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Friction stir processing of intermetallic particulate reinforced aluminum matrix composite

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ABSTRACT

Friction stir processing (FSP) is a novel solid state technique to refine the microstructure of metallic materials. The objective of this work is to apply FSP to change the morphology and distribution of intermetallic particles and achieve property enhancement. AA6061/8wt. % Al_3Zr composite was produced by the in situ reaction of molten aluminum and inorganic salt K_2ZrF_6. Optical and scanning electron micrographs revealed a uniform distribution of needle shape Al_3Zr particles in the aluminum matrix. The Al_3Zr particles were located in the inter granular spaces. A double pass FSP was carried out using a tool rotational speed of 1200 rpm, processing speed of 50 mm/min and axial force of 8 kN. A tool made of HCHCr steel; oil hardened to 62 HRC, having a hexagonal profile was used. The needle shape Al_3Zr particles were fragmented and converted into a spherical shape subsequent to FSP which resulted an increase in the hardness of the composite. Copyright © 2013 VBRI press.

Keywords: Metal matrix composite; casting; friction stir processing; intermetallic particle.

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Introduction

Aluminum matrix composites (AMCs) reinforced with various ceramic particles and intermetallic particles (Al_3Ti, Al_3Zr, Al_3Ni, Al_3Fe, etc.) have gained much attention in the last two decades because of their high elastic modulus and high specific strength both at ambient and elevated temperatures [**1–3**]. Al_3Zr is very attractive compared to other intermetallic particles due to higher melting point (1850 K) and relatively low density (4.12 g/cm^3) [**4**]. AMCs reinforced with intermetallic particles are fabricated using several methods including mechanical alloying, reactive sintering process, powder metallurgy and casting [**5–9**]. Casting method has the largest potential for industrial applications due to its simple operating process and the ability to obtain a large volume of material at low cost [**2,8**].

Some studies on the fabrication of AMCs reinforced with in situ formed intermetallic particles were reported in the literatures [**5, 8,10,11**]. Chianeh et al [**5**] prepared Al reinforced Al_3Ti AMC by the in situ reaction between Ti and Al during mechanical alloying and analyzed the formation mechanism of Al_3Ti particles. Zhao et al [**8**] fabricated Al reinforced Al_3Zr AMC by the in situ reaction of K_2ZrF_6 and molten aluminum. The Al_3Zr particles exhibited various morphologies such as spherical, tetragon, rod and fiber shape depending upon processing temperature. Tijun et al [**10**] developed Al reinforced Al_3Ti AMC by the in situ reaction between K_2TiF_6 and molten Al and observed uniformly distributed flaky shape Al_3Ti particles in the matrix. Roy et al [**11**] produced Al reinforced Al_3Ti AMC by the in situ reaction between Ti and Al during mechanical alloying and studied the tribological behavior of the AMC. The morphology, distribution and bonding features of intermetallic particles in the aluminum matrix play a crucial role to achieve higher mechanical and tribological properties.

Friction stir processing (FSP) was developed based on the principles of friction stir welding (FSW) to provide controlled microstructural modification in metallic materials [**12**]. FSP is fast emerging as a generic tool for material processing. FSP uses a rotating cylindrical tool having a shoulder and a pin which is pressed against the material to be processed and moved along the processing direction. The local heating due to friction and forging action of the tool deform and process the material at high temperature. Since the material flows at high temperatures, the process offers the possibility of redistributing the particles in AMCs.

Cavaliere [**13**] showed that FSP improved the properties of AA2618/Al_2O_3 AMC. Tewari et al [**14**] carried out FSP of Al/SiC AMCs and analyzed the effect of FSP on spatial homogeneity of SiC particles. He reported that FSP resulted in a significant microstructural modification which enhanced mechanical properties of the AMCs. Bauri et al [**15**] applied FSP to homogenize the TiC particle distribution in the Al/TiC in situ composite. The mechanical properties significantly improved after double pass FSP. Several investigators observed redistribution and fragmentation of ceramic particles in friction stir welded AMCs [**16–18**].

In this work, it has been shown that FSP can be used effectively to improve the homogeneity of the intermetallic particle distribution in Al/Al_3Zr in situ composites. The effect of FSP the microstructure and microhardness has been evaluated.

