

Synthesis, characterization and properties of stir cast AA6351aluminium nitride (AlN) composites

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In the present investigation, AA6351 aluminum alloy matrix composites reinforced with various percentages of AlN particles were fabricated by stir casting technique. The percentage of AlN was varied from 0 to 20% in a step of 4%. The prepared AA6351-AlN composites were characterized using scanning electron microscope (SEM) and x-ray diffraction (XRD). The mechanical properties such as micro-hardness, compression strength, flexural strength, and tensile strength of the proposed composite have been studied. X-ray diffraction patterns confirm the presence of AlN particles in the composites. SEM analysis reveals the homogeneous distribution of AlN particles in the AA6351 matrix. The mechanical properties of the composite were found to be noticeably higher than that of the plain matrix alloy due to augmented particle content. The produced composites exhibit superior mechanical properties when compared with unreinforced matrix alloy. Fracture surface analysis of tensile specimens show the ductile–brittle nature of failure in the composites.

I. INTRODUCTION

Aluminum based metal matrix composites are continuously replacing traditional engineering materials which have drawn the attention of automotive, aerospace, structural, transportation, and marine industries due to their outstanding combination of high specific strength, high elastic modulus, light weight, high stiffness, and improved wear resistance compared with nonreinforced alloy matrix.¹⁻⁴ 6xxx Al-Mg-Si alloys are widely used as medium-strength heat-treatable alloys for structural applications due to their excellent formability, weldability, and good corrosion resistance.⁵ The AA 6351 aluminum alloy is used in ship manufacturing due to its strength, bearing capacity to sea atmosphere, ease of workability, and weldability. It is also used in building boat, column, chimney, rod, mold, pipe, tube, vehicle, bridge, crane, and roof. One of the most important properties of AA 6351 aluminum alloy is that the treatment of solid solution is not so critical.⁶ As a monolithic, the 6351 Al alloy is used in wrought form where the dendritic structure is removed by hot working. There would be possibilities of the suppression of dendritic solidification through ex situ incorporation or in situ generation of particulates during synthesis of 6351 Al based AlMMCs, particularly when the particulates act as the nucleation sites.⁷ The properties of aluminum matrix composites depend on the type, size, shape, mass fraction, and spatial dispersion of the ceramic particles.⁸ In recent decades, most of the researchers have been using a wide variety of ceramic materials such as SiC, B₄C, Al₂O₃, TiB₂, AlN, Si₃N₄, ZrB₂, and WC and they have been significantly used as reinforcement materials in AMCs.⁹⁻¹² Among those hard reinforcing particles, aluminum nitride (AlN) is a potentially attractive reinforcement which has excellent thermal and chemical stability, good mechanical properties, high electrical resistivity, low dielectric constant, good compatibility with aluminum alloy and it is used for electronic packaging and structural industry applications.¹³ Huashun Yu et al. suggested that, AlN is particularly attractive because of good wettability and high stability with molten aluminum, high hardness, and elastic modulus.¹⁴ The fabrication techniques of the aluminum matrix composites include mechanical alloying, squeeze casting, stir casting, compo casting, rheo casting, spray deposition, liquid infiltration, and powder metallurgy method.15

Among the above process the stir casting route is a liquid state method of processing, where the matrix

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material is completely melted in a liquid form and the ceramic reinforcing particles are then introduced to the molten matrix material. Liquid state of initial composite is well stirred by a mechanical stirrer for the uniform dispersion of reinforcement particles.^{16,17} Stir casting process has some incomparable benefits like excellent matrix-particle bonding, easier control of matrix structure, more uniform distribution of the reinforcement in matrix metal, simple and inexpensive processing, flexibility and applicability to mass production or large volume production when compared to other fabrication processes.¹⁸ Several researchers expounded that production of aluminum matrix composites mostly employ the stir casting technique. Stir casting is a reasonably lowcost fabrication method when compared to the other processing routes.¹⁹ Only a few studies on processing and characterization of aluminum alloys reinforced with aluminum nitride particulate composites were stated in the literature.²⁰⁻²⁴ Fogagnolo et al. fabricated AA6061/AIN AMCs using powder metallurgy method and examined the mechanical behavior of the composite.²⁰ Fale et al. synthesized Al/AlN AMC by in situ technique and evaluated the microstructure, hardness, and wear resistance of the aluminum matrix composites.²¹ Ashok Kumar et al. prepared AA6061/AlN using the liquid metallurgy method, wherein preheated AlN_p were added into a vortex to form the aluminum matrix composites. The mechanical properties such as tensile strength and hardness of the AMC increased linearly with the in-crease in AlN content.²² Nassaj et al. prepared Al/AlN by melt stirring method and reported that the incorporation of Mg leads to an augumentation of wettability of the AlN_p.²³ Fale et al. fabricated pure aluminum reinforced AlN by liquid metallurgy technique. They have examined the effect of AlN particulate size on the microstructure, mechanical, and tribological properties of the AMC.²⁴

