Energy-efficient method for developing in-situ Al-Cu metal matrix composites using microwave sintering and friction stir processing

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Abstract
The problems associated with the fabrication of in situ metal matrix composites (MMC) by conventional methods can be avoided by using microwave sintering and friction stir stirring in combination. The current study investigates the mechanical and electrical properties of pure aluminum (Al-100 wt%) and Al-Cu MMC. The results showed that excellent ultimate tensile strength, toughness, and electrical conductivity can be acquired simultaneously. The obtained ultimate tensile strength in the case of Al-100wt% (184.5 MPa) has improved two-fold than that of a typical commercially pure aluminum AA1016 (90 MPa). Similarly, the electrical conductivity of developed pure aluminum (88.87% IACS) is 1.4 times higher compared to AA1016 alloy (62% IACS). For Al-Cu MMC the copper is added in steps of 5 wt% (5%, 10%, 15%, and 20%). The maximum ultimate tensile strength (205.2 MPa) and the electrical conductivity (71.53% IACS) obtained for Al-10wt%Cu are higher compared to the AA1016 alloy. The present investigation suggests a novel processing route and opens up new research avenues in the field of solid-state materials processing.

Introduction

The transportation sector is in the groundbreaking phase to replace aluminum and its alloys with in situ MMC. Finer and more thermodynamically stable intermetallic phases make them preferential materials for various critical automotive components. The in situ MMC is being manufactured by liquid metallurgy (LM) and powder metallurgy (PM). Kumar et al. [1] successfully performed studies on the casting of Al/SiC-Cr hybrid MMC to replace the use of existing Al-Si cast alloys for high-strength wear-resistant applications. The in situ formed Cr$_2$C$_2$ hard intermetallic compound had a positive impact on increasing the wear resistance of the composite. Similarly, Sharma et al. [2] reinforced SiC and Muscovite into Al-Mg-Si-T6 alloy by stir casting. The addition of SiC enhanced the wear resistance and Muscovite reduced the wear loss. Naik et al. [3] developed Al/Gr-CNT hybrid composite via stir casting. The adhesion wear of pure aluminum was overcome by the hybrid composite and exhibited abrasion wear. LM processing routes are economical and most suitable for mass production. However, involves exposing the metal to high melting temperatures and faster solidification rates which adversely gives rise to residual stresses and causes metallurgical defects like hot tears, dendritic microstructure, and detrimental intermetallic phases [4].

PM has the flexibility to use any material as matrix/reinforcement with no restriction on composition and a higher percentage of reinforcement is possible [5.] PM parts generally undergo conventional sintering and involve radiant heating by conductive and convective heating. The volumetric heating phenomena of microwave sintering has grabbed global attention in recent decades [6]. Numerous studies have proved that
microwave power reduces the sintering temperature and energy consumption, enhances the atomic kinetic energy, accelerates the grain boundary diffusion, and increases the densification rate [7] Matli et al [8], fabricated Al-Cu composite by vacuum and microwave sintering techniques. The Al-Cu composition changed by the addition of copper in 3, 6, and 9 vol.%. The maximum hardness of 102 HV and ultimate compressive strength of 384 MPa were observed in microwave sintered Al-9vol.%Cu. The controlled heating help to accomplish desired intermetallic phases, uniform microstructure, and better mechanical properties [9]. All these inherent qualities make microwave sintering a powerful technique to substitute conventional sintering [10]. Further to attain homogeneous distribution of intermetallic phases, ultrafine grains, reduce porosity, and increase bulk density post-processing via severe plastic deformation (SPD) is necessary.

