



## Microstructure and mechanical properties of rutile-reinforced AA6061 matrix composites produced via stir casting process

Subramanya R. PRABHU<sup>1,2</sup>, Arun K. SHETTIGAR<sup>2</sup>, Mervin A. HERBERT<sup>1</sup>, Shrikantha S. RAO<sup>1</sup>

1. Department of Mechanical Engineering, National Institute of Technology Karnataka, Surathkal, India;

2. Department of Mechatronics Engineering, Manipal Institute of Technology,  
Manipal Academy of Higher Education, Manipal, India

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**Abstract:** A novel process of fabricating aluminium matrix composites (AMCs) with requisite properties by dispersing rutile particles in the aluminum matrix was studied. A novel bi-stage stir casting method was employed to prepare composites, by varying the mass fractions of the rutile particles as 1%, 2%, 3% and 4% in AA6061 matrix. The density, tensile strength, hardness and microstructures of composites were investigated. Bi-stage stir casting method engendered AMCs with uniform distribution of the reinforced rutile particles in the AA6061 matrix. This was confirmed by the enhancement of the properties of AMCs over the parent base material. Rutile-reinforced AMCs exhibited higher tensile strength and hardness as compared with unreinforced parent material. The properties of the composites were enhanced with the increase in the mass fraction of the rutile particles. However, beyond 3 wt.% of rutile particles, the tensile strength decreased. The hardness and tensile strength of the AMCs reinforced with 3 wt.% of rutile were improved by 36% and 14% respectively in comparison with those of matrix alone.

**Key words:** AA6061–rutile composite; bi-stage stir casting; microstructure; mechanical properties; fractography

### 1 Introduction

Aluminium matrix composite (AMC) refers to the class of high performance, lightweight aluminium centric material with two constituent parts, aluminium being a matrix and a metal, ceramic or organic compound being a reinforcing material [1]. These AMCs are found in a variety of applications in marine, automotive and aerospace industries due to their superior qualities than the base material. AMCs exhibit ameliorated properties in terms of specific mass ratio, abrasion resistance, corrosion resistance, wear resistance, etc [2–4]. Also certain properties such as thermal and electrical conductivities and damping property can be tailored according to the application. AMCs have already replaced aluminium alloys in several applications where the tribological behavior of the material plays a critical role [5–7].

The reinforcing materials are the primary load bearing components in the AMCs. Therefore, particles having better mechanical properties than the matrix are selected while fabricating a composite. Several

researchers prepared AMCs by incorporating particles like SiC, B<sub>4</sub>C, Al<sub>2</sub>O<sub>3</sub>, ZrB<sub>2</sub>, TiC and TiB<sub>2</sub> and studied their mechanical and tribological properties [8–14]. Nowadays, minerals are transpired as potential reinforcement materials due to the environmental facets. Minerals are inexpensive, abundantly available and environment friendly materials which make them a vital reinforcement for the composites [15,16].

Rutile is a copiously available low cost mineral, composed mainly of titanium dioxide (TiO<sub>2</sub>). Being a natural form of TiO<sub>2</sub>, rutile exhibits more significant wear resistance, better electrical and mechanical properties and lower coefficient of thermal expansion [16–18]. RAMESH et al [19] worked on AA6061–TiO<sub>2</sub> composite and revealed that the composite showed higher hardness and better wear resistance properties. CHAUDHURY et al [20] reinforced rutile particles with Al–2Mg and found that there was substantial improvement in the mechanical properties. TROMANS and MEECH [21] showed that the rutile particles possessed excellent physical and engineering properties. AKBARI et al [22] noticed that TiO<sub>2</sub>-reinforced composite exhibited better wear resistance properties than the parent matrix

material. From the available literature, it is understood that very meager study has been carried out concerning rutile powder as reinforcement in Al-based MMCs. Hence, the present study has focused on the influence of rutile particles on the mechanical properties of the AA6061 based composites processed by a novel bi-stage stir casting method. The studied material was proposed for automotive, aerospace and marine applications. It is suitable for connecting rods, pistons, bike frames, valves, brake pistons, marine structures, marine fittings, aircraft couplings and fittings [23].

