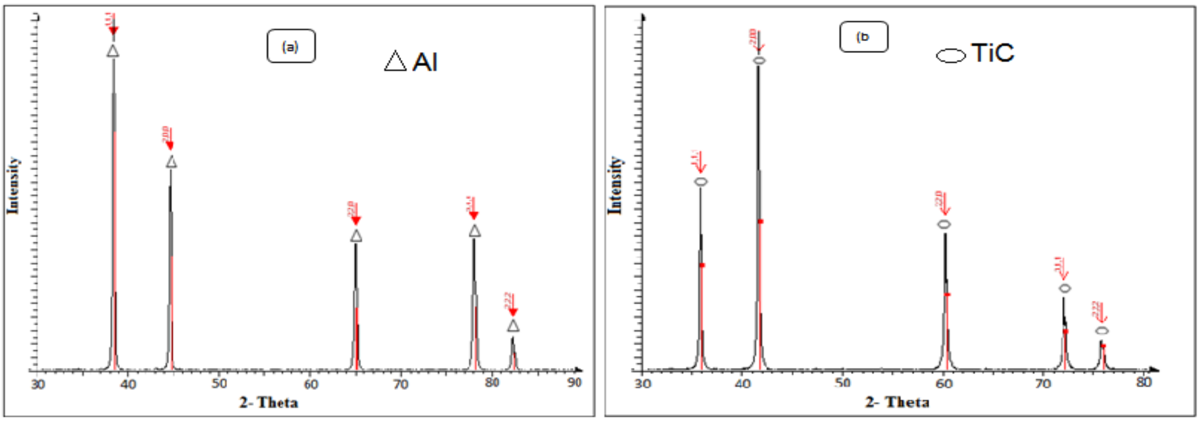

Figure 3. XRD pattern of as received (a) Al powder (b) TiC powder

The titanium carbide powder after milling for different duration (5h, 10h, and 15h) was characterized by XRD in order to study the effect of milling time on the particle size. The XRD patterns of TiC powder with different milling time are plotted in one graph, as shown in Figure 4.

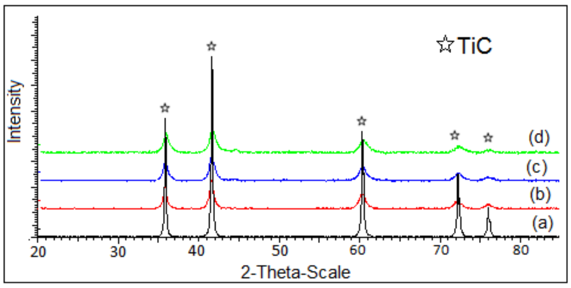

Figure 4. XRD patterns for TiC powder after milling for: (a) 0 h, (b) 5 h, (c) 10 h, and (d) 15 h

It is apparent that with increasing the milling time, the diffraction peaks for TiC powder become broader and their relative intensity decreases. During the milling process, particles underwent collision between balls and between balls to walls, so the particle size decreases for longer milling time. Average crystallite size for TiC before and after milling for 5h, 10h and 15h were 47.3, 17.4, 15.8 and 12.4 nm, respectively and average particles sizes of TiC 273.196 nm, 194.732nm, and 149.071 nm. The crystallite size and average particles of TiC decreased with increasing milling time due to large and continuous number of dislocations resulting from heavy deformation caused by high energy mechanical milling. The average crystallite size of TiC was estimated by using the Scherrer formula, $D = k/ B\cos\theta$, where D is the average crystallite size, k is the CuKa wave length, B is the diffraction peak width at half-maximum intensity and θ is the Bragg diffraction angle.

Basically, after the composites were sintered under argon atmosphere for 3 h, they were characterized with the X-ray diffraction technique. This process is to check the phases in the composition of the final product, as well as to check the stability of the raw materials used in the sample. This analysis was necessary in order to confirm that there was no formation of undesirable substance such as Al_4C_3, Al_2O_3, etc. The XRD patterns of Al based composites reinforced with 5, 15, and 25 wt% of TiC are shown in Figure 5 (a), (b), and (c), respectively.

