Characterization of Hybrid Silicon Carbide and Boron Carbide Nanoparticles-Reinforced Aluminum Alloy Composites

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Abstract

Hybrid nanocomposites based on aluminum alloy 6061 reinforced with different hybrid ratios of SiC (0.5, 1.0 and 1.5 vol. %) and B 4C (fixed 0.5 vol. %) nanoparticles were successfully fabricated using ultrasonic cavitation based solidification process. The fabricated cast specimens were characterized using SEM study with EDS analysis, hardness test, tension test and impact test. The results indicate that, by the ultrasonic cavitation effects namely transient cavitation and acoustic streaming, the nano reinforcements were successfully incorporated in the aluminum matrix. SEM study with EDS validates the presence of SiC and B 4C nanoparticles in the aluminum matrix. Compared to the un-reinforced alloy, the room temperature hardness and tensile strength of the hybrid composites increased quite significantly while the ductility and impact strength reduced marginally. The combination of 1.0 volume percentage SiC and 0.5 volume percentage B 4C gives the superior tensile strength. The major reason for an increase in the room-temperature mechanical properties of the hybrid composites should be attributed to the larger hybrid ratio of SiC and B 4C nanoparticles, the coefficient of thermal expansion mismatch between matrix and hybrid reinforcements and the dispersive strengthening effects.

Keywords: Hybrid Composites; Ultrasonic Cavitation; Nanoparticle Dispersions; Microstructural Studies; Mechanical Properties

1. Introduction

Conventional monolithic materials have limitations in achieving good combination of strength, stiffness, toughness and density. To overcome these shortcomings and to meet the ever increasing demand of modern day technology, composites are the most promising materials of recent interest [1, 2]. Metal matrix composites (MMCs) possess significantly improved properties including high specific strength; specific modulus, damping
capacity and good wear resistance compared to the unreinforced alloys. But the poor ductility and reduced fracture toughness limits the applications of conventional MMCs. To improve the ductility and fracture toughness of the conventional composites, the new class of materials known as Metal Matrix Nanocomposites (MMNCs) is developed by reinforcing particles in the nanometer scale [1, 2].

The current nanomanufacturing methods are not suitable for the bulk production of MMNCs. Ultrasonic cavitation effects namely; transient cavitation and acoustic streaming could be useful for dispersing nano ceramic particles in liquid melt. Transient cavitation is the formation and collapse of thousands of cavitation bubbles in liquids [3-5]. The collapsing of cavitation bubbles in liquids develops enormous amount of energy and this energy could be used for variety of purposes including homogenizing, grain refinement, gas removal and dispersing [6].

SiC and Al2O3 are the common reinforcing materials used in aluminum matrix composites [7]. Limited research has been conducted on nano B4C reinforced aluminum matrix composites due to the higher cost of B4C powders. But B4C is an attractive reinforcement material because of its excellent chemical and thermal stability; most importantly, B4C has lower density, higher hardness and good bonding property with aluminum relative to Al2O3 and SiC [7, 8].

Hybrid composites are those composites which have a combination of two or more reinforcements. The behavior of hybrid composites is a weighted sum of the individual components in which there is a more favorable balance between the inherent advantages and disadvantages [7, 8].

The aim of the work presented here is to investigate the possibility of combining the nano SiC and nano B4C particles with aluminum alloy (AA) 6061 to form a lightweight, high performance hybrid metal matrix nanocomposite materials. Particular attention is given to characterize the mechanical properties of these hybrid materials.

2. Materials and Methods

AA 6061 with density 2.7 g/cm³, tensile strength 310 MPa (T6 condition) and modulus of elasticity 70 GPa was used as a matrix material. 6061 is a precipitation hardening aluminum alloy, containing magnesium and silicon as its major alloying elements. It has significant applications in aircraft, marine and automobile industries.

The mixture of nano SiC and nano B4C particles were used as the reinforcement material. Three different volume percent of nano SiC (0.5 %, 1.0 % and 1.5 %) and fixed volume percent of nano B4C (0.5 %) were used in the experiments. Properties of matrix and reinforcements are shown in Table 1.

| Material | Density (g/cm³) | Modulus of Elasticity (GPa) | Co-efficient of Thermal expansion (m/m·°C) | Fracture Toughness (MPa·m) |
|----------|----------------|-----------------------------|------------------------------------------|----------------------------|
| AA 6061  | 2.72           | 70                          | 25×10⁻⁶                                  | 29.1                       |
| SiC      | 3.20           | 410                         | 4×10⁻⁶                                  | 4.6                        |
| B4C      | 2.51           | 450                         | 5×10⁻⁶                                  | 3.5                        |

The novel ultrasonic cavitation process was used as a fabrication method. In the ultrasonic cavitation process, transient micro hot spots with a temperature of about 5,000°C and a pressure above 1,000 atm can be formed. The strong heating and cooling rates during the process is composed of hot spots that can break the nano-particle clusters and clean the particle surface [9-11].