Depending on the kind of reinforcing elements, shape, size and morphology, the composites were prepared by various techniques such as squeeze casting, stir casting, liquid infiltration, spray deposition, vacuum hot pressing, powder metallurgy and friction stir processing (FSP) [24–26]. In powder metallurgy, the reinforcement and matrix powders were blended together and then subjected to sintering followed by plastic working. In casting, the reinforcements were added into the molten matrix at atmospheric pressure. In FSP, particles were reinforced into the matrix which was in solid state.

Among various processing methods, stir casting is one of the promising, commercially viable methods to process AMCs [27]. Being a liquid state method of fabrication, stir casting offers several advantages such as flexibility, simplicity and capability to produce huge magnitude of products at lower cost. Stir casting helps in fabrication of large sized product to the near-net shape. Stir casting technique can be adopted for wide range of materials, as it offers numerous processing conditions. One can achieve uniform dispersion of reinforcing particles in the matrix through this method. The reduction in the cost of processing composite through this technique is up to 66% in comparison with other available techniques. And, it is reduced to 10% for the high volume production [17,19]. Moreover, nowadays stir casting technique is commercially used to process Al-based composites [28,29].

Several factors need to be considered while preparing composites by stir casting technique. The distribution of reinforcements in the matrix, wettability between particle and the matrix, chemical reaction between matrix and reinforcing particle and the porosity in the cast AMCs are the factors to be considered while manufacturing the composite [30]. To obtain superior quality AMCs, the particles must be uniformly dispersed in the matrix and the bonding or wettability between the particles and the matrix need to be proper. To achieve a proper mixing of the particle and to obtain uniform distribution, bi-stage stir casting method was used. Wettability between the particles and matrix was improved by adding magnesium prior to the introduction

of particle into the matrix.

The present study focused on the fabrication of AMCs reinforced with rutile particles by using an improved bi-stage stir casting technique. The influence of mass fraction of the particles on the mechanical properties of the AMCs was evaluated.

## 2 Experimental

### 2.1 Processing of composites

In the present study, a modified stir casting technique was used to fabricate AMCs using AA6061 alloy as matrix and rutile with an average size of 10  $\mu\text{m}$  as reinforcement particulate. Table 1 lists the chemical composition of AA6061 matrix material. 3.0 kg ingot of AA6061 Al alloy was melted at 800  $^{\circ}\text{C}$  in a ceramic crucible using an electric furnace. The setup was covered by a suitable material to avoid the absorption of gases. Hexachloroethane tablets were added into the molten metal for degassing the melt. To enhance the wettability of reinforcement with the molten metal, 1 wt.% Mg was added to the molten AA6061 matrix. The addition of Mg improved the wettability of the reinforcement by reducing the surface tension of molten metal. Adding beyond 1 wt.% Mg into the melt altered the viscosity of the melt and thereby imperiled the proper distribution of the particles [30]. Simultaneously, rutile particles of given quantities were preheated at 700  $^{\circ}\text{C}$  for 1 h to remove any moisture content from the particle. Preheating also enhanced the wettability of the particle by forming a layer of oxide on its surface. Prior to the addition of particles, the melt was stirred using graphite-coated steel stirrer to form the vortex. Stirrer was immersed at 1/3 vortex from the top surface and operated at a speed of 300 r/min. Preheated particles were added to the melt at a constant temperature of 800  $^{\circ}\text{C}$ , in two steps to avoid the agglomeration of rutile particles in the molten metal. The addition of particles into the melt in single step severely altered the viscosity of the melt, thereby affecting the proper mixing of particles in the matrix. The addition of particles into the melt in two steps separated by a small time interval greatly enhanced proper dispersion of particles in the matrix. Stirring continued for 5 min after the addition of the particles. Nitrogen gas was purged on the surface of the melt during stirring to reduce oxidation. Finally, the composite was poured into the die and preheated at a temperature of 450  $^{\circ}\text{C}$ . Preheating of the die avoided

**Table 1** Chemical composition of AA6061 matrix material (wt.%)

Mg	Si	Fe	Mn	Al
0.8–1.2	0.4–0.8	0.7	0.15	Bal.