Experimental

Fabrication of the composite

AA6061-T6 rods (supplied by M/s Ultimate Enterprises, Chennai, India) were melted in an electrical resistance furnace (M/s Swamy Equipments, Chennai, India) using a graphite crucible. A coating was applied inside the crucible to prevent contamination. A measured quantity of inorganic salt K_2ZrF_6 (supplied by M/s Madras Fluorine Private Ltd., Chennai, India, 98.6% purity) of 179 g was introduced into the molten aluminum to form Al_3Zr. The amount of salt was calculated to produce a target weight percentage of Al_3Zr particles as eight in the composite. The temperature of the melt was maintained at 850^{0}C. The melt was stirred intermittently for 30 minutes. After removing the slag, the composite melt was poured into a preheated die to solidify. **Fig. 1a** shows the AA6061/8 wt.% Al_3Zr casting prepared at Coimbatore Institute of Technology, India.

Friction stir processing of the composite

Plates of size 100 mm X 50 mm X 8 mm were prepared from the casting to carry out FSP. The FSP was carried out using an indigenously built FSW machine (M/s RV Machine Tools, Coimbatore, India) at the center of the plate. The FSP parameters employed were tool rotational speed 1200 rpm, traverse speed 50 mm/min, axial force 8 KN and number of passes 2. A tool made of HCHCr steel; oil hardened to 62 HRC, having a hexagonal profile, shoulder diameter 19.2 mm, pin diameter 6 mm and pin length 5.8 mm was used. **Fig. 1b** shows the friction stir processed plate at Coimbatore Institute of Technology, India.

Fig. 1. Photograph of (a) AA6061/8 wt.% Al_3Zr casting and (b) friction stir processed plate.

Characterization of the composite

A specimen was obtained from the friction stir processed plate by cutting at the center of the plate perpendicular to FSP direction. The specimen was polished as per standard metallographic procedure and etched using Keller's reagent. The digital image of the macrostructure of the etched specimen was captured using a digital optical scanner. The microstructure was observed using an optical microscope (OLYMPUS-BX51M) and a scanning electron microscope (JEOL-JSM-6390). The microhardness of as cast and friction stir processed composite was measured using a microhardness tester (MITUTOYO-MVK-H1) at 500 g load applied for 15 seconds. The indentation was made at ten different locations and the average value was considered.

Results and discussion

Aluminum alloy AA6061 reinforced Al_3Zr intermetallic particulate composite was synthesized by the in situ reaction of K_2ZrF_6 salts to molten aluminum. The XRD pattern of the prepared AMC is shown in **Fig. 2**. The diffraction peaks of Al_3Zr intermetallic particles are clearly seen which give evidence for the formation of Al_3Zr particles. The in situ reaction between molten matrix alloy and inorganic salt at 850^0C as given in the following equation resulted in the formation of Al_3Zr particles. Absence of the peaks of any other compounds in **Fig. 2** indicates that the in situ formed Al_3Zr particles are in equilibrium with molten aluminum and thermodynamically stable. The interface between aluminum alloy and Al_3Zr particles tends to be free when no other compounds are present.

$$3K_2ZrF_6 + 13Al \rightarrow 3Al_3Zr + 4AlF_3 + 6KF \quad (1)$$

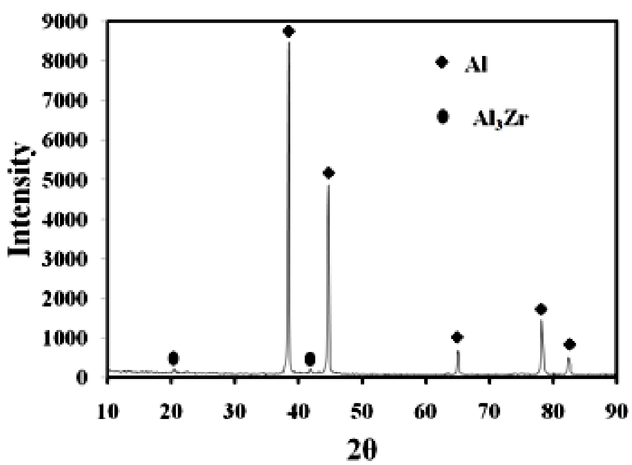

Fig. 2. XRD pattern of AA6061/8 wt.% Al_3Zr AMC.

