EFFECT OF PARTICLE SIZE DISTRIBUTION AND MIXING HOMOGENEITY ON MICROSTRUCTURE AND HARDNESS OF SINTERED AL-TiC NANO COMPOSITES

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ABSTRACT

Aluminium matrix infused with titanium carbide (TiC) particles was prepared by means of powder metallurgy process. The effect of TiC addition on microstructure, hardness and electrical conductivity of as-sintered Al-TiC with 2 μ m and $\leq$ 200 nm reinforcement were investigated. Microstructure studies reveal the even distribution of TiC particles in the aluminium matrix. With increasing addition of TiC, the hardness of composites increased after the 10% and suddenly reduced because the composites reduce its resistance and create more sites for crack initiation. Hardness and electrical conductivity of composites have been varied based on particle size variation of TiC.

KEYWORDS: *Microstructure, Al-TiC, Hardness, Density, Metal Matrix Composites & Electrical Conductivity*

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1. INTRODUCTION

Metal matrix composites (MMCs) are the focus of intense research and development worldwide for many industrial branches, where reduction in weight of elements along with the improvement in specific modulus, strength, thermal stability, wear resistance, and service life are required [1-3]. Nanoparticles-reinforced aluminium matrix composites (AMCs) are a new class of nanostructured materials, consisting of ceramic nanoparticles used as reinforcements. Various kinds of ceramic nanoparticles, E.g., TiC, SiC, B₄C, Al₂O₃ and MgO have been added to the AMCs to improve the microstructure and mechanical properties. Amongst these nanoparticles, TiC appears to be very popular, because it has a high specific strength, high modulus, low density, good wear resistance, relatively high temperature stability and good wettability with molten aluminium [4]. The aluminium alloy based metal matrix composites are generally preferred for their low density, wide alloying range, capability and response to heat treatment when used for monolithic counterparts and their intrinsic flexibility and responsiveness to processing [5-7].

In the case of Ti matrix composites, a controlled three-dimensional microstructure has the capability to enhance the properties of material, such as elastic modulus, tensile strength and fracture toughness, compared with well-distributed counterparts [8, 9]. Titanium carbide-based composites with nickel alloys and iron alloys are currently used in high performance applications where wear and corrosion are the main sources of material failure [10]. For example, additions of titanium carbide (TiC), one of the hardest refractory metal carbides, with a Vickers hardness of 19.6-31.4 GPa [11], can be expected to improve the abrasion resistance of many different alloy systems. Particle-reinforced metal matrix composite materials are used for structural applications in wear industry due to super toughness and wear resistance. Especially, alumina- and silicon carbide-based composite materials do not lose their hardness and wear resistances during high temperature applications [12-13].

Synthesized nano aluminium and titanium powders were conducted the safety analysis [14]. Nanoparticles have a great thermal behavior while compared with micron sized powder [15]. However, limited work has been reported about the influence of processing parameters on the behavior of alloys and composites fabricated by powder metallurgy [16-20].

Hence the current work is focused on investigating the behaviour of Al-TiC composites. Aluminium metal matrix composites reinforced with two different sizes of TiC particles at 5% were prepared using the powder metallurgy route, and the effect of the TiC content on the microstructure, hardness behaviour of the composites was investigated.

2. EXPERIMENTAL INVESTIGATION

Aluminium and Titanium Carbide powders, utilized in the current research, were procured from M/s. Alfa Aesar, England. Atomized aluminium powder and TiC were acquired with 99% and 99.5% purity with $45\ \mu\text{m}$ and $2\ \mu\text{m}$ average particle sizes respectively. The characteristics of the aluminium and TiC powder are listed in Table 1 & 2. The TiC powder was pulverized in a planetary ball mill for 12 h and the obtained particle size was approximately below 200 nm. The individual powders (Al, TiC) were fine-grained in a planetary ball mill using tungsten carbide grinding medium with Ball Powder Ratio (BPR) 1:20 for further 1.5 h and later it was blended on a mass basis with 5% titanium carbide. Then the combined Al-5TiC powders were mixed systematically in the high energy planetary ball mill for another 0.25 h to attain homogeneous mixture. Figure 1(a) shows the initial aluminium particles at 800X magnification; the particles are spherical in structure. Figure 1(b) shows the TiC particles at 1000X magnification and it has the structure of a pancake. Further Figure 1(c) shows Al-5TiC mixed powder after milling for 0.25 h.

(a) pure Al

(b) pure TiC

c) Al-5TiC mixed powder

Figure 1: (a-c) SEM Morphology

A cylindrical compact (Al-5TiC) was prepared using two separate particle size TiC namely $2\ \mu\text{m}$ & $\leq 200\ \text{nm}$ with 15 mm diameter & aspect ratio 1. This was compacted in a compression testing machine with $1.0 \times 10^6\ \text{N}$ capacity using the die set (figure 2). A compacting pressure of $6.7 \times 10^7\ \text{N/mm}^2$ was applied to acquire the aspect ratio. Zinc-sterate was utilized to lubricate the die set; the density variation and aspect ratio were sustained by mass and compacting pressure. Compacts with Metal Powder Industries Federation (MPIF) standard 42 were utilized [21].

