Tensile fractography of artificially aged Al6061-B₄C composites

Sathyashankara Sharma¹, Achutha kini¹, Gowri Shankar*¹, Rakesh T C¹, Raja H and Krishna Chaitanya¹, Manjunath Shettar¹

¹Department of Mechanical Engineering, Manipal Institute of Technology,
Manipal Academy of Higher Education, Manipal, Karnataka, INDIA-576104

Phone: +91-9844898535; Fax: +91-820-2571071

*Email: gowri.shankarmc@manipal.edu

ABSTRACT

Presence of various amount of B₄C reinforcement (2, 4 & 6% wt.) in Al6061 alloy on the hardness and tensile behaviour is studied in the present work. The influence of artificial aging due to presence of reinforcement on Al6061 alloy also evaluated. Brinell hardness and failure behaviour during tensile loading which impact the growth of failure physiognomies have been confirmed. The conventional age hardening treatment at three aging temperatures (100, 150 and 200°C) is performed on the composites, peak hardness and ultimate tensile strength variations at three aging temperatures is critically analysed. Lower temperature aging shows enhancement of hardness by 170% and ultimate tensile strength by 90%. The best results obtained during peak aging at 100°C is subjected to transmission electron microscope (TEM) analysis to look into the type of intermetallic responsible to strain the matrix alloy. TEM study recognises the Mg₂Si phase formation during peak aging. Precipitation of this intermetallic phase and presence of harder reinforcements leads to the enhancement of hardness and nucleation of void growth failure during artificial aging treatment.

Keywords: Matrix alloy; Aluminium Metal Matrix Composites; Boron carbide; aging; fractography; microstructure.

INTRODUCTION

Heat treatable aluminium magnesium-silicon alloy (Al6061) shows good formability and resistance to corrosion during precipitation hardening [1]. Aluminium Metal Matrix Composites (AMMC's) with Silicon carbide (SiC), Boron carbide (B₄C), Aluminium oxide (Al₂O₃) and Titanium carbide (TiC) has reinforcements, are the utmost frequently used materials in marine and automobile industries due to superior strength and least density. The AMMC's reinforced with B₄C particulates are harder, tougher with improved fatigue strength reveal substantial enhancement in properties compared to other composites [2-5]. Existence of reinforcements in AMMC's accelerates the aging sequence due to increase in dislocation densities to improve hardness due to the fine precipitation of intermetallics[6-9]. Hardness improvement with respect to aging temperature and time can be correlated to the phase changes through age hardening. Peak aged condition leads to increase in composite strength due to the vacancy assisted diffusion mechanism [10-13]. Fahrettin et al. [14], investigated the effect of artificial aging to get maximum hardness and yield strength for Al6061 alloy. The samples are solution treated in the furnace at 550°C for 2h, followed by quenching in water and keeping the specimen in the same water for 4h. The specimens are then subjected to artificial aging, at 200°C for 200-600 mins. The improvement in peak hardness, advances the ultimate tensile strength, that leads to the decrease in the strain hardening capabilities. This is

attributed to the formation of coherent precipitates from Mg and Si co-clusters. These meta-stable precipitates, with composition Mg_5Si_6 , are dominant in the microstructure of the peak aged material. Cun-Zhu et al. [15], work focused on production of B_4C reinforced AMMC's. The results revealed that there is an improvement in mechanical properties of composites. It is primarily due to uniform distribution of reinforcements and formation of interface between matrix and reinforcements. B_4C reinforced composites show more strengthening effects as compared to SiC reinforced composites. Gopal et al. [16], studied the effect of B_4C reinforcement on the tensile property of AMMC's. The hardness and tensile strength are found to increase with the increase in the wt.% of reinforcement. Presence of reinforcements in the matrix results an increase in the nucleation sites and decrease in the grain size. The presence of such hard surface area particles offers more resistance to plastic deformation which leads to increase in the hardness of composites. Increase in the tensile strength is due to the increase in hardness of the composite. The distortion of matrix lattice planes during Guinier Preston zones formation depends upon the aging condition which inturn impedes the movement of dislocation as long as the lattice coherency exists between the matrix and precipitates. B_4C reinforced composites show better interfacial bonding compared to SiC , Al_2O_3 etc., reinforced AMMC's [17-19]. Present research work focuses on the influence of B_4C reinforcements and artificial aging treatments on hardness and fractography behaviour of Al6061- B_4C AMMC's.

