Elevated Temperature Properties of Extruded Aluminium Matrix Composites Reinforced with Nickel Coated Silicon Carbide Particles

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ABSTRACT

The superior mechanical properties of Aluminium Matrix Composites (AMC) together with the weight saving makes them attractive for a variety of engineering applications. They also have improved mechanical properties at the operating conditions, such as high temperature, corrosive or wear environment, etc. This paper deals with the study of high temperature tensile properties of Al 2014-10 wt.% SiC composites prepared using stir casting method. The cast billets were extruded at 450 °C with ram speed of 2 mm/s and extrusion ratio of 8:1. The optical microscopy and scanning electron microscopy analysis were carried out on the as-cast and extruded base alloy and the composites. The room temperature and high temperature tensile properties were evaluated. The results of this study show that the extrusion process improves both the room temperature and high temperature strength of the base alloy and composites. The percentage increase is more in the case of base alloy than the composites.

INTRODUCTION

Aluminium alloys due to their high strength combined with low density, durability, machinability and availability play a significant role in the development of metal matrix composites. The superior specific strength, stiffness, wear resistance and improved high temperature properties of aluminium matrix composites (AMC) make it a potential material for automotive, aerospace and defence applications ^[1-3]. AMCs are widely used in a variety of automotive applications, namely connecting rod, drive shaft, cylinder liner, cylinder blocks and brake rotor ^[4-6], aircraft components, namely rotor vanes, fan exit guide vanes, etc. ^[7] and defence components, such as light weight body armour, track pad, etc. There are many methods available for the fabrication of particulate reinforced aluminium matrix composites such as diffusion bonding ^[8], powder metallurgy ^[9], infiltration techniques, spray deposition, stir casting, squeeze casting, etc. Among these methods, the conventional foundry based processes are quite promising. Further, the stir casting process is found to be more economical and suitable for mass production. The cost of preparing composite materials using casting route is about one-third to half that of the competitive methods and for high volume production, the cost will fall into one tenth ^[10]. Thermomechanical processes such as extrusion, rolling, forging etc. have been carried out on the cast composite billets in order to improve the strength and ductility by eliminating the cast defects. Among the above methods, hot extrusion is widely used for wrought aluminium alloys and their composites ^[11]. Hot extrusion has resulted in increased strength and tribological properties of the cast components ^[12,13].

Limited studies have been carried out on the evaluation of elevated temperature properties of composites, which are essential for components of engine, brake disc/drum, etc. This paper deals with the fabrication of Al 2014-SiC particulate composite, hot extrusion of the cast composites and the investigation of its microstructure and high temperature strength. The 2014 aluminium alloy is a high strength, heat treatable alloy widely used for aircraft components. Silicon carbide (SiC) is selected as a reinforcement owing to its high hardness and low coefficient of thermal expansion. SiC possesses high wear resistance and good mechanical properties, including high temperature strength and thermal shock resistance.

In preparing composites using casting route, factors such as particle distribution, wettability and interfacial reaction need considerable attention ^[14], because they affect the quality of the composite. Several approaches have been used by various researchers ^[15-17] to promote the wetting of ceramic particles by the metallic matrix. They are: (i) addition of alloying elements to the matrix alloy, (ii) coating of ceramic particles and (iii) treatment of ceramic particles. The SiC particles used in this study are coated with nickel to improve the wettability and to avoid unfavourable interfacial reaction. In this study, the base alloy and the composite were prepared using stir casting technique and further subjected to extrusion. Tensile testing of composites at room temperature and elevated temperature were carried out and the results have been discussed in detail.

EXPERIMENTAL SECTION

Materials

The matrix material used in this study is aluminium alloy 2014. The chemical composition of alloy was analyzed using an Optical Emission Spectrometer and the composition is found to be AI-5%Cu-0.4%Mg-1.2%Mn-1.2%Si. Silicon carbide particles having an average size of 30 µm was used as the reinforcement. The particles were given a thin metallic nickel coating using electro-less coating process in order to improve bonding.

Electro-less Ni coating of SiC particle

Initially, the SiC particles were dispersed in acetone for 15 min. to clean the surface contamination. After cleaning, the particles were immersed in nitric acid to roughen the surface. The particles were then sensitized by keeping in a solution containing 0.63 g SnCl₂, 0.2 ml HCl and 25 ml of water for 15 min. The sensitized SiC particles were further immersed in a solution containing 0.005 g PdCl₂, 0.2 ml HCl and 25 ml of water. Duration of 5 min. was adopted for the activation process. Finally, the palladium activated silicon carbide particles were kept in a nickel bath solution at 85 °C for about 30 min. The nickel coated SiC particles were then thoroughly cleaned in distilled water and air dried in an oven at a temperature of 100 °C for 30 min. The SEM images of the uncoated & coated particles and the EDS results of the coated particles are shown in Figure 1a-1c. The EDS spectrum of the coated particles as shown in Figure 1c confirms the presence of Ni. The quantitative results of various elements present in the coated SiC are also clearly seen.



