Original Article

Effect of texture evolution on mechanical and damping properties of SiC/ZnAl$_2$O$_4$/Al composite through friction stir processing

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**ABSTRACT**

In this investigation, special consideration was given to observe the influence of the acquired ultra-fine-grain (UFG) structure through friction stir processing (FSP) on mechanical and damping properties. As the mechanical behaviour of the composites are intensely related to their microstructure. For deeply understanding the possible mechanism and detailed microstructural observations at a longitudinal cross-section of friction stir processed pure aluminium and several composites reinforced with bare SiC, Al$_2$O$_3$ coated SiC and ZnAl$_2$O$_4$ coated SiC were investigated through EBSD analysis. The mechanical as well as thermal cyclic (from $-100$ to $400\, ^\circ\text{C}$) damping performance of the friction stir processed composites were studied, respectively. Our first principles calculations show that the storage modulus of the resultant composite SiC/ZnAl$_2$O$_4$/Al was enhanced by a factor of $\sim 1.9$ after FSP as compare to parent Al. The ultimate tensile strength (UTS) of the friction stir processed SiC/ZnAl$_2$O$_4$/Al composite was enhanced by a factor of 3.3 and the acquired microhardness was almost doubled as compare to parent FS processed pure aluminium mainly because of significant grain refinement according to Hall–Petch relationship. Finally, improved properties, attributable to stabilization, enhanced distribution of the tailored SiC particles, improved load transferring capacity and better interface bonding between encapsulated reinforcements and matrix of FS processed composites were obtained with potential application in aerospace and automobile industries.

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1. **Introduction**

Reinforcement–matrix interface is recognized to govern the response of the materials and functionality of the composite. Among the numerous physical properties, grain structure of the materials has also played a major role in defining the behaviour of metal matrix composites. In observation of previous studies, it is found that metal matrix composites (MMCs) were thoroughly explored for its mechanical and physical characteristics. MMCs are extensively used in aerospace structures, aircraft, automobile, defense equipments, several components IC engine, electronic packaging, towers for
power transmission and multiple recreational products [1,2]. Generally, superior physical and mechanical properties are essential requisite of MMCs which are principally used in mechanically loaded regions to stabilize and stimulate the integrity of fractures, stiffness, strength, wear resistance, fatigue behaviour, corrosion resistance and creep resistance [3].

Microstructural and topographic surface characteristics are shown to play essential role in defining the behaviour of the metals as well as alloys. Crystal structure could also be deliberated as a crucial element influencing all characteristics of metal matrix. In general, arrangement of atoms or molecules defined by the crystal structure in the solid state [4]. The grain refinement is a broadly accepted technique of governing mechanical properties of the composites and alloys. Ultra-fine-grain structural elements can be acquired by severe plastic deformation (SPD) technique in which substantial deformations took place at high strain rates and quite low temperatures [5–8]. Initially, Valiev et al. described the definition of SPD technique for metal forming in which exceptional grain refinement occurred at high strain rate without trivial change in the overall dimensions of the bulk material [5,9]. The ultrafine grained solids are generally defined as homogeneously structured with less than 1 μm average crystal sizes [10].

However, many of the SPD techniques have their own limitations to the production of bulk material. Also, increasing strength without compromising the ductility of the material is a great challenge for the researchers. Being developed on the fundamental principles of friction stir welding (FSW), solid-state joining techniques, which date back to two decades ago at The Welding Institute (TWI), friction stir processing (FSP) were reported in 2000 by Mishra et al. [11,12]. Friction stir processing (FSP) is found one of the novel emerging metal working technique for significant grain refinement, homogeneity and densification of the processed zone [12–15].