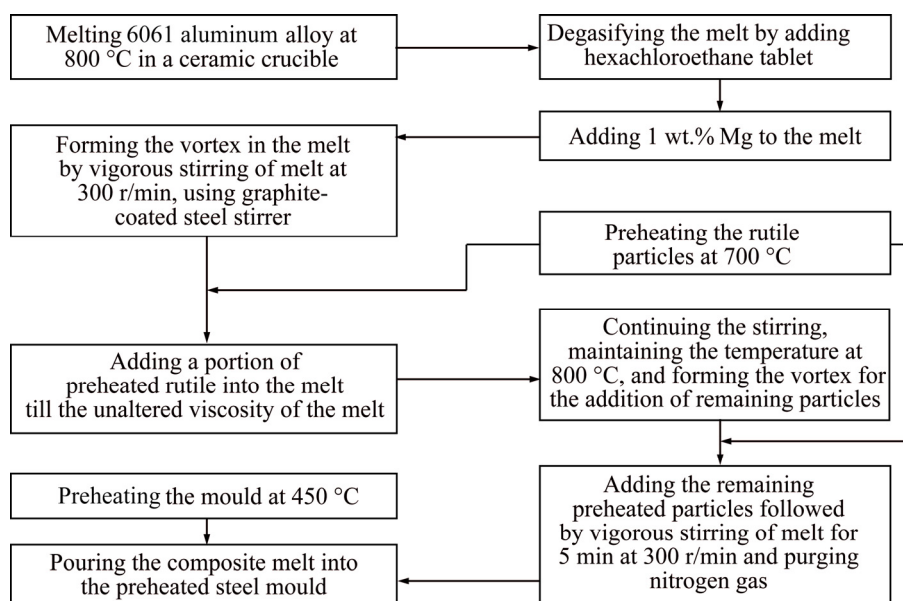


Fig. 1 Schematic representation of bi-stage stir casting process

sudden drop of temperature of molten metal as it was in contact with the die and ensured that slurry was in molten condition throughout the pouring. Figure 1 depicts the schematic representation of bi-stage stir casting process.

## 2.2 Tensile test

The specimens for the tensile test were prepared as per ASTM E8M standard as shown in Fig. 2. For each type of synthesized composite, five specimens were prepared for the test and the average of these values was considered as parameter value. The test was performed on a computerized tensometer (Maker: KUNDALE), which had a loading capacity of 20 kN. The cross slide speed was set to be 0.5 mm/min. Furthermore, the surface fractured during tensile test was examined for microstructural analysis using scanning electron microscope (SEM).

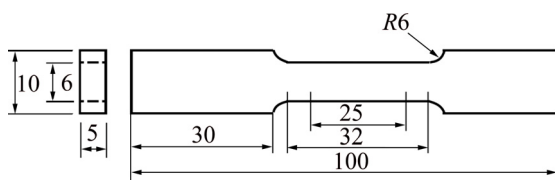


Fig. 2 Schematic view of tensile test specimen (unit: mm)

## 2.3 Hardness test

The hardness of the composite was measured using Vickers macro hardness tester (VM-120). The sample surface was polished prior to the hardness test. Test was carried out with an indentation load of 5 kg for 15 s. Readings were taken at eight different points on the

surface and the average of these values was considered as hardness value of the composite.