Figure 1. SEM images of a) uncoated, b) coated SiC particles and c) EDS results of coated SiC particles.

Composite Preparation

The base aluminium alloy, in the form of ingots was melted in a 6 kW electric furnace. The SiC particles (10 wt.%) were preheated to 400 °C in a separate muffle furnace for a soaking period of one hour. The melt was degassed with the commercially

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available hexachloroethane tablets. The coated particles were then added manually through the vortex created by stirring the molten metal. The stirrer blade was made of stainless steel and coated with ceramic material to avoid iron pickup by the hot molten metal. The stirring was done at 300 rpm. After particle addition, stirring was continued for 15 min. The composite melt was finally poured into a preheated cast iron die. The set-up of stir cast furnace with stirrer arrangement is shown in Figure 2. The prepared base alloy and composite specimens were then heat treated to T6 condition. The samples were solutionised at a temperature of 530°C for duration of 2 hours and quenched in cold water. Quenching is followed by artificial ageing. The artificial ageing was carried out at a temperature of 180°C for a period of 6 hrs.



Figure 2: Stir cast set-up for composite preparation.

Extrusion

The cast base alloy and composites were machined and then solutionised at 400°C for 16 hours in a muffle furnace. They were then extruded at a ratio of 8:1 using a 200 t extrusion press. The extrusion process was carried out at a constant ram speed of 2 mm/s and billet temperature of 450°C. The extrusion die was heated to 350°C and the billets were given a graphite coat for lubrication.

Metallography and Tensile Test

The microstructure of the as-cast and extruded samples was studied using optical microscope make Mecji, Singapore. Scanning electron microscope (SEM) studies were carried out using Carl Zeiss make, Supra 55 model FESEM. Tensile tests at room temperature and at elevated temperatures of 100, 200, 300 and 350°C were performed using Instron make universal testing machine (model UTE 40) in accordance with ASTM standard E8M.

RESULTS AND DISCUSSION

Microstructure

The optical micrographs of the as cast base alloy and composite samples are shown in Figure 3 and that of extruded base alloy and composites are shown in Figure 4.



Figure 3. Optical micrographs of as-cast materials: a) Al 2014 alloy and b) Al 2014- SiC composite.



Figure 4. Optical micrographs of extruded materials: a) AI 2014 alloy and b) AI 2014-SiC composite

The microstructure of as-cast base alloy depicts primary α -Al dendrites. The optical micrograph of the as-cast composite shows non-uniform distribution of reinforcement particles. The particles are pushed to the regions, which solidify the last. Hence, the regional clustering/agglomeration of particles are found in the matrix. Microstructural inhomogeneity of the composites is observed as a result of interaction between the reinforcement particles and the moving solid-liquid interface during solidification. However, the particle/matrix interface is found to be good with no cavities existing in the particle/matrix interface. This could be achieved because of coating on the particles. This is due to the fact that the reinforcing ceramic particles act as the grain nucleation sites. Deng et al. in their study had explained that the dynamic recovery and hence the grain refinement is sensitive to the reinforcement particles in the cast composites ^[18].

The optical microscopy of the extruded base alloy (Figure 4a) reveals grain refinement to a certain extent. In the case of extruded composites (Figure 4b), the secondary phase, namely the SiC reinforcement is oriented along the direction of extrusion. Particle fragmentation is noticed at some places, which is due to the generation of large compressive forces during extrusion ^[19]. It is stated that generally larger sized particle fracture more easily than smaller ones ^[20]. Extrusion has improved distribution of the particles. This can be explained by the rearrangement of SiC particles caused by the severe plastic deformation during extrusion ^[21]. Extrusion causes the weakly bonded SiC particles in the agglomerate to shear and separate into individual particles. Unlike the as-cast composites, the grain refinement is more uniform in the case of extruded composites, due to the more uniform distribution of particles in the extruded composite. Moreover, the dynamic recrystallization can also cause the reduction in grain size. The SEM image of extruded composite is shown in Figure 5.



Figure 5. SEM image of extruded AI 2014- SiC composite.