METHODS AND MATERIALS

The matrix alloy preferred in the present research work is Al6061 (0.50 Si, 0.95 Mg, 0.17 Mn, 0.50 Fe, 0.20 Cr, and 0.25 Cu by wt.%) as extensively usable 6XXX series aluminium alloys. The B_4C reinforcement particles used for the preparation of composite is brought from Boron Carbide limited, Mumbai. Shape and size of the reinforcement seems to be uniform and Scanning Electron Microscope (SEM) with Energy Dispersive X-ray (EDAX) plot of the same shown in Figure 1. Al6061- B_4C 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 furnace to convert solid into liquid phase. Small amount of scum powder is introduced to remove the slag or flux. The entire melt is then degassed by adding Hexa Chloroethane (C_2Cl_6 , 0.3 % wt.) tablet [17]. The B_4C particles are preheated to 250°C for 2h in order to remove the volatile substances [18]. Pre-heating of boron carbide particles results in removal of surface impurities and improvement in wettability of the reinforcements. The melt is allowed to form semi-solid state at 600°C . B_4C particles in varying proportions (2, 4 and 6% wt.) is poured into the vortex formed during stirring. A mild steel stirrer with axis in vertical position is utilised for dispersion of reinforcements in the melt. Stirring speed is maintained in the range of 150-200 rpm for 10min, which results in better dispersion of the reinforcements in the molten alloy [19]. Semi-solid slurry is heated once again to a temperature of $750\pm 10^\circ\text{C}$ and once again stirrer is actuated for 15 min. at 450 rpm. Molten melt is poured in the cast iron moulds, which are preheated to 500°C . Al6061- B_4C composites, are fabricated by varying weight percentage of B_4C reinforcements and melt is allowed to solidify in air and cooled to room temperature.

Brinell hardness testing machine is used to measure the hardness (250 kgf, load, 5mm diameter ball indenter and dwell time of 15 seconds) as per ASTM-E10 standards [20]. Tensile properties are determined to study the behaviour of the composite under tensile loads. ASTM-E8M standards [21] are used to prepare tensile specimen shown in Figure 2. The fractured specimen after tensile test are shown in Figure 3. 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 existence of Mg_2Si phase present in the stir cast completely dissolves at 558°C during solutionizing and precipitates during aging treatment as finer particles 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 property of the composites [9].

Figure 1. a) SEM micrograph with of B₄C particulates b) XRD plots of B₄C.

Figure 2. Tensile specimen as per ASTM-E8M.

Figure 3. Fracture tensile specimen a) Al6061 alloy aged at 100°C b) Al6061-B₄C (6 % wt.) aged at 100°C.

RESULTS AND DISCUSSION

Measurement of hardness

As-cast and peak aging conditions average hardness values aged at 100, 150 and 200°C are shown in Figure 4. Compared to the matrix aluminium alloy, B₄C reinforced composites show increase in hardness values. The hardness value enhances with increase in wt. percentage (2, 4 & 6% wt.) of boron carbide particles. Increased content of B₄C reinforcements in the matrix alloy results in more heterogeneous 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 density of dislocation leads to more resistance to plastic deformation by additional improvement in property of AMMC's [17].

Regardless of aging temperature, matrix aluminium alloy and AMMC's are very sensitive to age hardening. AMMC's display augmented rate of precipitation kinetics as related to Al6061 matrix alloy. Composites show accelerated aging kinetics with increase in wt. % of B₄C reinforcement particles and existence of higher dislocation density at the interface of the matrix alloy & B₄C reinforcement particulates. This in turn results in high diffusivity path due to the heterogeneous nucleation for the diffusion of secondary solute rich phases [18]. AMMC's show substantial hardness escalation as compared to matrix alloy, in both age hardened and as cast conditions. Positive effect on the values of hardness is perceived with escalation in weight percentage of B₄C reinforcements in AMMC's. Compared to higher aging temperature (200°C), lower aging temperature (100°C), shows substantial enhancement in hardness in both AMMC's and matrix alloy. More number of transitional zones during aging at 100°C results in larger quantity of finer solute rich secondary intermetallics with lesser interparticle spaces. The time required to reach peak hardness is less due to faster 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 improvement in hardness of AMMC's and matrix alloy.

Figure 4. Hardness of Al6061-B₄C (0, 2, 4 & 6% wt.) as-cast and peak aged condition.

Figure 5. TEM photographs along with bright field image of Al6061- 6% wt. B₄C AMMC's peak aged at 100°C and corresponding EDAX.

Ultimate tensile strength

As-cast and peak aged samples of matrix alloy and AMMC's are subjected to tensile test. Figure 6 shows the average ultimate tensile strength (UTS) readings in as cast and peak aged conditions. Considerable improvement in hardness and UTS is observed in AMMC's compared to base alloy and a maximum peak values are recorded for the composites with maximum weight percentage of reinforcements. Improvement in hardness and tensile strength are controlled by the presence of reinforcement particulates due to the strong interface between matrix and reinforcements, which results in increased UTS and elastic modulus due to distribution and load transfer from the matrix alloy to the B₄C reinforcements [23,24].