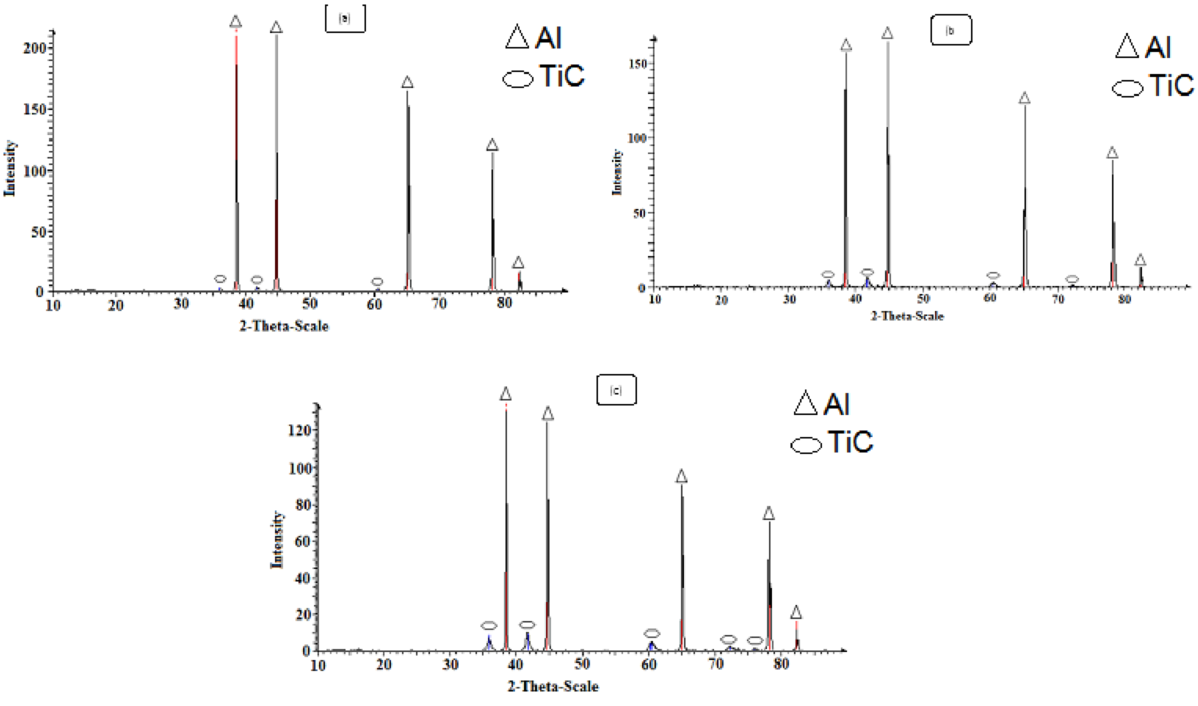

Figure 5. XRD patterns of (a) Al-5%wt TiC, (b) Al-10%wt TiC, (c) Al-25%wt TiC

Based on the XRD result, it is shown that the aluminum-titanium carbide powders do not have reaction with each other after sintering, and these two powders maintain their own intensities (no new phase formation). Furthermore, no peaks from the Al_4C_3 phase, which is often formed as an undesirable reaction product in Al-TiC composites, and no peaks from oxides, such as Al_2O_3, could be seen. All the samples show the presence of TiC peaks along with Al peaks. It is clearly observed that the intensity of TiC peaks has increased with increase in TiC content from 5% to 25%.

3.2. *SEM Images Analyses*

The distribution of the reinforcement (TiC) particles in the aluminum matrix has been observed by scanning electron microscopy (SEM). Figure 6 (a), (b), and (c) show the SEM micrograph of aluminum composites reinforced with 5, 15, and 25 wt% of TiC with particle size 149.071 nm.