The SEM image of the extruded composite indicates that the fragmented particles are oriented parallel to the direction of extrusion. No evidence of agglomeration is observed after extrusion. The particle/matrix interface seems to be good and the presence of detrimental intermetallic interface compound, namely AI_4C_3 or AI_4SiC_4 is not found. This is due to the fact that the Ni coating on the SiC particle prevents the undesirable interfacial reactions, which degrade the interface.

Tensile Properties

The room temperature and high temperature tensile test results of the as-cast base alloy and composites are shown in Figure 6. The reported strength values are the average of three specimens. It is observed from Figure 6 that the UTS and the YS of the composite are higher than that of base alloy at room temperature as well as at high temperatures. The composite strengthening is due to the increased dislocation density caused by the addition of SiC particles ^[22]. The increase in dislocation density is attributed the mismatch of coefficient of thermal expansion (CTE) between SiC particle and the Al 2014 matrix alloy ^[23]. It is also explained that the increase in strength of the composite compared to the base alloy is due to the shear transfer of load from the soft matrix

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to the hard reinforcements ^[24]. This behavior of composites compared to the base alloy is in line with many studies carried out elsewhere [^{14,17,20,25]}, while few literatures ^[26] reported lower strength of composites compared to their unreinforced counterpart. For example, Zhong et al. study on oxidized Al-Mg/SiC_p reinforced Al-Mg composites reported lower value of composite strength than the base alloy due to the interfacial reaction, in which MgO layers form at the particle–matrix interface resulting in depletion of Mg ^[26]. However, in the present study, no such detrimental interfacial reaction is found. This is attributed to the formation of sound interface caused by coating of SiC reinforcement particles.



Figure 6. Effect of temperature on UTS and YS of as-cast base alloy and composite

It is also found from the tension test results that the UTS and YS of both the composites and the base alloy decreases with increase in temperature. As the temperatures increases, the mobility of dislocation also increases leading to a lower flow stress. In the case of composites, the interfacial strength between the particle and matrix decreases at high temperature leading to debonding of particles from the matrix. This is the reason for the decreased strength values.

The strength values of extruded base alloy and composite at room temperature and at 300 °C are shown in Figure 7.



Figure 7. Effect of temperature on UTS and YS of extruded base alloy and composite

The extrusion process improves the yield strength and ultimate tensile strength of the base alloy and the composites, both at room temperature as well as at high temperature. After extrusion, the room temperature tensile strength values of the base alloy and the composite have increased by 63% and 17%, respectively compared to the as-cast condition. Similarly, the high temperature (300°C) tensile strength values of both the base alloy and the composite after extrusion have increased by 76%

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compared to the as-cast condition. After extrusion, the room temperature yield strength values of base alloy and composite have increased by 43% and 4%, respectively compared to the as-cast condition. Similarly, the high temperature (300 °C) yield strength values of the base alloy and the composite after extrusion have increased by 113% and 87%, respectively compared to the as-cast condition. A similar behavior of extruded composites at room temperature ^[27-30] and at high temperature ^[22] has been observed by other researchers. The increase in strength of extruded composites is attributed to the decreased grain size ^[31], orientation of particles in the direction of extrusion with uniform distribution of particles ^[32,33] and decreased porosity ^[31,34]. In this study, the uniform distribution and the preferred orientation of SiC particles (Figure 3b) in the direction of extruded base alloy is due to the elimination of cast defects during the deformation caused by extrusion and the substantial grain refinement.



Figure 8. SEM fractographs of specimens tested at 300 °C a) as-cast base alloy, b) extruded base alloy, c) as-cast composite and d) extruded composite