In addition to distribution and transfer of load, AMMC's comprises both metallic and ceramic properties which in turn result increase in hardness values due to microstructural changes during solidification. Further 55-60% improvement in the tensile strength values are recorded for AMMC's during aging treatment over matrix alloy. Increase in the weight percentage of reinforcement particulates (2 to 6%) in the AMMC's shows superior ultimate tensile strength during lower temperature of aging as shown in Figure 6. The combined effect of B₄C particulates having lower thermal co-efficient of expansion as compared to matrix alloy and heterogeneous nucleation of secondary solute rich precipitated phases results in substantial enhancement of UTS.

Figure 6. Ultimate tensile strength of alloy and its AMMC's in as-cast and peak aged conditions.

Fractography of matrix alloy and Al6061-B₄C AMMC's

Figure 7, shows predominantly dimple rupture fracture mode of Al6061 matrix alloy. Abundant cuplike miseries or dimples are detected from fractography. Combination of micro-voids that precipitate at localized strain region (secondary solute rich particles, dislocation of grain boundaries, vacancies etc.) results in formation of dimples. Figure 8 shows tensile fractography of peak aged samples with enormous number of dimples. More numbers of dimples with finer size indicating the development of micro-voids at several precipitated particles. Uniformly dispersed finer dimples are responsible for higher strength.

Figure 7. SEM micrographs of fracture surface of as cast Al6061 alloy.

Figure 9 reveals the fracture surface with SEM microstructure of Al6061-6% B₄C AMMC's in as-cast condition. Considerable decrease in dimple density is observed in comparison with Al6061alloy. In a localized region fracture may be due to tear or shear, resulting in the formation of

elongated dimples. Further, it is noticed that some of the dimples are shallow and these may be due to coalescence of micro voids by shear as shown in Figure 9(b). Whereas, in the case of peak aged sample at 100°C (Figure 10, a-f) the fracture mode is dimple rupture. Dimples are formed when numerous nucleation sites are activated resulting in the formation of micro-voids and thereafter coalescence of these micro-voids. Nucleation sites may be inclusions, second phase particles, grain boundary dislocation pile-ups etc. Fracture mode is significantly influenced by the distribution of these particles/defects. Nonuniform distribution of such particles/defects with various sizes results in the formation of dimples of different sizes. In few places, quasi-cleavage fracture is also noticed. Characteristics of both cleavage and plastic deformation (Figure 10f) leads to the formation of quasi-cleavage fracture.

Figure 8. Al6061 fracture surface peak aged at 100°C.

Figure 9. SEM micrographs of fracture surface of as-cast a) Al6061-6% wt. B₄C composite b) at higher magnification.

Figure 10. SEM micrographs of fracture surface of (a) Al6061-6% B₄C peak aged at 100°C, (b-f) at different locations

CONCLUSIONS

From the present work, it is observed that, 30-50% improvement in hardness is observed in as-cast composites compared to the matrix alloy. Both matrix alloy and Al6061-B₄C AMMC's show positive response during aging treatment with substantial improvement in properties such as hardness and UTS with the increase in reinforcement quantity in the matrix. Higher hardness values are noticed under peak aging condition at lower temperature of aging for both matrix alloy and AMMC's. Increase in hardness of 100-120% aged at 200°C and 150-170% aged at 100°C is measured in significant intervals for the AMMC's in comparison with matrix alloy. A minimum increase of 20% in UTS is witnessed in Al6061-B₄C AMMC's composite compared to matrix alloy in as cast condition. Maximum 90% increase in UTS of AMMC's over untreated matrix alloy is observed during aging treatment. TEM study shows the presence of rod shaped Mg₂Si phase in peak aged 6 wt % B₄C reinforced AMMC's. Peak aged tensile fracture surface of matrix alloy and its AMMC's fails by mixed mode of fracture. Fracture mode of Al6061 alloy is predominantly dimple rupture, whereas B₄C reinforced composite fracture surface shows quasi-cleavage fracture and plastic deformation.