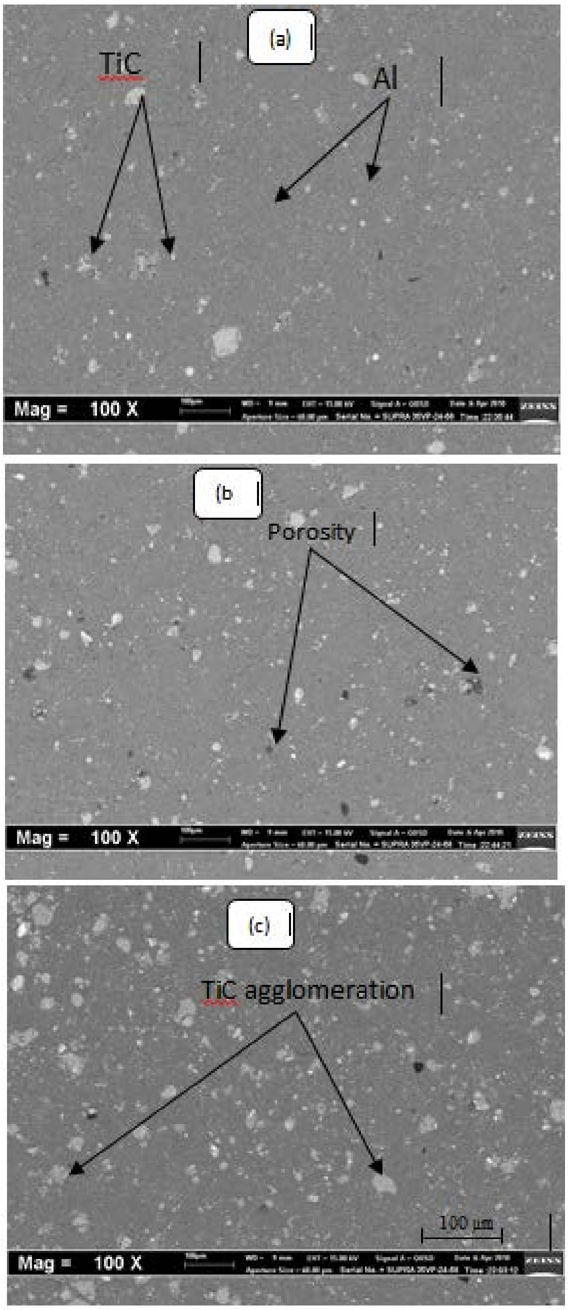

Figure 6. SEM images of the composites after 600^0C sintering temperature (a) Al-5%wt TiC, (b) Al-15%wt TiC, and (c) Al-25%wt TiC

As shown in Figure 6, the small particles are distributed homogenously between the big particle (agglomeration of many small particles together) and the particle size of the small particles is uniform. However, some pores can be observed. The formation of pores is mainly due to the non uniformity of the initial powder particles. Also, it is clear from Figures 6 (a, b and c) that the reinforcement particles of the composites are embedded in the aluminum matrix. A small agglomeration of TiC particles in the aluminum matrix has been noticed and this is mainly due to non-homogeneity involved in the mixing and blending process carried out before sintering. The microstructure evaluation also shows that, for a given series of composites, the size of the TiC particles in the composites increases as the TiC content increases.