Among the SPD techniques friction stir processing (FSP) a variant of friction stir welding (FSW) has proved to be an eminent technique in the fabrication of in situ MMC [11]. A specially designed non-consumable tool having a featured pin profile is made to interact with the substrate. The larger hydrostatic pressure associated with the process is sufficient enough to generate heat, plasticize the material, and intermix the constituent particles. Dynamic recrystallization and/or recovery mechanism is responsible for grain refinement and microstructural homogenization [12, 13]. Malik et al [14, 15], conducted finite element simulations to understand the intricacies of FSW. Further, Malik and Kailas [16, 17] have addressed the importance of tool geometry in intermixing the constituent materials and minimizing the void formation in FSP. The tool with a shoulder to pin diameter ratio of 2.5 significantly enhanced the intensity of mixing. The intermixing nature along with other inherent characteristics features led to the development of MMC [18]. Surface/bulk MMC can be fabricated by using FSP either as a primary or secondary process [19]. Zykova et al [20], conducted a study to reveal the reason for the formation of intermetallic compounds during FSP. The solid-state processing gives rise to diffusion-controlled reactions without the formation of liquid interfaces and hence intermetallic phases are developed. Papantoniou et al [21], deployed a new method in manufacturing surface composite. A groove made in AA5083 alloy was filled with copper sheet and was friction stirred. The addition of copper significantly increased the hardness from 77 HV to 138 HV. Hence it was claimed to be surface hardening. In the other previous studies, researchers have adopted casting coupled with FSP [22], PM route with conventional sintering followed by FSP [23], and PM route with microwave sintering tailed with hot extrusion [24] for the fabrication of in situ Al-Cu MMC. Prabhu et al [25], reviewed the use of FSP as a primary and secondary process in the manufacture of in situ MMC. Table 1 summarizes the material, process, and evolved intermetallic phases in the manufacture of in situ MMC.

No work was reported on the fabrication of in situ Al-Cu MMC by PM route with unusual microwave sintering and post-processed by FSP. Hence the present investigation aims to achieve the same. The proposed novel processing route circumvents the drawbacks associated with LM, conventional sintering in fabricating in situ Al-Cu MMC. The followed process hierarchy is as follows: ball milling → compaction → microwave sintering → friction stir processing. The individuals are dealt with in subsequent sections. Tensile and microhardness tests were performed to analyze mechanical properties meanwhile resistivity tests to analyze the electrical properties of the materials. Further, SEM micrographs, SEM fractography, and EDS were performed to support the analysis.

| Matrix/Reinforcement | Primary process          | Secondary process          | Intermetallic phases | Reference |
|----------------------|--------------------------|---------------------------|----------------------|-----------|
| AA6061/K, ZrF4 + KBF4 | Casting                  | Friction Stir Welding      | ZrB2                 | [26]      |
| AA7075/K, TiF4 + KBF4 | Casting                  | Friction Stir Processing   | TiB2                 | [27]      |
| AA6061/Zr            | Casting                  | Friction Stir Processing   | Al2Zr                | [28]      |
| AA6061/Fe            | Casting                  | Friction Stir Processing   | Al2Fe                | [29]      |
| AA6061/Cu            | Casting                  | Friction Stir Processing   | Al2Cu                | [30]      |
| AA6061/Ni            | Casting                  | Friction Stir Processing   | Al2Cu                | [30]      |
| Al/Cu                | Conventional Sintering   | Friction Stir Processing   | Al2CuCu2O CuO        | [30]      |
| Al/Ti                | Conventional Sintering   | Friction Stir Processing   | Al2Ti                | [30]      |
| Al/Cu Al/Ti          | Conventional Sintering   | Friction Stir Processing   | Al2CuAl2Ti           | [30]      |
| Al/Si                | Conventional Sintering   | Friction Stir Processing   | —                    | [30]      |
| Al/Al-Cu-Li          | Microwave Sintering      | Hot Extrusion              | Al2Li Al2Cu Li       | [24]      |
Materials and methods

Aluminum and copper in powder form are used as matrix and reinforcement respectively and the details are highlighted in table 2. The chemical composition of aluminum powder is as follows: Fe-0.35%, Si-0.15%, and other metallic impurities-0.05%.

The particulars of charge preparation are tabulated in table 3. The charge was mixed in a bench-top roller grinding ball mill (Make: Enerzi Microwave Systems Pvt. Ltd) set up at a frequency of 25 Hz for 60 min with a ball to powder ratio (BPR) of 5:1. The container was made of stainless steel (SS) and alumina balls were used as grinding media with three different diameters weighing equally (6 mm, 10 mm, and 15 mm). The dry milling operation was conducted at room temperature without the addition of a binder and a non-inert atmosphere.

The homogenized charge was cold compacted to attain green strength at 35 MPa pressure for a dwell time of 20 s. The green parts were sintered in a hybrid heating setup at 510 °C for a cycle time of 25 min (ramp-up: 15 min and soak: 10 min) in a multi-mode microwave furnace (Make: Enerzi Microwave Systems Pvt. Ltd) having a maximum power showering capacity of 5.8 kW. Further, the sintered specimens were secondary processed via FSP on a milling machine (Make: Praga) with a specially designed tool (material: HCHCr) possessing a tapered cylindrical pin profile of 2.5 mm length and shoulder diameter of 14 mm. The other processing conditions were as follows: spindle rotational speed 1200 rpm, traverse speed of 10 mm/min, spindle head tilt of 1.5°, and two successive passes unidirectional. Figure 1 portrays the overview of the process involved in the fabrication of in situ Al-Cu MMC.