Table 1: Characteristics of Aluminium Powder

	Sieve size (μm)	Wt. %
Aluminium	+106	00.26
	+90	02.54
	+75	14.73
	+63	17.58
	+53	24.86
	+45	12.33
	+38	06.27
	-38	21.42
Apparent density (g. cm^{-3})		1.030
Flow rate, (by Hall flow meter) (50g^{-1})		32.00
Particle size-dry sieve test standard		ASTMB-214

Table 2: Characteristics of Titanium Carbide Powder

Purity %	99.50
Sieve size (μm)	2.00
Density (g. cm^{-3})	4.93
Melting point $^{\circ}\text{C}$	3,140

After compaction, the preforms were removed from the die set assembly and loaded into the furnace for sintering. The sintering was carried out in an inert argon gas (to prevent oxidization) circulating furnace at 575°C for a period of 1 h. After that the sintered preforms were left in the furnace to cool until it reaches room temperature. A fine wire brush was used to clean the preforms after taking out from the furnace. The density of green compacts, as sintered specimens, was determined using the Archimedes principle [22]. Mustard oil was used as the immersion fluid [23]. Three polished samples were randomly weighed using an electronic weight with an accuracy of $\pm 0.001\text{g}$ and were immersed in pure water. Vickers hardness measurements were made on Al-5TiC preforms in sintered conditions with a load of 500g dwelling for 10s.

Figure 2: Compaction Methodology.

The electrical conductivity of Al-TiC composite was evaluated with eddy current instruments in accordance with ASTM standard E1004-02 [24]. The electrical conductivity measured by this equipment is usually expressed as a percentage of the conductivity of the International Annealed Copper Standard (% IACS). For metallographic study, the samples were prepared according to the standard metallographic procedure. X-ray diffraction (XRD), Energy Dispersive Analysis (EDA) and Field Emission Scanning Electron Microscope (FESEM) were used to investigate microstructure and phase composition of composites.

3. RESULTS AND DISCUSSIONS

The Al-TiC composites were successfully produced using the cold pressing method together with a sintering time of 1 h at 575°C, under a compacting pressure of $6.7 \times 10^7 \text{ N/mm}^2$. The theoretical density was measured at 90% in 2 μm & 95% in $\leq 200 \text{ nm}$ composites for the same compaction pressure. Microstructural evaluation studies were conducted by FESEM. The composite specimen showed the uniform distribution of reinforced particles. Observations reveal minimum presence of porosity in the materials [25]. Figure 3 & 4 shows the FESEM microstructure of the sintered composite samples of Al-5TiC with 2 μm & $\leq 200 \text{ nm}$ TiC particles respectively. A close observation of the above micrographs indicates the even distribution of TiC particles in the aluminium matrix Al-5TiC ($\leq 200 \text{ nm}$) composites when compared to Al-5TiC (2 μm) which shows slight agglomeration. It is very important to obtain homogeneous distribution of reinforcement in the matrix in order to enhance mechanical, electrical and thermal properties [26].

Figure 3: FESEM Image of Composite Mixture of Al-5TiC (2 μm).

Figure 4: FESEM Image of Composite Mixture of Al-5TiC ($\leq 200 \text{ nm}$).

The XRD analysis was performed for Al composite with 2 μm and 200 nm in order to determine whether a phase formed to provide bonding in the interface of aluminium (matrix) and titanium carbide (reinforcement) particles or not. The studies on made-up materials [Figure 5 & 6] point out the incidence of phases such as Al, TiC. XRD analysis of the sintered samples reveals the absence of any intermetallic, and reaction phase in the composites. The phase progression in the sintered composites was the same irrespective of the amount of TiC reinforcement. The diffraction patterns of the Al-TiC composites exhibits a variety of peaks matching to the Face Centred Cubic (FCC) phase of aluminium and TiC peaks to that of JCPDS file numbers 04-0787 & 32-1383 respectively. Figure 5 & 6 shows the Al reflecting planes (111), (200), (220) & (311) and TiC reflecting planes (111), (200), (220) & (311) obtained during XRD [31]. Energy Dispersive Analysis (EDA) results are exhibited in Figure 7 & 8. Figure 7 shows an enhanced the incidence of Al and O in the pure aluminium specimen. Figure 8 shows the incidence of Ti, Al and C particles in the fabricated Al-TiC composite.

Figure 5: XRD Pattern of Various Composite of Al-5TiC (2 μ m).

Figure 6: XRD Pattern of Various Composite of Al-5TiC (≤ 200 nm).

Figure 7: EDA Spectrum of a Aluminium Composite After Sintering.

Figure 8: EDA Spectrum of an Al-5TiC Composite after Sintering.