## 3 Results and discussion

### 3.1 XRD patterns

X-ray diffractometer (XRD) (Rigaku Miniflex 600, the 5th generation) was used to determine the details about various elements present in the prepared composites. XRD also provides the information about existence of any other phases in the as-cast composites. XRD images shown in Fig. 3 indicate the existence of  $\text{TiO}_2$  particulates within the matrix. The increase in rutile particles also shows an increase in intensity of  $\text{TiO}_2$  peaks in the XRD pattern. XRD patterns indicate that aluminium and  $\text{TiO}_2$  are the primary elements existing in the prepared composite and also imply the absence of intermetallic and impurities in the composite.

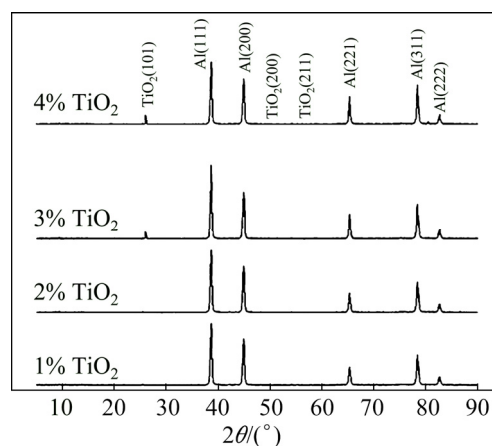


Fig. 3 XRD patterns of AA6061–rutile composites

### 3.2 Microstructures

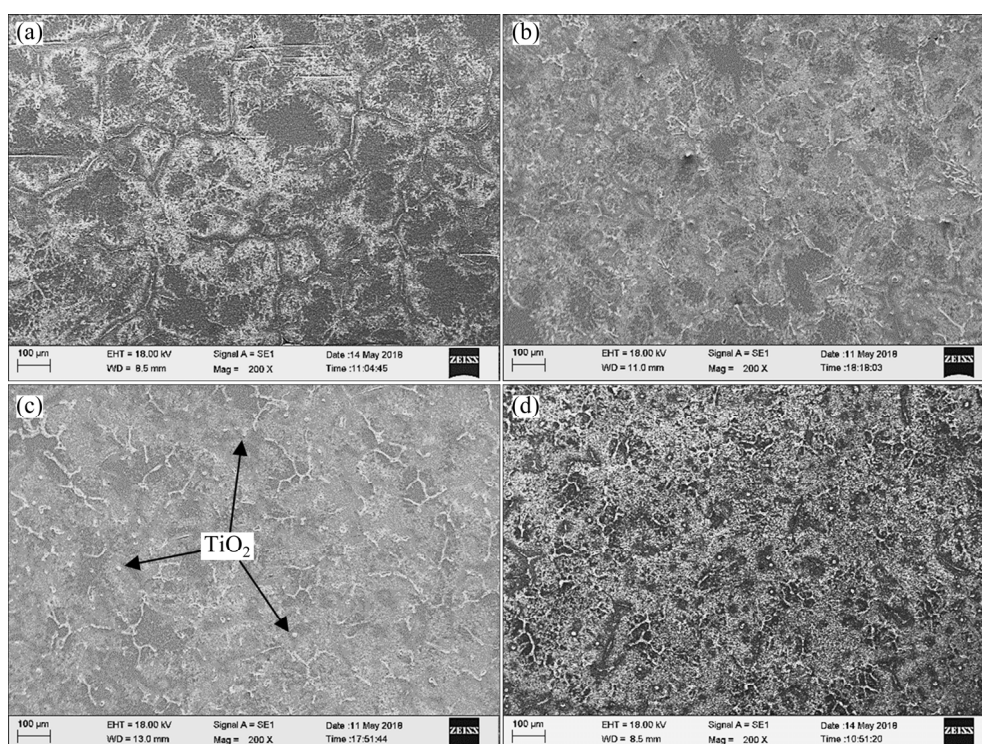
The microstructural study was performed using scanning electron microscope (SEM) (EVO MA18 with Oxford EDS(X-act), Maker: Zeiss). Figure 4 shows SEM images of the prepared composites with different contents of reinforcements, exhibiting typical dendritic structure. Reasonably uniform dispersion of rutile particles in the matrix with the absence of agglomeration of particles is observed. The addition of Mg into the melt during fabrication of composite produces a transitory layer between the matrix and the reinforcements, which greatly enhances the wettability. Low wetting angle of transitory layer reduces the surface tension of the liquid matrix, resulting in surrounding of particles with a similar structure of matrix alloy and the particles [30]. Proper wetting of rutile particles results in uniform distribution of the particles.

