EFFECT OF PRECIPITATION HARDENING ON STIR CAST
Al6061-B₄C REINFORCED COMPOSITE

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ABSTRACT

The focus of the present investigation is to enhance mechanical properties of two-stage stir cast Al6061-B₄C (2, 4 and 6 wt.%)) reinforced metal matrix composites by artificial aging treatment. The Optical and Scanning Electron Microscope show homogeneous distribution of reinforcements in the aluminum matrix. The composites were solutionized at 558 °C for 2 h and followed by water quenching. The samples were subjected to aging treatment in the range of 100, 150 and 200 °C for different time intervals. Lower aging temperature and higher aging time show substantial enhancement in hardness and tensile strength. Around 170% improvement in hardness and 100% improvement in tensile strength were observed when aged at 100 °C with an increased weight percentage of reinforced particulates as compared to the unreinforced Al6061 alloy.

KEYWORDS: Aluminum Metal Matrix Composites (AMMC’s), Boron carbide (B₄C), Stir Casting & Aging Treatment

INTRODUCTION

Aluminum is one of the most commonly used matrix material other than magnesium, titanium, iron, nickel, cobalt, silicon and silver, because of its density, easy fabricability, and good mechanical properties. The heat treatable aluminum metal matrix composites (AMMCs) reinforced with hard ceramic particles such as SiC, B₄C, Al₂O₃ and TiC are the most commonly used materials in aerospace, automobile and marine industries [1]-[3]. Stir casting, powder metallurgy, plasma spraying, spray atomization and co-deposition are the most commonly used manufacturing methods of AMMC’s. Stir casting is one of the most commonly used manufacturing methods because of its ease of production and cost [4]. Improvement in properties through ageing treatment is associated with the formation of GP-zones, β”, β’ and β phases. According to the literature related to the aging sequence, variation in hardness versus aging temperature and time can be correlated to the phase transformation kinetics during aging treatment [5]-[10]. The escalation in hardness during aging treatment is mainly due to the hindrance of secondary solute rich phases. When the weight ratio of Mg to Si in Al 6061 alloy is greater than 1.73, there are more chances of precipitation of β phase (Mg₂Si), θ phase (CuAl₂) and S phase (Al₂CuMg) [12]-[13]. The precipitation of coherent needle-shaped β particles results in an increase in hardness [14]. Lot of research work has been carried out by using SiC and Al₂O₃ as reinforcing materials. Because of the higher cost and wettability of reinforcement particulates with matrix alloy, little work has been observed on AMMC’s reinforced with B₄C reinforcement particles [3]. The objective of the present research is to investigate
the effect of B₄C reinforcement particles and artificial aging treatment on stir cast AMMC’s (Al6061-B₄C).

EXPERIMENTAL PROCEDURE

The matrix alloy preferred in the present research work is Al6061alloy (0.55% Si, 0.85% Mg, 0.17% Mn 0.50% Fe, 0.20% Cr, and 0.25% Cu). The B₄C reinforcement particles used for the preparation of composite is brought from Boron Carbide limited, Mumbai. A shape of the reinforcement materials is having irregular and Scanning Electron Microscope (SEM) with Energy Dispersive X-ray (EDAX) plot of the same shown in Figure 1. Al6061- B₄C AMMC’s are manufactured by stir casting technique with 2-6% wt. of reinforcement addition. The Al6061 billets are heated to about 750°C in a furnace to convert solid into the liquid phase. A small amount of scum powder is introduced to remove the slag or flux. The entire melt is then degassed by adding Hexa Chloroethane (C₂Cl₆, 0.3 % wt.) tablet [15]. The B₄C particles are preheated to 250°C for 2h in order to remove the volatile substances [16]. Pre-heating of boron carbide particles results in the removal of surface impurities and improvement in wettability of the reinforcements. The melt is allowed to form the semi-solid state at 600°C. B₄C particles in varying proportions (2, 4 and 6% wt.) is poured into the vortex formed during stirring. A mild steel stirrer with axis in a vertical position is utilized. Stirring speed is maintained in the range of 150-200 rpm for 10 minutes, which results in better dispersion of the reinforcements in the molten alloy [17]. A semi-solid slurry is heated once again to a temperature of 750°C±10°C and once again rousing is continued for 15 min. at 450 rpm. Molten melt is poured into the cast iron molds, which are preheated to 500°C. Al6061-B₄C composites are fabricated by varying weight percentage of B₄C reinforcements and melt is allowed to solidify in the air for 2h.