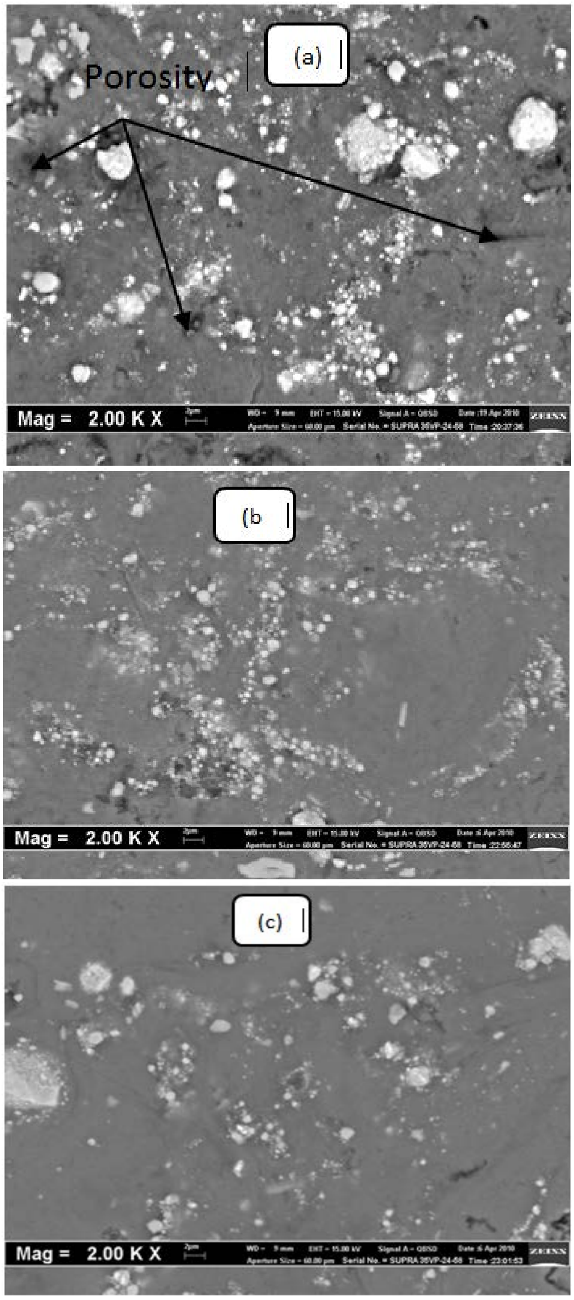

Figure 7. SEM images of the Al-25wt% TiC composite at different sintering temperatures

Figure 7 shows the SEM images of aluminum matrix composite reinforced with 25wt. % of TiC (particle size 149.071 nm) at different sintering temperatures.

From Figures 7 (a, b, and c), it is observed that the sintered surfaces were porous and the composite has small grains of TiC when it is sintered at 500ºC. With the increase in sintering temperature, the number of pores decreased and the rate of grain growth apparently increased. Moreover, significant grain growth was observed and the pores were almost eliminated at 550 ºC. Also the microstructural study revealed that TiC clusters and TiC-free regions were formed after consolidation at the high sintering temperature (600ºC). It seems that the aluminum matrix was locally moved during the consolidation to fill the voids (Kamrani *et al.*). Kamrani *et al.* suggested that the diffusion of the matrix into the interparticle pores is responsible for this observation.

Similarly, it is suggestible that the high diffusivity of the aluminum matrix close to the melting point caused inter-particle pore diffusion, causing the TiC clusters and TiC-free areas [19].

3.3. Effect the Particle Size of TiC on Density, Hardness, and Wear

The average particle size of TiC with different milling time was estimated by using the following formulas [20, 21]:

$$\text{Particle size} = \frac{6}{Density \times S.S.A}$$

Average particle size = ∑ Particle size / Number of calculation for Particles size

The effect of the particle size on the density of the Al-25wt% TiC composite is shown in the Figure 8.

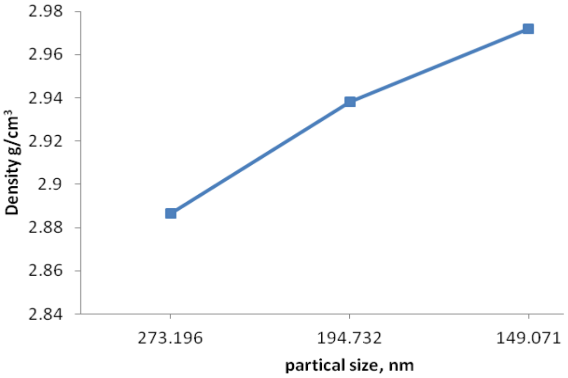

Figure 8. Effect of particle size of TiC on the density of Al-25wt.% TiC composites

The density of Al-TiC composites was determined according to Archimedes' principle.

The result indicates that the density of Al-TiC composites which were reinforced with TiC (149.071 nm) achieves the highest value of density. Meanwhile, composites, which were reinforced with TiC (273.196 nm), have the lowest density. The results obtained reveal that the densities of sintered composites show a marginal increase with the decrease in the particle size. On the other hand, the coarser particles lead to bigger voids which decreasing the density [20]. The volume of voids is affected by the relative particle size and weight fraction of TiC. The size of TiC particle was found to have influence in determining the hardness of the composites. The smaller the reinforcement particle size, the higher the hardness would be. Figure 9 illustrates the relationship between hardness and particle size.