The tensile test was accomplished for sub-size specimens as per ASTM E-8 standard on an electronic screw-driven machine (Make: MCS Instruments and Engineers & Model: TNE-5) at a constant crosshead displacement resolution of 1 mm/min. Following the ASTM E-384 standard Micro Vickers hardness test (Make: Chennai Metco & Model: Economet VH-1MD) was conducted for the samples at the stirred zone with a test load of 25gf and 10 s dwell time after loading. In line with the four-wire measurements principle an electrical resistivity test was carried out using a contact resistance meter (Make: Motwane & Model: LR-2045). To confirm the powder morphology of received and ball-milled powder compositions Scanning electron microscope (SEM) images were acquired. Further, a fractographic study using SEM and elemental analysis using energy dispersive spectroscopy (EDS) was performed on fractured tensile surfaces.

Results and discussion

Ball milling

Figure 2 depicts the acquired SEM pictures of as-received aluminum and copper powders; and ball-milled powder compositions. The lower magnification (1 kx) background images are overlapped with higher magnification (3 kx) images at the top right corner of an individual as highlighted in the red color border. The particle size distribution range and morphology of as-received aluminum (figure 2(a)) and as-received copper (figure 2(b)) powders were confirmed. The adopted processing conditions in ball-milling such as frequency of 25 Hz, and BPR of 5:1 account for a low energy ball-milling, and milling time of 60 min help in efficient intermixing. Mellmann[34] has reported that the tumbling motion of the balls and charge inside the rotating container cause subtype motions such as slumping, rolling, and cascading. These subtype motions have different levels of intermixing and cascading type is most preferred for effective intermixing. Figures 2(c)–(f) represent SEM images of ball-milled powder compositions. Increased particle size is observed in all cases and can be
Figure 1. Process outline.

Figure 2. SEM images of as-received powders (a) aluminum, (b) copper; homogenized powder mixtures (c) 5wt%Cu, (d) 10wt%Cu, (e) 15wt%Cu, and (f) 20wt%Cu.
ascribed to cold welding of particles. The continuous tumbling action leads to the coalescence of softer aluminum particles with other aluminum and/or copper particles.

Microwave sintering
Initially to perceive the rate of heating the aluminum, copper, and SiC powders were tested in a microwave furnace. The loose powder weighing 30 grams was heated for 10 min with 300 watts of power. The rate of heating was as follows: Al: 21.7 °C min⁻¹, Cu: 9.9 °C min⁻¹, SiC: 16.7 °C min⁻¹, and the trend is illustrated in figure 3(a). Further, the rate of heating obtained for loose powders will reduce for compacted samples since they have partially turned into bulk materials. The green compacts absorb the microwave power till the particle’s coalescence takes place and switches to reflect as it becomes a single entity. To aid further absorbing capacity of the material hybrid heating setup is deployed. The green compact is covered with a SiC susceptor which is a good
microwave absorbing material. The susceptor is again surrounded by an Alumina board which acts as a thermal insulator. A small provision is made in the setup for a K-type thermocouple to sense the actual temperature. The microwave power is showered from four magnetrons placed diagonally so that uniform heating is achieved. The entire setup makes an effective heating chamber.

The recorded temperature of the specimen and showered microwave power during the heating cycle are represented in figure 3. From the temperature and microwave power profile, it is evident that a uniform rate of heating and power showering is maintained in the ramp-up cycle (figure 3(b) and (d)) whereas instabilities are seen in the soaking cycle (figures 3(c) and (e)). After reaching the target temperature at the end of the ramp-up cycle, the system turns off the supply of microwave power. A maximum drop in temperature values is observed for the Al-100wt% sample in the soaking cycle. To stabilize and maintain a uniform temperature an additional microwave power is supplied. Further, the addition of copper has decreased the drop in temperature in the soaking cycle. This can be attributed to: (a) irregular morphology of the aluminum powder gives more surface area and increase the plasticity of the particles resulting in better compaction (b) a wider range of particle size distribution occupies most of the voids and decrease porosity and (c) microwave sintering which helps in enhanced consolidation to make a bulk material. Thereby, microwave sintering increases the rate of heating, reduces the cycle time of heating, and saves energy compared to conventional sintering [8].