According to research articles reported in the literature, only a few research findings have been reported on the mechanical properties of aluminum nitride based AMCs. As far as we know, no work is available on the AA6351 aluminum alloy reinforced with aluminum nitride particles fabricated by stir casting method. The present work focuses on the synthesis, microstructure characterization, and analysis of mechanical properties of AlN particulate reinforced AA6351 aluminum alloy composites prepared by stir casting route.

II. EXPERIMENTAL DETAILS

In this investigation, aluminum alloy AA6351 (Si: 1%, Fe: 0.6%, C: 0.1%, Mn: 0.5%, Mg: 0.7%, Cr: 0.25%, Zn: 0.1%, Ti: 0.2% and Al: balance) was used as a matrix material. The reinforcement used in this work is aluminum nitride (AlN) with the particle size of 325 mesh.

The SEM image of AlN particles is shown in Fig. 1. AA6351 ingots were placed inside the graphite crucible and melted in an electric furnace at a temperature of 850 °C. After reaching the molten state, the AlN particles were gently added at various weight percentages to yield the different composition such as, AA6351-0 wt% AlN, AA6351-4 wt% AlN, AA6351-8 wt% AlN, AA6351-12 wt% AlN, AA6351-16 wt% AlN, and AA6351-20 wt% AlN. The measured quantities of the AlN particles were preheated to a temperature of about 400 °C, which improves the wettability between the matrix and reinforcement, also removes the moisture within the reinforcement. The stirring was done for 10 min at the speed of 400 rpm. Then, magnesium is added in small amounts during stirring to increase the wettability.²³ After homogeneous stirring, the molten materials were poured into the cast iron mold of size $100 \times 100 \times 10$ mm.

The proposed composites were machined to prepare the samples for microstructure and mechanical characterization analysis. The microstructure was observed using a scanning electron microscope (SEM). X-ray diffraction (XRD) patterns were recorded using Panalytical x-ray diffractometer. Micro-hardness of the AA6351 and the composites were estimated by using Vicker's hardness tester (Wolper Wilson Instruments, Aachen, Germany) at a load of 0.5 kgf for a dwell time of 10 s. The micro-hardness was performed over the polished specimens at six different locations and the average values of all the readings were reported. Tensile tests were carried out using computerized universal testing machine (Model-INSTRON 8801, Instron, High Wycombe, United Kingdom) with a strain rate of 1.0 mm/min at room temperature. The tensile specimens were prepared as per ASTM E08 standard with a gauge length of 40 mm, a gauge width of 7 mm, and a thickness of 6 mm. Figure 2 shows the fabricated tensile specimens before and after fracture.

The compression strength of the AA6351 and the AA6351-AlN composites were determined using a computerized universal testing machine at room temperature. The compression specimens were prepared as per ASTM E9 standard. Three point bending tests were conducted to measure the flexural strength. The three point maximum bending load value was then converted into flexural strength. For every composition, the specimens with a size of $80 \times 11 \times 20$ mm³ were tested with a loading rate of 1.0 mm/min over a 50 mm span.

III. RESULTS AND DISCUSSION

A. SEM analysis

Figures 3(a)–3(g) illustrates the SEM images of as cast AA6351 and AA6351-AlN composites. From Fig. 3(a) depicts the presence of magnesium (Mg) and silica (Si) particles of the AA6351 alloy. Figures 3(b)–3(g) illustrates the SEM microphotographs of the AA6351-AlN composites. SEM micrographs reveal the majority of



FIG. 1. SEM image of AlN powders (a) higher magnification (b) low magnification.