It has been proved that FSP technique can deliver processing of the materials with cutting-edge multifunctional properties that make them constructive for various noble applications [16–20]. The grain refinement also affects the damping properties of the materials. Several scientists have improved damping capacity of aluminium alloy by adding nano-grain refiner without sacrificing mechanical properties [21–24]. Also, enhanced damping and mechanical properties were obtained by adding unmodified and modified reinforcements in aluminium matrix [25]. Moreover, there is no literature available on investigating the damping behaviour of the FS processed composite materials. Previously, we presented the results of an investigation on mechanical as well as damping behaviour of aluminium (Al) metal matrix composites (AMMCs) incorporated with bare SiC, Al₂O₃, SiC and ZnAl₂O₄, SiC core–shell micro-composites of submicron size synthesized via solgel route [26]. In this study, FSP technique was employed for grain refinement of these composites. Subsequently, the damping as well as mechanical properties of these composites were analyzed as a function of grain refinement after FSP. More exclusively, the effects of grain refinement of the composites on these properties have not been addressed previously.

2. Experimental

2.1. Friction stir processing

Detailed description on the fabrication of aluminium metal matrix composites (AMMCs) was provided in our previous work [26]. The as-cast composites were cut into the pieces according to the dimension of 150 mm × 50 mm × 6 mm. A non-consumable tool with specific geometry (smooth concave and threaded cylindrical) of die steel material (D2 steel) with shoulder diameter 18 mm, length of shoulder 25 mm, pin diameter 6 mm, length of pin 5.5 mm was prepared. Simultaneously, the prepared tool was heat treated and oil quenched. Subsequently friction stir processing has been performed with appropriate process parameter such as tool rotational speed 600 rpm, tool feed rate 20 mm/min, tool tilt angle 1°, axial force of 10 kN and no overlapping for the next pass. The schematic diagram of tool and work material arrangement in friction stir processing is shown in Fig. 1.

2.2. Characterization

Damping properties of the various as-cast composites and friction stir processed composites were conducted with dynamic mechanical analyzer “DMA” Q800 V20.26 Build 45, USA with 3 point bending clamp, frequency of 1 Hz and specimen’s size (40 mm × 10 mm × 1 mm). DMA data was collected from the lower temperature of −100 °C to the higher temperature of 400 °C. Liquid nitrogen was supplied for reaching at −100 °C. For each experiment, 3 °C/min heating rate and 5 °C/min cooling rate were kept constant. According to B557M-10 (ASTM standard), the tensile specimens were made and then tested on universal testing machine INSTRON, Model 5982 (USA). FE-SEM system equipped with electron backscatter diffraction (EBSD) characterization was utilized to investigate the microstructural evolution for various composites after FSP. Samples for EBSD analysis were prepared through polishing with different grade sand papers and cloth polishing with diamond paste (purchased from HMI Pvt. Ltd) followed by electro polishing (with a solution of perchloric acid (70%) and methanol (20:80) at −30 °C). Finally, orientation imaging microscopy (OIM) analyses of electro-polished surfaces were obtained on FEI QUANTA 3D FEG (FEI Netherlands). Microhardness was tested on the same samples used for microstructure analysis with MHVS-AUTO (Model) delivered from OMNI Technology, Pune (India).