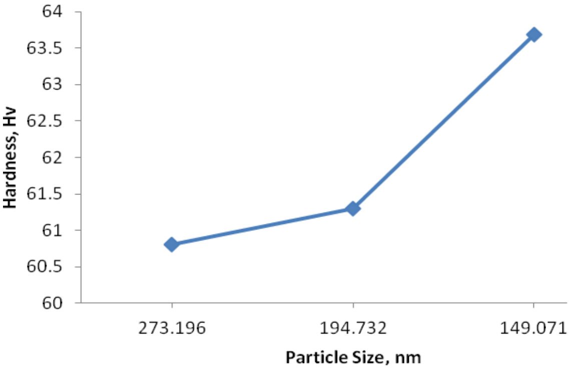

Figure 9. Effect of particle size of TiC on the hardness of Al-25wt.% TiC composites

The result indicates that the hardness increases with decreasing the particle size. At 15 h of milling time (149.071 nm), the microhardness achieves the highest value of 63.7 HV. Meanwhile, composites which were reinforced with TiC milled for 5 h (273.196 nm) have the lowest hardness value of 60.8 HV. Small reinforcement particles permit larger contact area with aluminum particles. On the other hand, large reinforcement particles have small area of contact and prevent the diffusion process from progressing [23]. The TiC particles act as barriers to dislocation flow in aluminum matrix. Composites reinforced with smaller TiC particles have a higher number of barrier per unit area compared to composites reinforced with larger particles at the same weight percentage [22, 24]. The particle size of the reinforcement is one of the intrinsic factors that could have an effect on the wear resistance besides other mechanical properties of particulate reinforced AMCs. Figure 10 shows the effect of particle size of TiC on the wear rate.

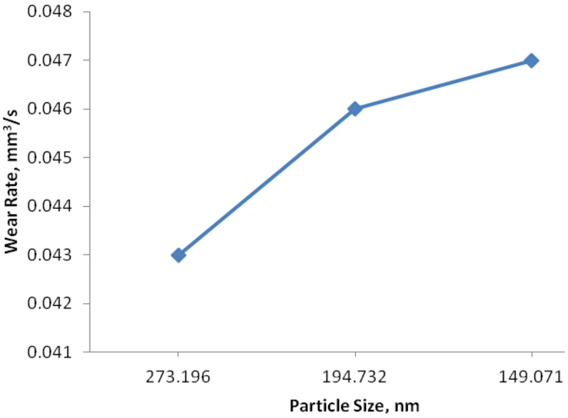

Figure 10. Effect of particle size of TiC on the wear rate of Al-25wt.% TiC

The wear resistance of metal matrix composites is known for improving considerably with increasing the size of reinforcing particles. Figure 10 shows that the composites with 5 h milling (particle size 273.196 nm) exhibit the lowest wear rate (0.043 mm^3/s). Meanwhile, composites which were reinforced with TiC milled for 15 h (particle size 149.071 nm) have the highest wear rate value (0.046 mm^3/s). During the friction process bigger TiC particles on the friction surface are impressed deeper to the tested surface and are not easily releasable from the surface, and simultaneously they reinforce the composite surface. On the other hand, smaller TiC particles on the friction surface are easily released from the surface of composite and become constituent of mobile friction layer, which abrade the friction surface of tested samples. This result is in agreement with those reported earlier [25]. The larger size of reinforcing particles can offer protection to the matrix during sliding. Once the reinforcing particles fracture or loosen from the matrix, they can be removed easily from the matrix, resulting in a certain amount of material loss. In order to investigate the wear mechanism, the surfaces of the worn composites were examined under SEM. Figures 11 (a- c) show typical worn surfaces of the Al-5, 15, and 25 wt.% TiC composites, respectively. The worn surface of the composites (Figures 11 (a- c)) shows a small plough groove and a little dimple. The abraded surfaces of the composites also show scratches on the worn surface, and there is little evidence of particulate fracture, even in composites with the highest weight fraction. The reason for this is explained as follows: when the surface of the composite initially comes in contact with abrasive paper, adhesive contact occurs; the TiC abrasive particles with sharp edges then cause microploughing and grooving in the surface of aluminum matrix; therefore, materials, in the form of chips, are removed from the grooves, thereby exposing TiC particles; thus, the increase in the weight fraction of TiC particles appears to reduce the severity of grooving and plastic deformation; this is because the hard particulate phase is well bonded by Al-based matrix and strong enough to withstand abrasive wear induced by the counter wear ring at applied load [4, 24].