**Friction stir processing**

The unique characteristics like three-dimensional deformation and material flow during the process help to improve the distribution and morphology of intermetallic particles in the material. This desirably enhances the strength and extends the use of composite materials in load-bearing applications. Zeng et al [35] reported that the process dynamics result in a higher strain of 35 and a strain rate of 75 per second which is responsible for the formation of ultrafine grains and to some extent nanocrystalline structures. The grain evolution mechanism involved in the process starts from rolled grain → dislocation walls and tangles → sub-grains → dislocation multiplication → new grains. Severe plastic deformation and high heat input trigger the dislocation activities in the rolled grains to form dislocation walls and tangles. Further, to balance the total energy of the system dislocation walls and tangles get transformed into sub-grains. This is attributed to the dynamic recovery mechanism. The induced strain is responsible for the multiplication of dislocations. Due to continuous dynamic recrystallization subgrains rotate and form new grains. In addition, the grain evolution modes include sub-grain boundaries → sub-grain boundaries, and high angle grain boundaries → high angle grain boundaries. The sub-grain boundaries result in elongated grains, mixed-mode boundaries account for equiaxed grains and oriented grains are the outcome of high angle grain boundaries [35–38]. Dinaharan et al [22]. reported the formation Al2Cu intermetallic phase manufactured by stir casting. FSP was successful in the elimination of cast dendritic microstructure into a completely transformed new desired structure. The formation of Al2Cu played the role of grain refiner wherein the average grain size of 206 μm for 0-wt%Cu was reduced to 116 μm for 15-wt%Cu. It was further reduced to 16 μm and 4 μm respectively after FSP. In another study Al-Cu composite fabricated via conventional sintering also resulted in the development of Al2Cu intermetallic phase and were uniformly distributed using FSP [23]. Similarly, several studies have reported that the evolution of intermetallic phases significantly influenced enhancing the mechanical and electrical properties in the Al-Cu system [39–47].

Figure 4 represents the location of sliced testimonials out of the friction stir processed region for I-tensile, II-hardness, and III-electrical resistivity tests respectively. Further, the individual test results are discussed in subsequent sections.

![Figure 4. Extraction of test samples from the processed region; (I) tensile specimen with arrows showing actual specimen and schematic diagram of dimensions, (II) hardness specimen, and (III) electrical resistivity specimen.](image)
Strength and hardness of the stirred zone
Figure 5 depicts the engineering stress-strain curves. The mechanical properties like yield strength (YS), and ultimate tensile strength (UTS) are extracted from engineering stress-strain curves. Toughness is calculated using equation 1. These values along with hardness are plotted in figure 6. The YS, UTS, and hardness were found to increase. Here the possible formation of the Al$_2$Cu phase is accountable for the strengthening of the material [22]. Reduced grain size strengthens the material following the Hall-Patch mechanism [23]. The breakdown and homogeneous distribution of hard intermetallic particles act as load-bearing elements and interfaces are responsible for load sustainment. This affords Orowan strengthening. The strength is attained by intermetallic particles and dislocations form Orowan loops around non-deformable intermetallic particles and cause pinning action. The thermal mismatch between matrix and reinforcement generates strain fields that arrest the dislocation’s mobility. In addition, the strain fields include dislocations induced by deformation which in turn enhance the strength. Fine and ultra-fine grains bear the maximum tensile load before fracture. A better
interfacial bonding assists in the transfer of load to intermetallic particles [48–50]. A previous study reported that increased Cu content enhanced the UTS from 192 MPa (0wt%Cu) to 280 MPa (15wt%Cu) at the stirred zone. However, % elongation was found to be reduced from 12.3% (0wt%Cu) to 8% (15wt%Cu) [22]. A similar investigation reported a two-fold increase in hardness of the processed region (160 HV) compared to the as-sintered condition (80 HV) and was attributed to the homogeneous distribution of Al2Cu particles [23]. In line with these studies, the present investigation also reveals the increasing trend of UTS and hardness from 0wt%Cu to 10wt%Cu. On the contrary, toughness is sacrificed.