The agglomeration of hard reinforcement particles, a common defect that has been seen in the AMCs in the past, usually results in toughness reduction in the composite. Such a type of defect is not seen in the stir cast composite. The reason for better dispersion of rutile particle in the matrix without the agglomeration of particles could be the bi-stage addition of the particles into the melt. The improved bi-stage addition of particle into the melt probably controls the viscosity of the molten matrix. When the preheated reinforced particles were added all at once to the molten alloy the viscosity of the melt increases, which creates problem in the proper stirring of the melt. Possibly, the bi-stage addition

of particles eliminates the difficulty in stirring, thereby avoiding agglomeration of particles. This results in fairly uniform dispersion of reinforcing particles in AA6061 matrix.

Compared with the composites with 1%, 2% and 3% reinforcements, the composite with 4% reinforcement shows slightly heterogeneously distributed, agglomerated rutile particles in the matrix, as seen in Fig. 4(d). The wettability of particles controls the incorporation of rutile particles into the molten AA6061. Though the wettability was improved by the addition of magnesium into the melt, the addition of  $\text{TiO}_2$  only up to 3% mixes properly with the melt. However, up to 20% of  $\text{Al}_2\text{O}_3$  and SiC can be incorporated into the molten aluminium [31]. This is due to the fact that  $\text{TiO}_2$  has much lower apparent density compared with the SiC and  $\text{Al}_2\text{O}_3$ . The ratio of density to apparent density is much greater in the case of rutile particles, which probably leads to improper incorporation of particles in the matrix [31] during the addition of  $\text{TiO}_2$  beyond 3%.

The SEM images exhibited that the addition of rutile particles reduced the grain size of the matrix metal depending on the mass fraction of the rutile. As the mass fraction of the rutile increased the grain size decreased. The presence of the reinforcements at the grain boundaries suppressed the grain growth due to pinning effect and hence, the size of the grains became smaller and finer [16]. Higher mass fraction of rutile showed higher grain refinement.



**Fig. 4** SEM images of stir cast AA6061–rutile composite reinforced with different mass fractions of rutile particles: (a) 1%; (b) 2%; (c) 3%; (d) 4%

### 3.3 Density

Figure 5 depicts the theoretical density and the measured density of prepared composites. The measured values indicate that presence of rutile has miniscule effect on the density of prepared composites. The actual densities are close to the theoretical values, which indicates that bi-stage stir casting technique yields composite with minor pores. Small difference in the theoretical and actual densities is probably due to some of the particles entrapped into the slag [32]. The density of rutile particles is  $4.2 \text{ g/cm}^3$ , whereas density of base matrix alloy is  $2.7 \text{ g/cm}^3$ . The maximum density is  $2.73 \text{ g/cm}^3$  for the composite reinforced with 4 wt.% rutile particles with an increase of 1.12%. This increase in density is due to the presence of higher density rutile particles in the matrix of lower density. As the addition of rutile particles increases, the porosity of the composite also increases, showing the maximum porosity of 1.09% for the composite reinforced with 4 wt.% of rutile. The increase in porosity may be due to the gas entrapment during stirring, during pouring melt from crucible to mold or due to the shrinkage during solidification. It has been observed that the addition of rutile particles beyond 3 wt.% significantly increases the porosity. This may be due to the clustering of particles in the matrix and non-uniform dispersion of the particles resulting from the poor wettability with higher mass fraction of reinforcements [31].