Brinell hardness testing machine is used to measure the hardness (load:- 250 kgf, steel ball indenter:- Ø 5mm, ASTM-E10). Tensile properties are determined to study the behavior of the composite under tensile loads. ASTM-E8M standards are used to prepare the tensile specimen. The hardness and tensile specimens are subjected to aging treatment by soaking at 558°C for 2h, followed by water quenching maintained at room temperature. The specimens after quenching are artificially aged at 100, 150 and 200°C for different intervals of time till the peak hardness condition is reached. The secondary precipitated strengthening Mg₂Si phase completely dissolves at 558°C during solutionizing and precipitates during aging treatment results in particle strengthening by retaining coherency with the matrix. It is reported that solution heat-treated AMMC’s at 558°C reveal substantial improvement in the property of the composites [18]. Precipitation of secondary phases are analysed by using transmission electron microscopy (TEM) - JEM 2200 JEOL-TEM with the bright-dark field image technique.

![Figure 1: (a) SEM Micrographs of Boron Carbide Particles and (b) Energy Dispersive X-ray (EDAX) Plots of B₄C Particles](image-url)
RESULTS AND DISCUSSIONS

Optical Micrography

Manufacturing of AMMC’s is one of the most important aspects for homogeneous and uniform dispersal of the reinforcement. Presence of reinforcement particles in the matrix alloy could give perception into the composite quality. In order to ensure the uniform dispersal of B₄C reinforcement particles, the cast samples are examined by optical inverted metallurgical microscope. The optical microscopy explains the microstructures of Al6061 composites with 0, 2, 4 and 6% wt. B₄C. Figure 2, reveals the uniform dispersal of the B₄C particle in Al6061 alloy matrix. Also, the microstructure does not reveal the existence of the blow holes or air pockets.

Figure 3 shows the SEM micrographs of polished Al6061-6% wt. B₄C composite and corresponding Energy Dispersive X-ray (EDAX) spectra of B₄C particles. Surrounding these particles, some stress zone exist resulting prominent contrast compare to particles and matrix. EDAX was taken from these particles as well from surrounding regions, indicated by numbers. Figure 3(c), shows, EDAX of the reinforcement particle present in the matrix with boron as prominent peak and carbon as the shallow peak. This is the clear evidence for the presence of beneficial carbon in the reinforcement. EDAX matrix location shows composition Al6061 alloy as shown in Figure 3(d).

Figure 2: Optical Microphotograph of Al6061 Alloy and Al6061-B₄C Composites

Figure 3: SEM Micrographs of Polished Al6061-6% B₄C Composite and Corresponding Energy Dispersive X-ray (EDAX) Spectra of B₄C
Density Measurement

The existence of porosity, its dispersal in the alloy, plays a significant role in governing the enhancement of AMMC’s properties. The level of porosity should be minimum to achieve better performance in overhaul applications. Presence of porosity in AMMC’s is mainly due to the entrapment of air bubbles in the molten slurry during stirring or envelop of air to the B₄C reinforcing particles during manufacturing of composites.

Figure 4, displays that the values of theoretical and experimental density are in line with each other. Percentage of porosity level ratifies the suitability of the liquid metallurgy technique for the preparation of AMMC’s. For different wt.% of B₄C reinforcements, the values of experimental and theoretical density are very close to each other. The density of the AMMC’s is found to be lower than that of the base Al6061 alloy. Addition of less dense reinforcement particle (B₄C-2.52 g/cc) to a high-density matrix (Al6061 alloy-2.7 g/cc) decreases overall density of the composites.

Percentage porosity increases the macroscopic defects in the material, which contributes to the precipitation process. Increase in wt.% of B₄C in Al6061 composites increases the percentage porosity level, which also improves the nucleation sites for secondary hardening. According to Kok M et al, [19] and Prabhu et al,[20] stated that up to 4% of porosity levels are acceptable levels in cast composites. The adoption of two stage stirring technique is responsible for the low porosity level and minimal casting defects in the composites produced.

Figure 4: Dependence of Theoretical, Experimental Densities and Porosity with the Weight Percentage of B₄C Reinforcement in Composites