The cast samples were characterized mechanically by conducting hardness test (ASTM E10), tension test (ASTM E8) and impact tests (ASTM E23). Tensile tests were performed on INSTRON make universal tension testing machine and impact tests were performed on YAMA make impact testing machine. Field Emission Scanning Electron Microscopy (FESEM) was carried out to analyze the particle distribution patterns in the composites. Energy Dispersive Spectroscopy (EDS) study was used to identify the various elements present at the different parts of the sample. SEM and EDS was conducted on CARL ZESIS SUPRATA Field Emission Scanning Electron Microscope (FESEM).
3. Experimental Procedure

The experimental setup for manufacturing the hybrid composites is shown in Fig. 1 (a). The setup consists of non-ferrous melting furnace, ultrasonic wave generator with transducer and an argon gas supply unit. The specially designed SS crucible used for melting the aluminum is shown in Fig. 1(b). The handle provided in the crucible facilitates the easy pouring of the molten material in to the pre-heated steel die. The ultrasonic processing device consists of a transducer with a rated power 2 kW and the fixed frequency of 20 kHz. During ultrasonic processing, intermittent vibrating mode with pulse ON and OFF time fixed at 1 s and 0.5 s was used for all the experiments.

The required amount of AA 6061 was melted in a resistance furnace at 680°C and choked for about ten minutes. Argon gas is passed continuously to prevent the oxidation of aluminum. The ultrasonic probe made of titanium alloy (Ti6Al4V) was dipped into the melt for about 30 mm height. The top portion of horn connected to the transducer as shown in Fig. 2 (a). Initially, alloy melt was stirred mechanically to get the homogenous mixture then, the reinforcement mixture (nano SiC and nano B₄C) was added slowly from the top of the melt. Mechanical stirring was continued to get the primary dispersion of nanoparticles. After this, the stirrer was removed and ultrasonic horn dipped in the melt. The melt was ultrasonically processed for about 60 minutes before the ultrasonic probe was removed. Then the melt was cast into a steel permanent mold (150×100×15 mm) which was preheated to 500°C. The solidified composites are shown in Fig. 2 (b). The addition of nanoparticles significantly increases the viscosity of the melt; hence after efficient ultrasonic processing higher casting temperature of 850°C was used for the better flowability. For comparison, an AA 6061 alloy sample was also cast under the same conditions.

During experimental work, the reinforcements were added slowly to obtain better dispersion. Proper particles feeding techniques are necessary to further improve the dispersion.
4. Results and Discussions

During the experiments for the hybrid combination of nano SiC 1.5 vol. % and nano B₄C 0.5 vol. % the mixing was a major problem. This is due to, the more the particles addition more will be the agglomeration of particles due to the cohesive nature of the particles. While the particles were fed into the Al melt, they tended to float on the surface due to the high surface tension of the melt. High intensity ultrasonic cavitation effects such as transient cavitation and acoustic streaming ensures the incorporation hybrid reinforcements and their uniform distribution in the melt.

4.1 SEM and EDS

Low resolution (10,000 X) SEM image of the AA 6061/ (0.5 vol. % SiC + 0.5 % vol. % B₄C) is shown Fig. 3 (a). It indicates the nearly uniform distribution of nanoparticles in the aluminum matrix, but at this magnification it is very difficult to identify the SiC and B₄C phases. Hence, Field emission HRSEM (2,79,000 X) was carried out on the sample AA 6061/ (0.5 vol. % SiC + 0.5 vol. % B₄C) and the image is shown in Fig.3 (b). This image also indicates the relatively good dispersion of SiC and B₄C nanoparticles in the aluminum matrix. In this image B₄C nanoparticulates are seen as dark phases and SiC nanoparticulates are seen as grey phases in the AA 6061 matrix structure.

Under the transient cavitations (formation, growth, pulsation and collapse of bubbles) and acoustic streaming (liquid metal flow due to acoustic pressure gradient) effects produced by the high intensity ultrasonic treatment, the clustered nanoparticles are separated and dispersed uniformly. The uniformly distributed particles can be considered as the main prerequisite for achieving good mechanical properties.