3. Results and discussion

3.1. Effect of friction stir processing on microstructure evolution

Excellent mechanical characteristics promote the functionality of the friction stir processing of material and depend on the tool geometry and processing parameters. In this experiment, the amount of refinement is acquired in single step without overlapping. To make better understanding about the micromechanical behaviour of FS processed pure
aluminium and various composites, microstructural analysis were conducted by means of electron backscattered diffraction (EBSD) also known as orientation imaging microscopy (OIM) to explore the grain structure for all composites. Detailed description on microstructure of pure aluminium, bare SiC reinforced Al, Al$_2$O$_3$ coated SiC reinforced Al and ZnAl$_2$O$_4$ reinforced Al metal matrix composites (MMCs), conducted via optical microscopy were reported in our previous work [26]. Fig. 2 depicts typical microstructural electron backscattered diffraction (EBSD) analysis with pole figures and grain size distribution as a function of area fraction of as-cast pure Al, Al matrix reinforced with bare SiC, Al matrix reinforced with Al$_2$O$_3$ coated SiC and Al matrix reinforced with ZnAl$_2$O$_4$ coated SiC particles. For as-cast pure aluminium, average grain is 160 $\mu$m in size having large area fraction as revealed in Fig. 2(a). Average grain structure has been reduced from 160 $\mu$m to 90 $\mu$m by incorporating the pristine SiC particulates into aluminium matrix as shown in the plot between grain size and area fraction, Fig. 2(b). Similarly, significant grain refinement at room temperature were achieved by solidifying aluminium matrix reinforced with Al$_2$O$_3$ coated SiC and nanocrystalline ZnAl$_2$O$_4$ spinel coated SiC core–shell composites. Thus, an average grain size of 38 and 31 $\mu$m were obtained through EBSD microstructural analysis for Al$_2$O$_3$ coated SiC/Al and nanocrystalline ZnAl$_2$O$_4$ spinel coated SiC/Al, as shown in Fig. 2(c, d). Fig. 3 depicts typical microstructural (EBSD) analysis with pole figures and grain size distribution as a function of area fraction of FS processed pure Al, Al matrix reinforced with bare SiC, Al matrix reinforced with Al$_2$O$_3$ coated SiC and Al matrix reinforced with ZnAl$_2$O$_4$ coated SiC particles. In EBSD maps, the different grain orientations depicted though different colour contrast for pure Al and other composites. It is by and large accepted that the grain refinement is because of dynamic recrys-tallization. Therefore, the components impacting the nucleation and development of the dynamic recrystallization focused the resultant microstructural grain refinement in the SZ. Materials chemistry, geometry of the tool, vertical downward force, surrounding temperature conditions have significant impact on recrystallized grains [27]. For all the composites, average grain size is significantly reduced after FSP. FSP technique contributes in refining the microstructure of the composites to the ultrafine grain size and the measure of grain refinement is extremely critical since it is done in one stage. As shown in Fig. 1, diverse orientation of grains is observed in these four distinguishing regions (base metal (BM), heat affected zone (HAZ), thermo-mechanically influenced zone (TMAZ) and stir zone (SZ)) as a result these areas experience distinctive level of distortion of metal and temperature. During FSP, material flow pattern is different in advancing side as well as retreating side [28, 29]. The average grain size of the SZ portion is somewhat smaller than the average grain size of HAZ, TMAZ because of close vicinity of this region to transition zone. The contrastingly arranged grains saw in distinctive areas inside of the SZ demonstrate the vicinity of a conceivable strain angle [30, 31]. This can be ascribed to distinctive material flow pattern of the workpiece around the tool. Average grain size is reported as 25 $\mu$m for friction stir processed pure aluminium. Similarly, for the composites incorporated with unmodified SiC and modified SiC with Al$_2$O$_3$ and nanocrystalline ZAO spinel, an average grain of 15, 11 and 6 $\mu$m were obtained within the vicinity of SZ, respectively. Due to occurrence of dynamic recrystallization (DRX) of as-cast Al and other composites, uniform equiaxed grains revealed in SZ by the EBSD maps in Fig. 3(a–d). The [001], [110], [111] and [113] inverse pole figure (IPF) presented in Fig. 3 show the orientations developed at the stir zone. The designated maximum intensities are the times random grain orientation which are 5.313, 5.128, 5.973 and 6.708 for parent Al, SiC/Al, SiC/Al$_2$O$_3$/Al and SiC/ZAO/Al composites, respectively.
Fig. 2 – Typical EBSD microstructural images (IPF and grain boundary) and grain size distribution as a function of area fraction for various composites: (a) pristine Al, (b) bare SiC reinforced Al, (c) Al₂O₃ coated SiC reinforced Al and (d) ZAO encapsulated SiC reinforced Al.