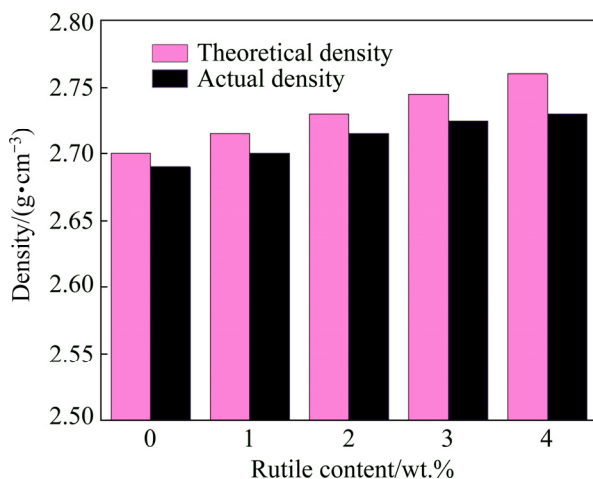


Fig. 5 Effect of rutile content on density of composites

### 3.4 Mechanical properties

Table 2 lists the mechanical properties of AA6061–rutile composites with various mass fractions of rutile particles. The presence of hard rutile particles and the porosities surrounding the particles are the major influencing factors in evaluating the AMCs ductility and other mechanical properties. It has been observed that

mechanical properties such as hardness and strength are enhanced at the cost of ductility. The composite shows increasing trend of mechanical properties with the increase in mass fraction of hard rutile particles in the AA6061 matrix.

Table 2 Mechanical properties of AA6061–rutile composites with various mass fractions of rutiles

Mass fraction of rutile/%	Yield stress/MPa	Ultimate stress/MPa	Elongation/%	Hardness (HV)
0	95±3	145±2.5	8.8±0.3	63±2
1	99±2.5	152±3	8.2±0.2	73±1.5
2	107±3	159±2.5	7.9±0.2	78±1.5
3	112±2	165±2	7.5±0.3	86±2
4	102±3	155±3	7.1±0.1	91±3

Figure 6 shows the hardness values of the AA6061 matrix and the composites prepared using bi-stage stir casting method. The hardness test indicates that there is a significant increase in the hardness value with increasing content of rutile particles in the matrix. With the increase of rutile particles, the hardness of specimen increases up to 50% compared with that of the base matrix alloy.

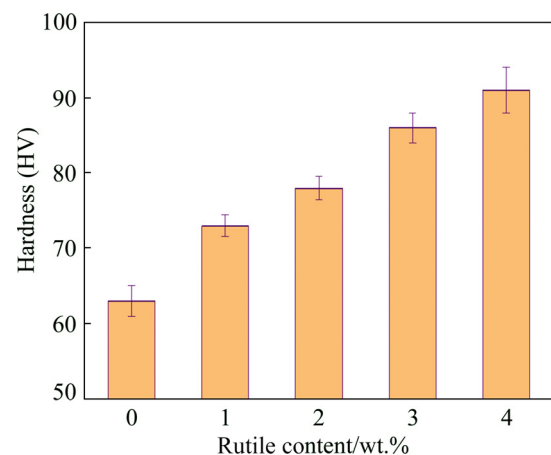


Fig. 6 Variation of hardness of composites with various mass fractions of rutile

It is evident that the inclusion of hard particles into the less hard ductile matrix enhances the hardness by various mechanisms [33]. Harder reinforced particles act as hurdles for the movement of dislocation. The presence of small individual hard reinforcements in the matrix can hamper the motion of dislocations, provided these reinforcements are stronger than the base material. Compared with the matrix, rutile particles have higher stiffness and hardness and enhance the matrix resistance to plastic deformation. Hence, the hardness of the composites also increases. Also, the reinforced particles improve the hardness of the composites by reducing the