As in the case of as-cast condition, the strength of the composite is higher than the base alloy in the extruded condition also. The room temperature tensile strength of the extruded composite is found to be 8% higher than the extruded base alloy. The high temperature tensile strength of the extruded composite is found to be nearly 22% higher than the extruded base alloy. Though extrusion process improves the strength of both the base alloy and the composites, the increase is more significantly pronounced in the case of base alloy than the composites. It has been observed that the percentage increase of the room temperature and high temperature yield strength due to extrusion is very much higher in the case of base alloy compared to that of composites. Similar observation is found by Ranjit and Surappa ^[35]. In their study, they found that the increase in strength of Al8090 Al-Li alloy/18 vol.% SiC in the extruded condition is insignificant compared to that of unreinforced alloy. Also, Khalifa and Mahmoud studied the ambient and high temperature tensile properties of AI 6063 reinforced with 5, 10 and 15 wt.% of SiC, in the as-cast and extruded condition [36]. They reported that both for as-cast and extruded composites the tensile properties increase up to 10 wt.% SiC and with the further addition of reinforcement, there is a drop in the tensile strength. This behavior, in the case of extruded composites, is explained in the light of presence of ceramic particles and their fracture under the application of load during extrusion. In general, the improvement in strength after extrusion is due to the micro structural homogeneity, grain refinement and porosity reduction which are common for both base alloy and composites. However, in the case of composites, the presence and role of ceramic reinforcement has to be considered. The fracture surfaces of tensile tested base alloy and composite are shown in Figure 8. The fractograph of extruded base alloy is compared to that of as cast base alloy (Figure 8a and 8b). It reveals the presence of more dimples indicating that the former has undergone extreme plastic deformation before failure, resulting in the enhanced strength. The fractographs of both the as-cast composite and extruded composite show the presence of finer dimples indicating the ductile failure of the composites. However, the presence of microcracks and particle fracture in the extruded composites (Figure 8d) is the reason for final failure and the minimal increase of strength of extruded composites compared to extruded base alloy. As studied by Cocen and Onel, the failure of extruded composites is related to the particle cracking and void formation in the matrix within the cluster of small particles [25]. The microcracks often originate from the sharp corner of the SiC particles [37].

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The high temperature strength is lower than the room temperature strength for both the base alloy and the composite in the extruded condition. This behavior is similar to the case of as-cast condition. The strength of the base alloy and composite at elevated temperature is reduced compared to that at room temperature, which is due to the dynamic balance between hardening and softening at high temperature ^[22]. The decrease in strength of base alloy and composites at high temperature is attributed to the decrease of dislocation density by cross slip and dislocation climb phenomena ^[38]. This leads to a reduction in flow stress and strain hardening rate leading to a decrease in strength at elevated temperature. In the case of composites, the lower strength at higher temperature is due to the rapid coarsening of precipitates due to the presence of particles.

The ductility in terms of percentage elongation of the as-cast and extruded base alloy and composites at room temperature and at 300°C is shown in Figure 9.



Figure 9. Ductility in terms of % elongation of base alloy and composite

The ductility of composite is higher than the base alloy both at room temperature and at high temperature in the as-cast condition. This is contradicting to the findings of many of other researchers ^[39], who have reported that though composite materials possess higher strength than the unreinforced alloy, the ductility is lower due to the segregation of particles along the grain boundaries, which causes embrittlement. The higher ductility of composites, in this study, compared to the as-cast samples is due to the good interface between SiC particle and the aluminium matrix. The Al-SiC system is believed to be highly reactive system, leading to formation of interfacial compounds, namely Al_4SiC_4 and Al_4C_3 , which are brittle in nature resulting in lower elongation ^[40]. However, the application of thin layer of metallic or oxide coating on the surface of ceramic particle prevents such interfacial reactions ^[41]. In this study, the nickel coated SiC_p reveals good interfacial bonding causing enhanced ductility. The extrusion process has resulted in increased ductility of both the base alloy and composites. The lower ductility of as-cast base alloy and as-cast composite compared to the extruded ones is due to the inherent cast defects, namely porosity and the early void formation at lower strains during tensile testing.

In the extruded condition, the room temperature ductility of the composite is higher than the base alloy by about 30%. While the high temperature ductility of the extruded composite is lower than the extruded base alloy by 50%. This is attributed to the formation of cracks originating from the sharp corners of SiC particle as explained by the fracture surfaces in Figure 8. Molliex et al. demonstrated that the mechanical forces during plastic working cause microscopic damage, such as particle cracking and interfacial debonding, which result in early void formation during hot tensile test ^[42]. This lowers the ductility of composite.

CONCLUSIONS

In this work, the AI 2014 alloy and its composite reinforced with 10 wt.% SiC particle were fabricated by stir casting process. After subjecting to hot extrusion process, the microstructure, room temperature and elevated temperature tensile properties were studied. The main conclusions of this study are summarized below:

(1) Extrusion has resulted in microstructural homogeneity as well as uniform and oriented distribution of SiC particles in the aluminium alloy matrix.

(2) The room temperature and high temperature strength of the extruded composite is higher than the base alloy.

(3) The strength enhancement of extruded base alloy is much significant compared to that of extruded composite, which was explained by the particle fracture and origination of microcracks.

(4) The extruded base alloy and composite exhibits superior ductility compared to the as-cast samples. This is due to the use of coated particles which prevent unwanted interfacial reactions.

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