FIG. 2. Photographs of tensile test specimens (a) before fracture (b) after fracture (ASTM E8).

evenly-dispersed AlN particles in the aluminum matrix alloy. From the micrographs, it clearly displays that there was no voids in the composite and also depicts no other massive reaction products occurred between AA6351 matrix and AlN reinforcement particle, and that indicates an excellent interfacial bonding between the AlN and matrix alloys. The addition of a small amount of Mg to the molten aluminum increases the wettability,²³ which in turn enhances the strong mechanical bonding between the AlN and AA6351. A homogeneous distribution and the fine clear interface of AlN is vital to achieve an effective load bearing capability of the composite and it enhances the mechanical properties of the AA6351-AlN composites.

No agglomeration was observed for the composite contains 20% of reinforcement as shown in Fig. 3(f). It was achieved by suitable mechanical stirring of molten composite during casting. Figure 3(g) displays the clear interface of AA6351-20 wt% AlN composites, which exhibits reaction free and strong interface between the AlN and AA6351 matrix. From the above investigation, it has been declared that AlN particles are successfully dispersed in the AMCs. Similar behavior has been observed by Chen et al. in the case of AA2219/TiB₂ AMCs.²⁵

B. XRD analysis

XRD analysis was carried out on alloy and composite specimens in which fabricated by stir casting with

a weight percentage of 0, 4, 8, 12, 16, and 20 AlN respectively as shown in Fig. 4. The XRD pattern proved the presence of phases related to AA6351 aluminum alloy, AlN particles in the developed composites. The obtained pattern indicates the absence of interfacial reaction product in the composites. The occurrence of AlN peaks ensures the presence of AlN particles in the composite. The AlN peaks increase with the increase of the weight percentage of AlN. These XRD results exhibit the presence of aluminum (in the largest peak) and the presence of AlN (revealed by minor peak). It is evident in (Fig. 4) that the AA6351 peaks in the developed composites are somewhat shifted to lower 2 theta when compared to that of AA6351 plain matrix alloy owing to the inclusion of AlN particles in the AA6351 matrix. No other massive reaction product was detected in the composites, which confirms that AlN particles were thermodynamically stable.

C. Effect of AIN on hardness of AA6351-AIN composites

Figure 5 depicts the effect of addition of AlN on micro-hardness of AA6351-AlN composites. It is evident that as the weight percentage of AlN increases, a remarkable improvement in hardness was observed on the produced composites. Hardness of the composite was mainly depends on the hardness of the matrix and the reinforcement. AA6351 matrix alloy exhibits the hardness of 67 HV and the AlN reinforcement exhibits the



FIG. 3. (a-g) SEM images of AA6351 and AA6351-AlN composites.



FIG. 4. XRD patterns of AA6351 and AA6351-AlN composites.



Vicker's hardness of 12 GPa. When the 20 wt% of AlN is introduced the microhardness increases by 35.86% as compared to the base alloy. During the processing of the composites, the hard AlN enhances the large quantity of dislocation density which offers more enhanced resistance to the plastic deformation resulting in improved hardness. Thus, the present investigation concludes that the composite containing 20 wt% of aluminum nitride poses maximum values of micro-hardness. A similar improvement is also reported by the researcher Sakip Koksal et al. for the Al-AlB₂ composite.²⁶

D. Effect of AIN on tensile strength of AA6351-AIN composites

Figure 6 exhibits the influence of the AlN content on the ultimate tensile strength of AA6351-AlN composite.



FIG. 6. Tensile strength of AA6351 and AA6351-AlN composite.

The ultimate tensile strength of AA6351 is 133 MPa but when the 4 wt% of AlN is reinforced with a matrix it increases to 150 MPa and further with the addition of 8. 12, 16, and 20 wt% of AlN it peaks to 245 MPa. The tensile strength of the composite is higher than that of the nonreinforced AA6351 matrix alloy. The presence of AlN particles in the composites enhanced the tensile strength of the composites. It could be ascribed to the fair homogeneous dispersion of reinforcement in the matrix material.^{11,25} The increase in ultimate tensile strength is also due to strong mechanical bonding between the AA6351 matrix alloy and the AlN particles as evident by the SEM images shown in Figs. 3(a)-3(g). The difference in coefficient of thermal expansion between the matrix and the reinforcement, generates a massive amount of dislocation density around the surface of the reinforcement particles, during the solidification of the composites and which can effectively serve as an impedance to the dislocation movement of the AA6351 alloy matrix. Hence, the tensile strength of the composites increases linearly with the increase in AlN reinforcement content. The result has good agreement with previous researchers who reported for stir cast aluminum matrix composites.^{13,22,24} Further, escalation in tensile strength may be ascribed to the reaction free, clear, and stable interface bond, which can transfers and effectively distributes the load from the AA6351 matrix material to the AlN reinforcement. Therefore tensile strength of the composite increases steadily with the increase in the weight percentage of AlN.