Figure 7 unveils the SEM micrographs of fractured surfaces of pure aluminum and composite materials. The images are represented with two magnifications 250× and 750× respectively. A typical feature seen in lower magnification is spotted and is shown with a higher magnification. No traces of dimples and relatively flat fracture surfaces indicate that the material has not undergone sufficient plastic flow before failure. This signifies a brittle mode of fracture. Jerby et al [51] described the concept of material solidification in microwave heating and is due to the localized melting of a cluster of powder particles. Along the same lines, in the present investigation microwave sintering resulted in localized melting both in pure aluminum and Al-Cu MMC. There is a high possibility that localized melt develops a liquid interface between the constituent particles to form Al-Cu intermetallic compounds. Further, FSP helped in breaking down the melt portions and distributed them uniformly and a few remnants are shown in figures 7(a), (b), and (d).

**Electrical conductivity of the stirred zone**

Copper is a well-known conductor possessing resistivity of 1.75E-08 Ωm, a conductivity of 5.8E + 07 S m−1. The international annealed copper standard (IACS) has standardized the electrical conductivity of commercially pure copper as 100% IACS. The % IACS directly signifies the conductive of the material [52]. The electrical properties such as resistivity, conductivity, and % IACS of pure aluminum and Al-Cu MMC are shown in figure 8. The pure aluminum exhibited an exceptional electrical conductivity of 88.87% IACS. This is nearly 1.4 times higher than that of the conductivity of AA1016 (62% IACS). It is evident in the temperature profile (figure 3(b)) that the Al-100wt% sample after reaching the target temperature starts dissipating the heat at a faster rate. Hence a drop in temperature is seen. This is a sign of higher thermal conductivity. Thereby, Al-100wt% pure aluminum material can replace the existing aluminum alloys in heat exchangers and heat sink applications. The increasing trend of electrical conductivity is seen up to 10wt% addition of copper beyond which decreased. Bowyer’s [53] study revealed that the presence of Cu2O considerably reduces the electrical conductivity of the material. This might be the reason for reduced electrical conductivity in Al-Cu MMC. Researchers have reported improved electrical properties parallel to mechanical properties in FSW of Al-Cu materials [52, 54].
Elemental analysis

The EDS point scan was performed on the SEM micrograph for Al-10wt% Cu (figures 9(a)–(c)) and Al-20wt% Cu (figures 9(d)–(f)). In the SEM microstructure, dark patches represent Al and bright patches represent Cu or Al\(_2\)Cu. For both the samples, EDS results were acquired at point 1 (matrix region) and point 2 (reinforcement region). The matrix and reinforcement region scans revealed the presence of Al and Cu peaks. This can be attributed to the homogeneous distribution of Cu particles in the Al matrix. In Al-Cu binary system, below 500 °C six equilibrium phases and a few metastable intermetallic phases are possible to form. Among these AlCu, Al\(_2\)Cu, and Al\(_4\)Cu\(_9\) are the stable phases. Since Al\(_2\)Cu has a lower free energy of formation and has an affinity to form at shorter heating exposure to a limited amount of copper. Besides, the controlled microwave heating
conditions can avoid the formation of the Al₄Cu₉ brittle intermetallic phase [55]. Hsu et al [23] reported the formation of Al₄Cu intermetallic phases upon conventional sintering of Al-Cu composite at two different temperatures of 500 °C and 530 °C followed by two FSP passes. In the present work, the nearly same sintering temperature is maintained (510 °C) with two FSP passes. Hence the authors infer Al₄Cu intermetallic phases might have formed during microwave sintering and are uniformly distributed after FSP.

Conclusions

The feasibility of energy-efficient techniques like Microwave Sintering and FSP in combination was explored to develop in situ Al metal matrix composites. The experiments yielded the following noteworthy observations:

1. Ball milling with low impact energy settings assisted in blending of the initial powder mix, compaction helped in acquiring green strength, microwave sintering aided in the consolidation of the material, and finally, friction stir processing supported grain refinement.

2. A superior combination of the mechanical and electrical properties was acquired in Al-100 wt% aluminum samples with UTS of 184.5 MPa and IACS of 88.87% which is significantly higher than commercially pure aluminum alloys.

3. The addition of copper enhanced the mechanical and electrical properties of the MMC. The increasing trend was up to 10wt%Cu and beyond which it decreased.

The proposed processing route offers a potential technique to manufacture Al-Cu MMCs possessing a combination of properties, which are contradictory and are difficult to acquire by regular techniques.

Acknowledgments

The authors acknowledge KLS Gogte Institute of Technology for setting up the Center of Excellence for Industrial Microwave Application Development in association with an industry partner Enerzi Microwave Systems Pvt. Ltd and for supporting this research by financial aid.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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