REFERENCES

- [1] Hussain F, Abdullah S, Nuawi MZ. Effect of temperature on fatigue life behaviour of aluminium alloy AA6061 using analytical approach. *Journal of Mechanical Engineering and Sciences*. 2016 ;10(3) :2324-2235.
- [2] Nieh TG, Karlak RF. Aging characteristics of B₄C-reinforced 6061-Aluminium matrix. *Scripta. Matereialia*.1984;18:25-28.
- [3] Salleh MNM, Ishak M, Romlay FRM, Aiman MH, A study on bead-on-plate welding of AA7075 using low power fiber laser. *Journal of Mechanical Engineering and Sciences* 2016 ;10(2) : 2065-2075.
- [4] Muhamad H, Hussin, Nur A, Che L. Effects of temperature on the surface and subsurface of Al-Mg-Si welded joints. *Journal of Mechanical Engineering and Sciences*. 2017 ;11(2) : 2743-2754.
- [5] Ozturk F, Sisman A, Toros S, Kilic S, Picu R. Influence of aging treatment on mechanical properties of 6061 aluminum alloy. *Material and Design*. 2012; 31:972–975.
- [6] Miao WF, Laughlin DE. Precipitation hardening in aluminum alloy 6022. *Scripta-Matereialia*. 1999; 40:873–878.
- [7] Rajasekaran SN, Udayashankarand K, Jagannath N. T4 and T6 Treatment of 6061 Al-15vol.% SiCp Composite. *International Scholarly Research Network*. 2012; 1:1-5.
- [8] Rajan TV, Sharma CP, Sharma A. Heat treatment principles and techniques. PHI, India, 2012: 118-119.
- [9] Siddiqui RA, Hussein A, Abdullah, Khamis RAB. Influence of aging parameters on the mechanical properties of 6063 aluminium alloy. *Journal of Material Processing Technology*. 2000; 102:234-240.
- [10] Siddiqui RA, Abdul W, Pervez T. Effect of aging time and aging temperature on fatigue and fracture behavior of 6063 aluminum alloy under seawater influence. *Material and Design*. 2008; 29:70–79.
- [11] Avner SH. Introduction to physical metallurgy, Second edition, Mc. Graw Hill, ISBN07-85078-1.1974;1:190-191.
- [12] Wang HQ, Sun WL, Xing YQ. Microstructure analysis on 6061 aluminum alloy after casting and diffuses annealing process, *Physics Procedia-Elsevier*. 2013; 2:68-75.
- [13] RamakoteswaraRao V, Ramanaiah N, Srinivasa Rao M, Sarcara MMM, Kartheek G.Optimisation of process parameters for minimum volumetric wear rate on AA7075-TiC

- metal matrix composite. International Journal of Automotive and Mechanical Engineering. 2016 ;13(3):3669-3680
- [14] Fahrettin O, Esener E, Toros S, Picu CR. Effects of aging parameters on formability of 6061-O alloy. Materials and Design, 2010; 31(10):4847-4852.
- [15] Cun ZN, Gu JJ, Liu JL, Zhang D. Production of boron carbide reinforced 2024 aluminum matrix composites by mechanical alloying. Materials Transactions. 2007;48(5):990-995.
- [16] Gopal KUB, Sreenivas RKV, Vasudeva B. Effect of percentage reinforcement of B₄C on the tensile property of aluminium matrix composites. International Journal of Mechanical Engineering and Robotic Research. 2012;1(3):290-295.
- [17] Kulkarni K, Krishnamurthy SP, Deshmukh, MRS. Effect of particle size distribution on strength of precipitation-hardened alloys. Journal of Material Research. 2014; 19:2765-2773.
- [18] Baradeswaran. and Elaya P. Influence of B₄C on the tribological and mechanical properties of Al 7075–B₄C composites. Composites, Part–B. 2013;54:146–152.
- [19] Shorowordi KM, Laoui T, Haseeb JP, Celis F. Microstructure and interface characteristics of B₄C, SiC and Al₂O₃ reinforced Al matrix composites: a comparative study. Journal of Material Processing Technology. 2003; 142:738–743.
- [20] ASTM E8/E8M (2013). Standard Test methods for Tension Testing of Metallic Materials. ASTM International, West Conshohocken, PA.
- [21] ASTM E10 (2017), Standard Test methods for Brinell Hardness of Metallic Materials of Metallic Materials. ASTM International, West Conshohocken, PA.
- [22] Gracio JJ, Barlat F, Rauch EF, Jones PT, Neto VF, Lopes AB. Artificial aging and shear deformation behaviour of 6022 aluminium alloy. International Journal of Plasticity. 2004;20(3): 427-445.
- [23] Ibrahim MF, Ammar HR, Samuel AM, Soliman MS, Almajid A, Samuel FH. Mechanical properties and fracture of Al–15 vol.% B₄C based metal matrix composites. International Journal of Cast Metals Research. 2014;27(1):7-14.
- [24] Auradi V, Rajesh GL and Kori SA. Processing of B₄C particulate reinforced 6061aluminum matrix composites by melt stirring involving two-step addition. Procedia Materials Science 2014; 6:1068-1076.