The optical and scanning electron micrographs of the as cast AA6061/8 wt.% Al_3Zr AMC is shown in **Fig. 3**. A homogeneous distribution of Al_3Zr particles in the aluminum matrix is seen (**Fig. 3a**). Al_3Zr particles exhibit needle shape. The average size of the needle is 20 µm. The in situ formed Al_3Zr particles are well bonded to the matrix alloy. The in situ reaction between organic salt and molten matrix alloy is exothermic in nature which increases the local melt temperature which results in good bonding. Al_3Zr particles are not surrounded by any reaction products. The interface between Al_3Zr particles and matrix alloy is clear. The solidification phenomena of the composite melt influences the distribution of Al_3Zr particles in the matrix alloy. The molten matrix and Al_3Zr particle have a density difference of 1.5 g/cm^3 which causes the in situ formed Al_3Zr particles to suspend in the melt. The movement of the Al_3Zr particles is retarded due to the wetting effect between Al_3Zr particles and the melt which leads to the distribution of Al_3Zr particles in the melt for a long time. When solidification begins immediately after pouring the composite melt, α– aluminum crystallizes as the primary phase and concurrently Al_3Zr particles are pulled towards the solidification interface. It is evident from **Fig. 3b** that the in situ formed Al_3Zr particles are located in inter granular regions. The distribution of Al_3Zr particles in the melt is influenced by convection current in the melt, movement of the solidification front against particles and buoyant motion of particles [**15**]. The velocity of the solidification front influences the particle to be distributed in intra or inter granular regions. When the velocity of the solidification front is below a critical velocity, the particles are pushed by the solidification front leading to inter granular distribution and vice versa. The critical velocity is a function of the particle size and the temperature gradient. Observing the Al_3Zr particle distribution, it appears that the particles are pushed by the solidification front leading to inter granular regions. The fabricated AMC was subjected to friction stir processing to enhance the microstructure and mechanical properties.

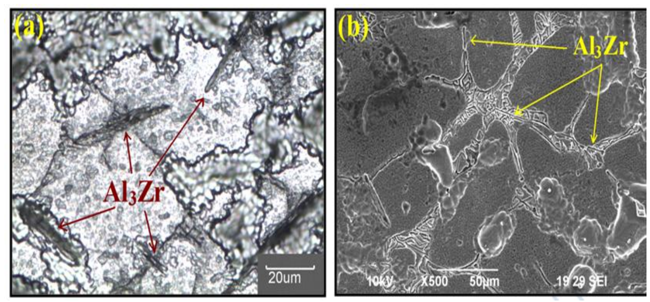

Fig. 3. (a) Optical and (b) SEM photomicrograph of as cast AA6061/8 wt.% Al_3Zr AMC.

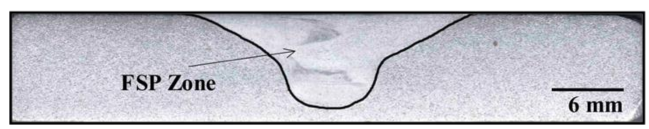

Fig. 4. Macrograph of friction stir processed AA6061/8 wt.% Al_3Zr AMC.

The macrostructure of friction stir processed AA6061/8 wt.% Al_3Zr AMC is shown in **Fig. 4**. The FSP zone is free from typical defects such as tunnel, pin hole, piping and worm hole. The process parameters employed in this work are adequate to generate sufficient frictional heat to plasticize the AA6061/Al_3Zr AMC and form a defect free FSP zone. The macrostructure consists of parent composite, heat affected zone, thermo mechanically affected zone and FSP zone.

The optical and scanning electron micrographs of friction stir processed AA6061/8 wt.% Al_3Zr AMC is shown in **Fig. 5**. The FSP zone displays a homogeneous distribution of Al_3Zr particles. The morphology and distribution of particles are significantly changed subsequent to double pass FSP. The needle shape Al_3Zr particles are fragmented due to severe plastic flow during FSP and the stirring action of the rotating tool. The morphology of Al_3Zr particles is changed into spherical shape which can be attributed to the abrading action of the tool [**16**]. The average size of Al_3Zr particles observed in the FSP zone is 500 nm. FSP induces high plastic strain which redistributes Al_3Zr particles. The effect of FSP on the microstructure can be very clearly observed from this figure. The grain boundaries in the as cast material (**Fig. 2**) are delineated by Al_3Zr particles due to their segregation at the grain boundaries. The FSP zone, on the other hand, exhibits no such grain boundary feature due to homogeneous distribution of particles throughout the matrix. Inter granular distribution of Al_3Zr particles is changed into intra granular distribution subsequent to FSP. The microstructure of the friction stir processed AA6061/8 wt.% Al_3Zr AMC also exhibits very fine grains due to continuous dynamic recrystallization.