The SEM images of the tensile fractured surface of the hybrid composites are shown in Fig. 3 (c) and 3 (d). Fig. 3-c shows the SEM image of the tensile fractured surface of the 1.5 vol. % SiC and 0.5 vol. % B₄C hybrid composites at 400 X magnification. This image shows the shrinkage cavities. These cavities are formed due to the presence of SiC and B₄C nano reinforcements in the matrix structure. The fractured particles as well as interface decohesion can cause voids in the matrix which may result in the reduced tensile strength in the 1.5 vol. % SiC and 0.5 vol. % B₄C reinforced hybrid composites. To confirm the nature of fracture in the hybrid structure another fracture surface SEM image was taken at 3000 X magnification (Fig. 3-d). The granular and shiny appearance in this image indicates that the fracture is brittle in nature.

Energy Dispersive Spectroscopy (EDS) analysis of Al matrix near the nanoparticles is shown in Fig. 4. The aluminum peak, silicon peak, boron peak and carbon peak in the EDS confirms the incorporation of SiC and B₄C nanoparticles in Al melt. Also, the oxygen content is very low which indicates that the nanocomposites were well protected during fabrication.
4.2 Hardness

The cast specimens were subjected to Brinell’s hardness test to find the hardness values and the results are shown in the Fig. 5. A load of 500 kg was applied using 10 mm steel ball over the specimen for a dwell period of 20 seconds. Test results reveal that the Brinell hardness values of the hybrid composites are higher as compared to the unreinforced counterpart, and also the values increases as the hybrid reinforcement ratio increases. The added SiC and B₄C nanoparticles enhance hardness, as these particles are harder than Al alloy, which render their inherent property of hardness to soft matrix. It is also observed that the presence of stiffer and stronger hybrid reinforcements in the Al matrix, effectively impede the movement of dislocations which ultimately enhances the hardness and strength of the hybrid composites.

To study the uniform distribution of hybrid reinforcements, the hardness value is measured on the various portions of the cast bar [12]. Five specimens were cut from each casting at a distance of 20 mm, 40 mm, 60 mm, 80 mm and 100 mm from the base of the cast bar. As evident from the Fig. 5 the hardness values are nearly uniform throughout the sections for the hybrid ratios up to 1.0 vol. % SiC and 0.5 vol. % B₄C, which show that the nano reinforcements are uniformly distributed. But for the hybrid combination of 1.5 vol. % SiC and 0.5 vol. % B₄C too much of fluctuation is observed in the hardness values. This shows that in some places hardness is very high and in some places it is very low. Hence it can be concluded that at this combination, the hybrid reinforcements are not uniformly distributed.

The non-uniform distribution of hardness should be attributed to the more agglomeration of nano reinforcements at this combination at some regions and increased porosity content as the particle volume
percentage increases. Better understanding and proper controlling of process parameters are required to reduce the agglomeration tendency of nanoparticles at higher volume percentages.

The implosion of cavitation bubbles in the melt generates enormous amount of energy and this energy was used to separate the clustered nanoparticles into single nanoparticle. The conventional stir casting route could also be used to disperse the nanoparticles, but, once stirring stops the nanoparticles either will float on the top of the melt or sediment at the bottom of the crucible.

![Fig. 5 BHN distribution for the un-reinforced and hybrid composites](image)

**4.3 Tensile Behavior**

To study the tensile behavior of the nanocomposites, specimens were prepared and tested as per ASTM E8 standard. The stress – strain diagram of the as-cast alloy and as-cast hybrid composites is shown in the Fig. 6. From the graph it is understood that the tensile strength values of the hybrid composites are higher than that of the unreinforced counterpart and the combination 1.0 % SiC and 0.5 % B$_4$C give superior tensile strength value. The improvement in tensile strength was attributed to presence and uniform distribution nano reinforcements. The incorporated reinforcements act as barrier to dislocation movements. This dispersive strengthening mechanism normally referred to orowan strengthening [13-15].

Another strengthening mechanism that shares the maximum load bearing effort in the hybrid composite is the strengthening due to high dislocation density caused as a result of co-efficient of thermal expansion (CTEs) difference between the matrix and reinforcements [13-15]. The CTE value of aluminum is 25×10^{-6}/°C, for SiC the CTE value is 4×10^{-6}/°C and for B$_4$C the CTE value is 5×10^{-6}/°C. The large difference in CTE mismatch between the aluminum and hybrid reinforcements could actually induces the enormous amount of dislocations in the hybrid composites. These induced dislocations act as a barrier for the dislocations movement. Hence the strength of the hybrid composites are increased significantly even for the addition of very low volume percentage of the nano reinforcements.