Fig. 4 summarizes the influence of friction stir processing on grain refining of pristine Al and various composites incorporated with unmodified and modified ceramic particles through grain boundary maps with image quality map (IQM), orientation distribution function (ODF), misorientation angle histogram and inverse pole figure (IPF). From the IQM (Fig. 4(a–d)), different misorientation angle distribution of low angle grain boundary (LAGBs) (θ ≤ 15°) and high angle grain boundary (HAGBs) (θ ≥ 15°) are shown with different colours. LAGBs further divided into two categories, one from 2° to 5° represented as red colour and the other from 5° to 15° represented as green colour. Generally, LAGBs ranging from 2° to 5° is known as subgrain boundaries, formed by dislocation rearrangement. HAGBs are represented as blue colour. Basically, rearrangement of dislocations into sub-grain boundaries take place because of high stacking fault energy of aluminium.
Fig. 3 – Typical EBSD microstructural images (IPF and grain boundary) and grain size distribution as a function of area fraction for various composites after friction stir processing: (a) pristine Al, (b) bare SiC reinforced Al, (c) Al₂O₃ coated SiC reinforced Al and (d) ZAO encapsulated SiC reinforced Al.
Fig. 4 - Grain boundary maps with image quality map (IQM), orientation distribution function (ODF), misorientation angle histogram and inverse pole figure (IPF) for: (a) pristine Al, (b) SiC/Al, (c) Al₂O₃ grafted SiC/Al and (d) ZAO encapsulated SiC/Al.
dynamic recrystallization (DRX) during thermo-mechanical processing. Accordingly, an increased grain refinement was observed that is attributed to a higher degree of DRX. As noticed in Fig. 4(d), the fraction of LAGBs is higher as compared to pristine Al, uncoated SiC/Al and Al2O3 grafted SiC/Al composites. The increment in fraction of LAGBs is noticeable at the expense of HAGBs and for SiC/ZAO/Al composite significant change in LAGBs fraction occurred. Enhanced value of LAGBs is potential sign of a sub-grain structure getting strengthened. This is also confirmed by the orientation distribution function (ODF) selected at $\varphi = 45^\circ$ and inverse pole figure (IPF) as illustrated in Fig. 4(a–d).

### 3.2 Influence of texture evolution on damping properties

The damping property of the fabricated composites was studied using storage modulus and tan-s obtained from dynamic mechanical analysis (DMA) over an exceptionally wide range of temperature, previously [26]. Now, it is interesting to compare current findings obtained after friction stirring processing of the as-cast composites with observations reported in the literature. Fig. 5 illustrates the storage modulus ($E'$) and the internal friction ($\tan \delta = E''/E'$) upon heating and cooling as a function of temperature for Al, bare SiC incorporated Al, Al2O3 grafted SiC incorporated Al and ZAO grafted SiC incorporated Al matrix composites, respectively at a fixed loading frequency of 1 Hz. In this study, significant improvement in damping flexural characteristics of the FS processed pristine aluminium as well as other composites incorporated with unmodified and modified SiC particles is observed. Initially, maximum and minimum value of the storage modulus for pure Al is reported as 47 GPa at −100°C and 36 GPa at 400°C, respectively as revealed in Fig. 5(a). Additionally, the value of storage modulus decreases on increasing the temperature and this kind of behaviour is known as the relaxation phenomena. Further, storage modulus increases after reaching 350°C, it may be because of recrystallization of pure aluminium. As shown in Fig. 5(b), storage modulus further increases during cooling after reaching highest temperature of 400°C for the pure aluminium. However, damping and strengthening microscopic mechanisms are not independent for most of the metals. Materials that simultaneously exhibit excellent damping capacity and better mechanical properties are of great interest. Through, this task can be accomplished by developing two-phase or three phase materials where each phase plays a definite role [32]. Lattice defects such as dislocations, point defects, interfaces between reinforcements and matrix and grain boundaries are primarily responsible for defining the damping characteristics [33–35]. It is noticed in Fig. 4(c) that the SiC incorporated Al has enhanced enhanced damping capacity. At the temperature −100°C, storage modulus dramatically increases to 68 GPa for the sample incorporated with SiC particles in Al matrix. As a result, storage modulus of SiC/Al was increased by a factor of 1.4 as compared to storage modulus of pure Al. Again, storage modulus of SiC/Al increase during cooling and reaches to 86 GPa as demonstrated in Fig. 4(d).