Vickers hardness measurements were made on Al-5TiC samples in sintered conditions with a load of 500g dwelling for 10s. At least three measurements of hardness were performed on each sample and the average values are shown in table 3.

Table 3: Vickers Hardness of Al-5TiC Sintered Composite	
Vickers Hardness (Hv) 500g,10s	
TiC of ≤ 200 nm size TiC of 2μ m size	
41.5	34.5
44.6	36.4
45.6	36.2

Figure 9: Hardness Value of Various Composites.

A Vickers hardness test was carried out on the composite samples (Table 3) and it shows an increase in the hardness value of the composite samples as the particle size of titanium carbide reduces. The work piece used in this experiment was Al-5TiC composites of TiC particles reinforcements (2 μ m and ≤ 200 nm). Figure 9 reveals the manner by which the diffused nano-TiC reinforcement led to a substantial rise in hardness of aluminium nano composites. This is

credited to the occurrence of the moderately reduced grain size, harder ceramic TiC particulates in the matrix, their powerful resistance on the ductile aluminium matrix. Further observation indicates that, the preforms with higher relative density (>90%) possess better hardness compared to less relative density (<90%) preforms. The hardness of the composites decreases with an increase in particle reinforcement above 10%. The composites reduce its resistance and create more sites for crack initiation and hence lower down the load-bearing capacity of the reinforcement [27-33]. The particles are no longer isolated with the ductile aluminium alloy matrix since the number of contacts between TiC particle increases. Therefore, cracks will not get arrested by the ductile matrix and propagate easily between titanium carbide particulate [31].

Table 4: Electrical Conductivity of Al-TiC Composites

%TiC	Electrical conductivity % IACS	
	TiC of $\leq 200\text{ nm}$ size	TiC of $2\text{ }\mu\text{m}$ size
0	61	61
5	52.5	53.2
10	44.6	45.2
15	39.5	40.2

Table 4 illustrates the results of electrical conductivity of Al-TiC composites, produced via compacting method, based on content of titanium carbide with $2\text{ }\mu\text{m}$ & $\leq 200\text{ nm}$ respectively. With the increase in addition rates of titanium carbide, the electrical conductivity of composites decreased. While the electrical conductivity of Al with no addition was measured as 61% IACS, the electrical conductivity of aluminium matrix Al-5TiC ($2\text{ }\mu\text{m}$) composites with 5, 10 and 15%TiC addition was measured as 53.2, 45.2 and 40.2% IACS, and the electrical conductivity of aluminium matrix Al-5TiC ($\leq 200\text{ nm}$) composites with 5, 10 and 15%TiC addition was measured as 52.5, 44.6 and 39.5% IACS, respectively.

Porosity and oxidation could be asserted as the reason for low electrical conductivity of pure Al [32]. Interaction between free electrons and nucleus is weak in the metals. Therefore, electrons easily move and accordingly electrical conductivity of metals is good. However, electrons are firmly bonding to the nucleus in the carbides and electrons do not move. For this reason, electrical conductivity of carbides is weak [33]. Since rate of Al matrix in TiC addition in the Al-TiC composite decreased, it is an expected result that electrical conductivities of the composites would decrease with increasing addition of TiC. Reinforced TiC particles in the composite exhibited an effect inhibiting the movement of Al electrons.

The decrease of electrical conductivity with increasing the TiC content may be related to the following several aspects. Firstly, the increase of TiC content can certainly lead to the decrease of the electrical conductivity due to the intrinsic high electrical resistivity of TiC. Secondly, with increasing the TiC content, the density of dislocations induced by the difference of thermal expansion coefficients between TiC and Al matrix increases. Increased dislocation density intensifies the scattering of electrons on interface Al-TiC, resulting in decreased electrical conductivity. And finally, the presence of TiC can effectively refine the grains of Al-TiC composite. Further observation indicates that, the electrical conductivity in the aluminium matrix Al-5TiC ($\leq 200\text{ nm}$) composites shows slight reduction compared to Al-5TiC ($2\text{ }\mu\text{m}$) because the strength and hardness of the composites increase with the increasing amount and size variation of particle reinforcement.

4. CONCLUSIONS

From the investigation of experimentation, it is concluded as below:

- Mechanical alloying method was used to prepare preforms with improved density (90%-95%) using mixture of Al & TiC powders.

- Microstructure studies reveal that TiC particles were distributed uniformly in the Al matrix.
- The presence of Al and TiC was confirmed by X-ray diffraction analysis. XRD analysis of the sintered samples reveals the absence of any intermetallic, and reaction phase in the composites.
- With increasing addition of TiC, the hardness of composites increased after the 10% and suddenly reduced because the composites reduce its resistance and create more sites for crack initiation.
- High TiC content decrease the electrical conductivity of Al-TiC composites as expected. The highest electrical conductivity for Al-TiC (2 μm) composites was obtained in the Al-5%TiC composite, with approximately 53.2 % IACS.

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