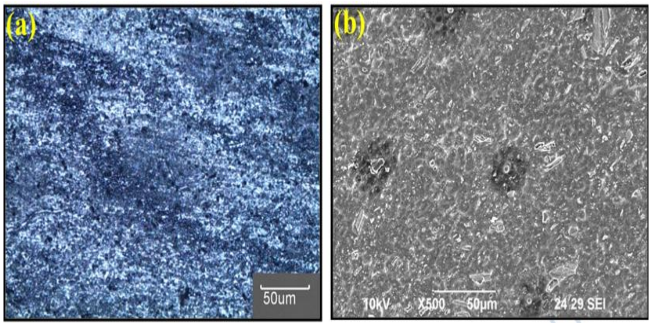

Fig. 5. (a) Optical and (b) SEM photomicrograph of friction stir processed AA6061/Al_3Zr AMC.

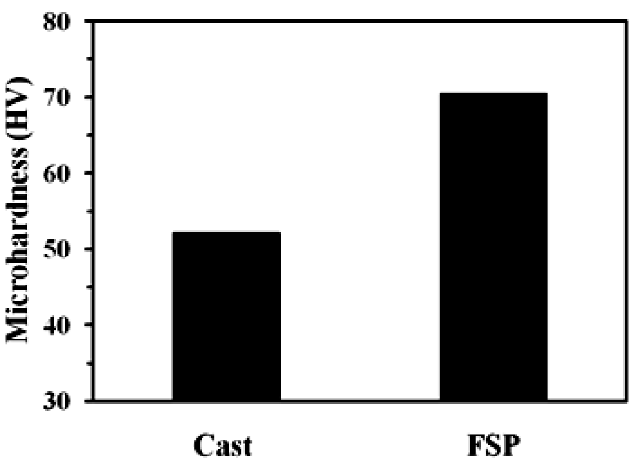

Fig. 6. Microhardness of as cast and friction stir processed AA6061/8 wt.% Al_3Zr AMC.

The microhardness of as cast and friction stir processed AA6061/8 wt.% Al_3Zr AMC is shown in **Fig. 6**. It is evident from the figure that FSP significantly improved the microhardness of the cast AMC. The average hardness of as cast and friction stir processed AMC are respectively 52 Hv and 70 Hv. The percentage improvement in hardness is 35. The improvement in microhardness after double pass FSP can be attributed to the increased dislocation density of the AMC as explained below. The grain boundaries are a source of dislocations in a polycrystalline material. The interface between the Al_3Zr particles and the matrix is also a major source of dislocation in AMCs due to mismatch in thermal expansion coefficient between the particles and the matrix. The differential deformation behavior of the Al_3Zr particles and the aluminum matrix during FSP also generates additional dislocations at the interfaces giving rise to a high dislocation density in the matrix. Since the Al_3Zr particles are homogenously distributed during FSP, more number of particle–matrix interfaces is created. The number of these interfaces is expected to be higher during the second FSP pass as any particle cluster which may remain after the first pass will also be broken down. As the grain size is refined during the first FSP pass, higher grain boundary area is available during the second pass. Therefore, the total number of dislocation sources available during the second pass is much greater.

Conclusion

AA6061/8wt. % Al_3Zr AMC was produced by the in situ reaction of molten aluminum and inorganic salt K_2ZrF_6. Al_3Zr particles having a needle shape were distributed uniformly in the aluminum matrix. Al_3Zr particles were located in the inter granular spaces. The friction stir processed AA6061/8wt. % Al_3Zr AMC exhibited a uniform distribution of spherical shape Al_3Zr particles. FSP converted the needle shape morphology of Al_3Zr particles into spherical shape which resulted in an increase in hardness of the composite.

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