Friction stir treated materials may govern the possibility of controlling the composite behaviour in contact with the microstructure and topography. Also, crystal structure can be considered as a key factor influencing damping characteristics of ZAO coated SiC incorporated composites. Subsequently, storage modulus of SiC/ZAO/Al composite is increased by a factor of ~1.9, ~1.3 and ~1.2 at −100°C along with at elevated temperature as compare to pristine Al, SiC/Al and Al2O3 coated SiC/Al composites. For enhanced damping behaviour of resultant composite is mainly attributable to dislocation motion between the interface of reinforcement and the Al matrix. Whereas, the influence of texture evolution through friction stir processing on the value of storage modulus for pristine Al, SiC/Al, Al2O3 coated SiC/Al and ZAO coated SiC/Al has been enhanced by a factor of ~1.06, 1.4, 1.3 and ~1.6, respectively as compare to all four samples without friction stir processing.

### 3.3 Mechanical properties

#### 3.3.1 Ultimate tensile strength (UTS)

The mechanical characteristic (axial tensile strength) of composite is defined as the quotient of its maximum load at failure to average cross-sectional area and likely dependent upon transferring of externally applied load from matrix to reinforcement. Fundamentally there are four major strengthening mechanisms functioning in strength enhancement: Orowan strengthening, fine grain size according to Hall–Petch rule, work hardening (because of strain misfit between the particles and the matrix) and strengthening due to difference in coefficient of thermal expansion (CTE) [36]. The overall strength of the composites may be stated as the following equation:

$$r_C = r_M + \Delta r_{\text{Hall–Petch}} + \Delta r_{\text{Load}} + \Delta r_{\text{Orowan}} \tag{1}$$

Where $r_C$ and $r_M$ are the strength of the composite and matrix respectively. Whereas, $\Delta r_{\text{Hall–Petch}}$, $\Delta r_{\text{Load}}$ and $\Delta r_{\text{Orowan}}$ are the increment due to refinement of grain size, effect of load transfer and effect of Orowan strengthening, correspondingly. Furthermore, the equation for individual strengthening mechanism could be expressed as:

$$\Delta r_{\text{Hall–Petch}} = k \left( \frac{1}{\sqrt{d}} - \frac{1}{\sqrt{d_0}} \right) \tag{2}$$

$$\Delta r_{\text{Load}} = \frac{V_F r_M (l + t) A}{4!} \tag{3}$$

$$\Delta r_{\text{Orowan}} = M G b \ln \left( \frac{a d_i 4 b}{2 \pi y \sqrt{1 - \nu}} \right) \tag{4}$$

where $k$, $d$ and $d_0$ represent Hall–Petch coefficient of aluminium matrix, average grain size of fabricated composite and average grain size of pure aluminium, respectively. The $A = (l/t)$, $l$ and $t$ are aspect ratio, thickness and size in parallel of load direction of the incorporated particles. While, $G$, $M$, $\theta$, $d_i$ and $y$ symbolize as shear modulus, Taylor factor, Possion’s ratio, uniform diameter of the particle and inter-particle spacing, respectively. Fig. 6 represents the yield strength (offset 0.2%) and ultimate tensile strength (UTS) after FSP of aluminium (without reinforcement), SiC/Al, Al2O3 grafted SiC/Al and ZAO grafted SiC/Al, respectively. At first glance,
enhancement of strength for individual surface composites is due significant grain size reduction according to Hall–Petch relation. As shown in Eq. (1) grain size directly affect the strength of the Al metal matrix composite. Though, formation of intermetallic compounds and inhomogeneity due to agglomeration of reinforcement are mainly responsible for reducing composites modulus elasticity and strength [37]. Unfortunately, the pure Al cannot wet the SiC properly through liquid route and the $Al_4C_3$ will be produced. Poor wettability and formation of $Al_4C_3$ compound is mainly responsible for the reduced strength of SiC/Al composite. Furthermore, the UTS of SiC/ZAO/Al composite significantly higher than the UTS of SiC/Al and SiC/$Al_2O_3$/Al composite might be due to improvement in incorporated particles distribution in the matrix and enhanced interface bonding between matrix and reinforcement. Agglomeration of SiC particles significantly diminish by encapsulating of SiC particles with nanocrystalline ZAO spinel also responsible for strength
enhancement and reduction of grain size. In this research, it is revealed that the yield strength and ultimate tensile strength of the friction stir processed parent Al enhanced to 46 (±4) MPa and 85 (±6) MPa, respectively. Similarly, the UTS for FS processed SiC/Al, SiC/Al\textsubscript{2}O\textsubscript{3}/Al and SiC/ZAO/Al composites are 237 (±8), 256 (±7) and 289 (±8) MPa, respectively. Furthermore, the UTS of SiC/ZAO/Al was improved by a factor of ~3.3, ~1.2 and ~1.1, respectively as compare to pure aluminium and SiC/Al and SiC/Al\textsubscript{2}O\textsubscript{3}/Al composites. While, the yield strength of pure Al and other composites have been improved after FSP. However, the role of Orowan strengthening and deference in coefficient of thermal expansion in strength enhancement are largely dependent upon volume fraction of reinforcements. In spite of the fact that the ultimate strength enhances due to uniform homogenization of the particulate in Al matrix additionally, then again because of significant grain refinement through friction stir processing this is further responsible of reduction in grain size and homogeneous distribution of incorporated ceramic particles.