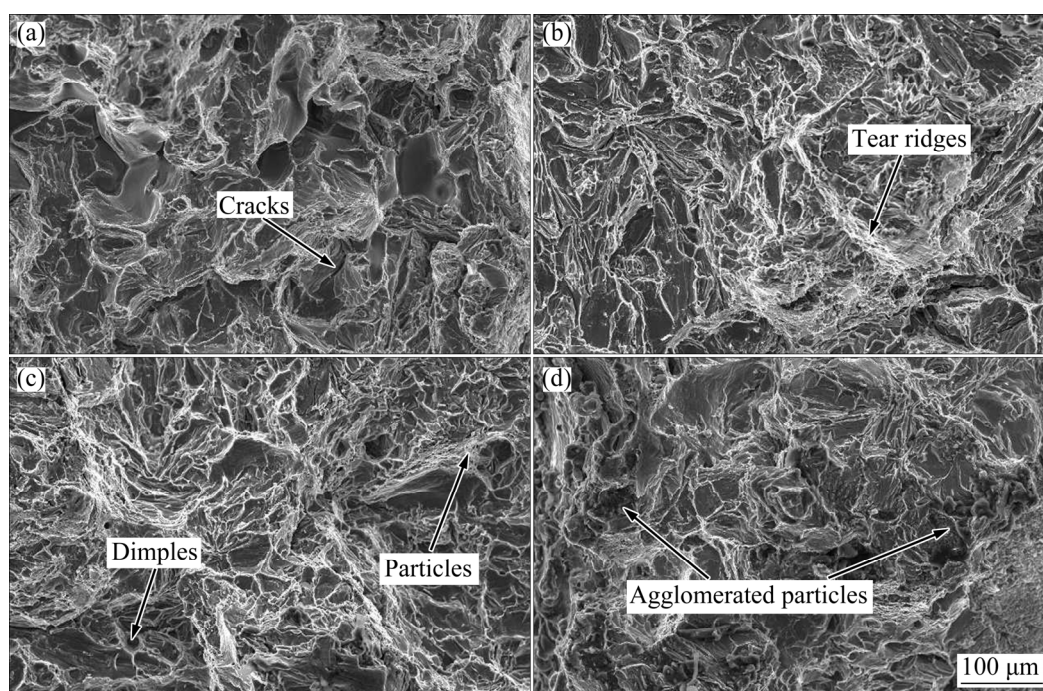
grain size of matrix material. In stir casting, hard particles are added to the molten matrix and these particles offer preferred heterogeneous nucleation sites for the matrix grains. The matrix microstructure gets refined by increasing the content of rutile particles. This leads to the increase of hardness of composites with the increase of reinforcement content in the matrix.

Table 2 indicates that the ultimate tensile strength (UTS) and yield strength (YS) remarkably increase by the addition of rutile particles, at the expense of ductility. AMCs containing 3 wt.% of rutile have exhibited the highest strength. Increasing the mass fraction of the particles improves the load bearing capacity and results in higher yield strength. The grain refinement due to the addition of rutile particles also contributes to the increase of the yield strength [34]. Grain boundaries act as obstacle for the movement of dislocation and thereby result in the increase of yield stress. As per Orowan mechanism, uniformly dispersed, high volume, fine and stable particles act as obstacle for the movement of dislocations [29]. Hence, the dislocation loops developed surrounding the particles increase the stress required for the deformation, resulting in higher strength. However, the composite with 4 wt.% rutile particle shows slightly lower UTS and YS. This may be due to the segregation and the uneven distribution of reinforced particles in the matrix. Also extensive formation of porosity due to shrinkage at the interface considerably reduces the strength of the AMCs, even though it has higher content of rutile particle.

On the contrary, the brittle nature of rutile particle reduces the ductility of AMCs. This could be due to the existence of rutile which leads to strain hardening during deformation, thereby reducing its ductility [35,36]. Also, the existence of porosities (especially at the interface of AA6061 matrix and the rutile particles) could be another reason for the reduction of ductility in the composites.