E. Tensile fracture surface analysis of AA6351-AIN composites

Figures 7(a)-7(f) show tensile fracture morphology of the unreinforced alloy and the AA6351-AlN composites. Evenly distributed and bigger size dimples are visible in



FIG. 7. (a-f) Fracture surface images of AA6351 and AA6351-AIN composites.

the fracture morphology [Fig. 7(a)] of aluminum matrix alloy (AA6351). It clearly indicates that the fracture mode is ductile. Figs. 7(d) and 7(e) clearly shows the ductile– brittle nature of failure in the composites and it had been attributed by brittle nature of AlN. The fracture morphology of the produced AA6351-20 wt% AlN composites [Fig. 7(f)] show tinier size dimples compared to that of matrix alloy. The addition of AlN particles to the AA6351 matrix alloy refined the grain size of matrix which resulted in tinier size dimples. The AlN particles are observed to be intact at many places due to excellent bonding between the matrix and the reinforcement. These findings agree with the earlier studies by various researchers.^{11,13,22,25}

F. Effect of AIN on compressive strength of AA6351-AIN composites

The compressive strength of the AA6351 and the AA6351-AlN composites are depicted in Fig. 8. The graph exhibits that the compression strength increases with

increase in weight percentage of AlN in the composite which serves as obstacles to the passage of dislocation. This obstacle leads to increase the compression strength of the composite.¹² Therefore, the compression strength of the composites is dramatically increased. The enhanced compressive strength of the composites can be mainly ascribed to the presence of evenly and more homogeneously dispersed hard AlN particle.²⁷ Therefore, the increasing rate of the compression strength is drastically high at 20 wt% of AlN_p content when compared to the non-reinforced matrix alloy. The reason for the increase in compression strength of composite is the presence of hard nature of AlN particles in the soft matrix which resists the plastic deformation during compressive load.

G. Effect of AIN on flexural strength of AA6351-AIN composites

From Fig. 9 it can be seen that the increase in the weight percentage of AlN in AA6351 matrix significantly affects



FIG. 8. Compression strength of AA6351 and AA6351-AlN composite.



FIG. 9. Flexural strength of AA6351 composite alloy.

the flexural strength of the composites. The flexural strength of composites increased drastically when compared to the plain matrix alloy in accordance with the increase in the amount of reinforcement content.²⁸ This can be attributed to the transfer of the load from the matrix metal to the reinforced AlN particles. The increase in flexural strength is also due to excellent interfacial bonding between the aluminum nitride particles and the AA6351 matrix material. The maximum value of flexural strength of the composite was observed as 578.18 MPa for composites contain 20 wt% of AlN.

IV. CONCLUSIONS

Experimental investigation of stir casting for AA6351-AlN composite was successfully fathomed and its results were summarized as follows. (1) SEM micrographs reveal the majority of even dispersion of AlN particles throughout the AA6351 matrix and the XRD pattern evident the presence of AlN in the composite.

(2) The hardness of AA6351-20% AlN composites increased by 35.86% as compared to the unreinforced matrix alloy.

(3) AA6351-20% AlN composite reveals 84.21% greater tensile strength and 53.07% superior compression strength when compared to AA6351 plain matrix alloy.

(4) The excellent interfacial bonding between AlN particles and matrix enhanced the mechanical properties of the composite. The AA6351/AlN composite containing 20 wt% of AlN has higher mechanical properties than the plain matrix alloy.

(5) Tensile fracture surface analysis reveals the ductilebrittle fracture phenomena for AA6351-AlN composites and ductile mode of fracture for AA6351 alloy.

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