Aging Curve and Hardness

The average hardness values of cast and peak aged samples aged at 100, 150 and 200°C are shown in Figure 5. Compared to the matrix aluminum alloy, boron carbide reinforced composites shows an increase in hardness values. The hardness value enhances with an increase in wt. percentage (2, 4 & 6% wt.) of boron carbide particles. Increased content of boron carbide particles in the alloy matrix results in more sites for nucleation for precipitation of secondary intermetallic particles due to increase in density of dislocation during solidification. To accommodate the lesser volume expansion of the particulates during solidification, matrix deforms plastically which in turn alters the matrix microstructure. Augmentation in the density of dislocation leads to more resistance to plastic deformation by additional improvement in property of AMMC’s [21].
Regardless of aging temperature, matrix aluminum alloy and $\text{B}_4\text{C}$ reinforced AMMC’s are very sensitive to age hardening. The main process of aging in matrix alloy always involves the diffusion controlled mechanism of solute atoms in the aluminum matrix. Change in diffusivity of alloying elements takes place for any modification in the matrix due to the presence of reinforcements and also changes the aging response of the matrix. During the precipitation hardening process, the existence of $\text{B}_4\text{C}$ reinforcements in Al6061 alloy accelerates the kinetics of aging treatment. After solutionizing treatment, the presence of chemically stable and low co-efficient of thermal expansion (CTEs) $\text{B}_4\text{C}$ particles induces stresses in the matrix to cause substantial plastic flow, which in turn leads to the higher density of dislocation at the interface of matrix and reinforcements. High dislocation density and induced stresses at the surrounding area serves for both heterogeneous nucleation and short circuit path for pipe diffusion which can accelerate the aging process [23].

Aging at a lower temperature (100°C) shows substantial enhancement in hardness in both AMMC’s and matrix alloy as compared to the higher temperature of aging (200°C). More number of transitional zones during 100°C of aging treatment results in a larger quantity of finer solute-rich secondary inter-metallics & lesser interparticle spaces. The time required to reach peak hardness is less due to the higher rate of diffusion during higher temperature of aging compared to lower aging temperature [9]. It can be established from the experimental results that aging treatment has a profound effect on the improvement in the hardness of both AMMC’s and matrix alloy.

The Al6061-6% wt. $\text{B}_4\text{C}$ composite shows maximum hardness at lower aging temperature. Figure 6, shows TEM images of Al6061-6% wt. $\text{B}_4\text{C}$ AMMC’s peak aged at 100°C under higher magnification. Presence of $\beta''$ and $\beta'$ in matrix alloy give rise to satellite spots and splashes [14]. The amount of precipitated phases shows the presence of Mg and Si, suitably confirmed by the precipitation of semi-coherent Mg$_2$Si ($\beta''$) in peak aged condition as shown in Figure 6 of the EDAX image. The presence of a high percentage of Al is due to matrix effect as the size of the particle is very small. According to Gracio J. J et al, [14], the presence of a large number of tiny dots in the bright field images represents the $\beta'$ needles along [001] Al.
Tensile Strength

A tensile test is carried out for both cast and peak aged samples of matrix alloy and AMMC’s. Figure 7 shows a marginal increase in the ultimate tensile strength (UTS) with the addition of reinforcements when compared to unreinforced alloy in as-cast condition. Improvement in ultimate tensile strength in AMMC’s was minimum with the accumulation of reinforcement particles as compared to matrix alloy in as-cast condition. Mechanical properties are controlled by the presence of reinforcement particulates due to the strong interface between matrix and reinforcement, which results in increased UTS and elastic modulus due to distribution and transfer of load from the matrix alloy to the B$_4$C reinforcement particulates. In addition to the above characteristics, AMMC’s comprises of both metallic property and ceramic property which in turn results in an increase in hardness values due to microstructural changes during solidification. Increase in the weight percentage of reinforcement particulates from 2-6% in the AMMC’s shows superior ultimate tensile strength during lower temperature of aging as shown in Figure 7. The combined effect of B$_4$C particulates having lower thermal coefficient of expansion as compared to matrix alloy and heterogeneous nucleation of secondary solute-rich precipitated phases result in substantial enhancement of UTS.
CONCLUSIONS

The following are the conclusions drawn from the research study:

- Al6061-B\textsubscript{4}C composites with 2, 4 and 6\% wt. of reinforcements (B\textsubscript{4}C) are successfully fabricated by two-step stir casting technique.
- Homogeneous distribution of B\textsubscript{4}C reinforcement particles was observed in the fabricated composites.
- The density of composites is lower than that of the base matrix, further, the density decreases with increase in reinforcement content in the composites.
- In as-cast condition, 30-50\% improvement in hardness is observed in as-cast composites compared to the matrix alloy.
- Both matrix alloy and Al6061-B\textsubscript{4}C AMMC\’s shows positive response during aging treatment with substantial improvement in property such as hardness and UTS.
- Hardness improved by 90-120\% aged at 200°C and 140-170\% (2, 4 & 6 wt.\% B\textsubscript{4}C) aged at 100°C is observed for the AMMC\’s as compared to matrix alloy
- TEM observations confirm the presence of fine precipitates during the aging process.
- At least 20\% increase in UTS is observed in Al6061-B\textsubscript{4}C AMMC\’s compared to matrix alloy in as-cast condition.
- Maximum 80-100\% increase in UTS of AMMC\’s over untreated matrix alloy during aging treatment.

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