### 3.5 Fractography

The particle–matrix interfacial bonding strength plays a vital role in evaluating the fracture mode of the AMCs. During deformation, particle fracture will occur if the interfacial bonding strength is high. Contrarily, if the bonding strength is weak, decohesion between aluminium matrix and the rutile will happen prior to the particle fracture [37,38]. Figures 7(a–c) depict the particle fracture mode for the AMCs having strong interfacial bond. The existence of dimple in the aluminium matrix is also evident for this composite. It can be observed that the cracks are initially formed in the hard and brittle reinforced particles and move into the matrix, resulting in final fracture. However, in Fig. 7(d) debonding of particles is visible at matrix particle interface. Debonding of rutile particle prior to the matrix deformation indicates the weaker interfacial bonding and exhibits lower tensile strength. So, it may be concluded that increasing content of reinforcements beyond 3% does not have favorable effect on composite strength.



**Fig. 7** SEM images taken from fracture surfaces of AA6061–rutile composites with different mass fractions of rutile: (a) 1%; (b) 2%; (c) 3%; (d) 4%



## 4 Conclusions

(1) The fabrication of AA6061–rutile composites was successfully done using a novel bi-stage stir casting technique.

(2) XRD analysis shows that aluminium and rutile particles are the principal elements present in the composites and that the prepared composites do not have any impurities. SEM microstructural study indicates that the rutile particles are distributed uniformly in the matrix.

(3) The density of the fabricated composite is higher than that of the base material due to the presence of high density rutile particles.

(4) The mechanical properties such as hardness and tensile strength are enhanced with the increase of content of rutile particle in the composite, at the expense of ductility. The hardness values of the composites are improved by 15%, 24%, 36% and 44% by the addition of 1%, 2%, 3% and 4% rutile particles respectively in comparison with the base material.

(5) The tensile strengths of the composite are improved by 5%, 10%, 14% and 7% by the addition of 1%, 2%, 3% and 4% rutile particles respectively in comparison with the base material.

(6) The addition of rutile particles beyond 3 wt.% does not have favorable effect on the tensile strength of composite due to the agglomeration as well as heterogeneous distribution of particles.

(7) SEM study reveals that the failure of the composite gradually shifts from ductile to brittle behavior with an increased content of rutile particles in the composite.

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## 搅拌铸造金红石增强 AA6061 基复合材料的 显微组织和力学性能

Subramanya R. PRABHU<sup>1,2</sup>, Arun K. SHETTIGAR<sup>2</sup>, Mervin A. HERBERT<sup>1</sup>, Shrikantha S. RAO<sup>1</sup>

1. Department of Mechanical Engineering, National Institute of Technology Karnataka, Surathkal, India;

2. Department of Mechatronics Engineering, Manipal Institute of Technology,  
Manipal Academy of Higher Education, Manipal, India

**摘 要:** 采用一种新型的双级搅拌铸造法制备铝基复合材料(AMCs)。将不同质量分数的金红石颗粒(1%, 2%, 3% 和 4%)分散于 AA6061 基体中, 研究复合材料的密度、抗拉强度、硬度和显微组织。双级搅拌铸造法使金红石颗粒均匀分布于 AA6061 基体中, AMCs 的性能相较于母材得到提高。与未增强的母材相比, 金红石增强的 AMCs 具有较高的抗拉强度和硬度, 且增强效果随金红石颗粒含量的增加而增加。然而, 当金红石颗粒质量分数超过 3% 时, 样品的抗拉强度降低。与母材相比, 3%金红石颗粒增强的 AMCs 的硬度和抗拉强度分别提高了 36% 和 14%。

**关键词:** AA6061–金红石复合材料; 双极搅拌铸造; 显微组织; 力学性能; 断口形貌

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