**Micro hardness**

Fig. 7 illustrates the microhardness profile for the aluminium and different composites in friction stir processed region. The value of hardness for as-cast pure aluminium was found 28 (±4) as reported earlier by someone else [38]. It is interesting to note that microhardness of these fabricated composites is also enhanced after friction stir processing. As represented in Fig. 7, hardness for all the samples measured in SZ after friction stir processing was significantly improved as compared to base materials. The average hardness in SZ of the SiC/Al, SiC/Al\textsubscript{2}O\textsubscript{3}/Al and SiC/ZAO/Al composites are 68 (±5), 72 (±5) and 90 (±7), respectively as shown in Fig. 7(b–d). In addition, enhancement in hardness is strictly attributed to the resulting mean grain size and grain refinement of the composites according to Hall–Petch relationship. Significant improvement in hardness can be ascribed to homogeneity of reinforced particles dispersion after FSP which gives rise to more effective distribution hardening. Further, dislocation density and substantial grain refinement are also responsible for enhancement in the hardness.

**4. Conclusion**

Herein, friction stir processing was found one of the novel techniques for refining grain structure of the composites. The influence of FSP on microstructural, mechanical and damping properties of as-cast aluminium (without reinforcement),
SiC/Al, Al₂O₃ encapsulated SiC/Al and ZAO grafted SiC/Al composites were investigated and the major conclusions are as follows:

a. Microstructural EBSD analysis has revealed that there is significant amount of grain refinement in single pass. Further, homogeneous and ultrafine equiaxed grains are obtained with well-defined grain boundaries.

b. Storage modulus of the resultant composite SiC/ZAO/Al was enhanced by a factor of ~1.9, ~1.3 and ~1.2 at ~100°C along with elevated temperature as compare to pristine aluminium, SiC/Al and Al₂O₃ coated SiC/Al composites.

c. The results from the tensile analysis represented enhanced failure strains in the samples containing LZO coated SiC reinforcements Al as compare to pure Al, SiC reinforced Al and Al₂O₃ coated SiC reinforced Al due to grain refinement, further improvement in distribution of reinforcements in the Al matrix and improved bonding interface between the reinforcements and matrix. The UTS for FS processed SiC/Al, SiC/Al₂O₃/Al and SiC/ZAO/Al composites have been demonstrated as 237 (±8), 256 (±7) and 289 (±8) MPa, respectively.

d. Microhardness observations demonstrate gradually improvement in hardness of the composites incorporated by unmodified and modified SiC particles after FSP. The hardness of resultant composites is almost double to pure Al.

e. The improvement in storage modulus, damping capacity, UTS and hardness is attributed to Orowan strengthening mechanism, strengthening from difference in CTE between the particulates and matrix, Hall–Petch relationship and work hardening resulting from the strain misfit between the particulates and the matrix.

Conflicts of interest

The authors declare no conflicts of interest.

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