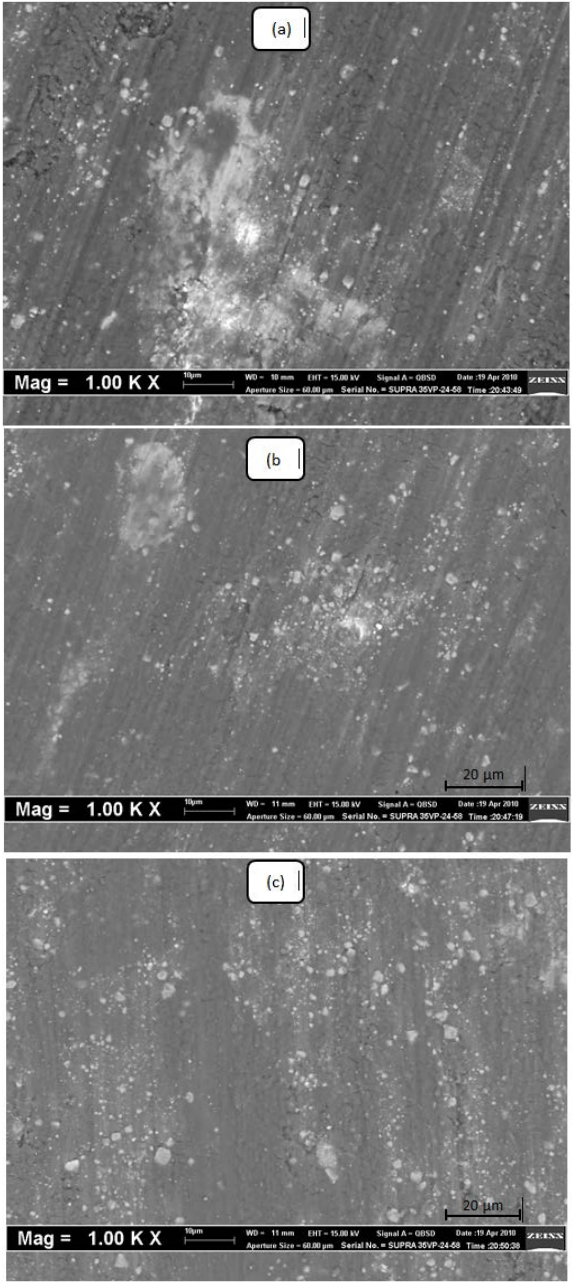

Figure 11. SEM images show worn surface of Al-TiC, (a) Al-5wt% TiC, (b) Al-15wt% TiC, (c) Al-25wt% TiC

4. Conclusions

In summary, we have obtained the Al-TiC composites by various average particles sizes of TiC Nano-particles, different weight ratios of TiC, and different sintering temperature. In general, the aluminum is ductile material and shows a highly non-linear stress-strain relationship up to the maximum strength with lower Young modulus. The crystallite size and average particles of TiC decreased with increasing milling time due to large numbers of dislocations resulting from heavy deformation caused by high energy mechanical milling. The coarser reinforcement particles, bigger voids formed leading to a lower density. Composites reinforced with smaller TiC particles have higher number of barrier per unit area compared to composites reinforced with larger particles at the same weight percentage. With the increase in sintering temperature, the number of pores decreased and the rate of grain growth apparently increased. The results obtained reveal that the densities of sintered composites increase with the decrease in the particle size. Al-25wt. % TiC composites (particle size of TiC =149.07 nm) with 600^0C sintering temperature exhibited highest hardness (63.7 HV). Al-25wt. % TiC composites with 273.196 nm TiC particle size exhibit the lowest wear rate (0.043 mm^3/s).

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