The tensile strength of 1.5 % SiC and 0.5 % B$_4$C reinforced nanocomposite is actually lesser than that of the nanocomposite reinforced with 1.0 % SiC and 0.5 % B$_4$C. The possible reason could be; increased agglomeration of the nanoparticles and increased porosity content as the particle volume percentage increases. Agglomerated nanoparticles leads to the microclusters and the loosely packed particles in the clusters makes the material as weaker structure and hence reduction in tensile strength. The presence of porosity in the solidified microstructure reduces the mechanical properties of cast hybrid composites as the plastic deformation is initiated from the voids formed.

Compared to the unreinforced counterpart, the elongation of the nanocomposites, which is the measure of ductility, is marginally lower, and its value decreases as the particle volume percentage increases (Fig. 7). It could
be due to the presence of more secondary particles, and the early void formation at low strains during tensile elongation. Moreover, it is clear from the results that the hybrid composites show better tensile properties than the conventional composites.

![Graph of Stress-strain relationship of cast alloy and cast hybrid composites](image)

**Fig. 6** Stress-strain relationship of cast alloy and cast hybrid composites

### 4.4 Impact Energy

To study the impact behavior of the hybrid composites, Charpy impact tests were performed on YAMA make impact testing machine. The machine can provide a range of impact energies from 1 J to 360 J. Charpy Impact specimens were prepared as per ASTM E23 standard. Test specimens measured 50 x 10 x 10 mm. The notch had a depth of 2 mm with a notch tip radius of 0.02 mm.

The impact test results of the hybrid nanocomposites and the un-reinforced alloy are shown in the Fig. 7. It is clearly indicated in the figure that, as the volume fraction of nano hybrid reinforcement phase in the metal matrix increases, the impact energy of the composite, when compared to the unreinforced counterpart, actually decreases. Higher the percentage of particulates in the matrix lesser is the impact energy. This is actually due to the failure of reinforcements both by cracking and decohesion. This is also attributed to the presence and role of intrinsic defects in the composite microstructure when compared to the unreinforced aluminum alloy. Granular and shiny appearance in SEM images and shrinkage cavities (Fig. 3 (c) and Fig. 3 (d)) is the indication of totally brittle fracture.

![Graph of Hybrid reinforcement’s vol. % vs. % Elongation and Impact Energy](image)

**Fig. 7** Hybrid reinforcement’s vol. % vs. % Elongation and Impact Energy
Even though the impact strength of the hybrid nanocomposite is lower when compared to the un-reinforced counterpart, the reduction amount is very marginal. Also, the composites reinforced with the nanoparticles show the better impact property than the conventional composites.

5. Conclusions

In summary, aluminum alloy 6061 reinforced by 0.5, 1.0 and 1.5 vol. % SiC nanoparticles with concurrent reinforcement of 0.5 vol. % B₄C nanoparticles were fabricated by high intensity ultrasonic cavitation process. From the experimental results the following conclusions were obtained.

- From the SEM analysis, it was found that the nanoparticles were uniformly distributed in the metal matrix for the hybrid ratios up to 1.0 vol. % SiC and 0.5 vol. % B₄C. But for the hybrid ratio of 1.5 vol. % SiC and 0.5 vol. % B₄C the agglomeration tendency increases. Fracture surfaces show the evidence of intrinsic defects and shrinkage cavities. EDS study validates the presence of SiC and B₄C in the solidified microstructure.

- Hardness of the composites increase as the hybrid ratio increases. Results also confirmed that the hardness values are uniform throughout the cast bar for the hybrid ratios up to 1.0 vol. % SiC and 0.5 vol. % B₄C.

- The addition of small amount of nanoparticles results in the significant improvements in tensile strength. The superior tensile strength was obtained at the hybrid ratio of 1.0 vol. % of SiC and 0.5 vol. % B₄C and after that tensile strength reduced. Ductility of the composites reduces marginally as the hybrid ratio increases.

- The impact energy of the hybrid composite was found to be marginally lower than the unreinforced counterpart.

- This work is a preliminary study; detailed study is required to evaluate the contribution of SiC and B₄C nanoparticles on the mechanical properties of the hybrid composites.

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