Original Article

A comparative study of structural and mechanical properties of Al–Cu composites prepared by vacuum and microwave sintering techniques

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ABSTRACT

In this paper, the aluminum metal matrix composite reinforced with copper particulates (3, 6 and 9 vol.%) were fabricated by high energy ball milling, followed by vacuum sintering (VS) and microwave sintering techniques (MS) separately. The effects of Cu content and preparation methods on the microstructure and compression mechanical behavior of Al–Cu matrix composites were investigated. The microstructural characterizations revealed a homogeneous distribution of Cu particles in the Al matrix and also fine microstructures of microwave sintered samples. The microwave sintered specimen exhibited the highest hardness and better mechanical properties compared to vacuum sintered specimens. Furthermore, the hardness and compressive strength increased 137.2% and 30.3% for the microwave sintered Al–9 vol.% Cu composite, respectively. The increase in mechanical properties with the increasing volume fraction of Cu particulates can be ascribed to the presence of harder Cu particles reinforcement. The developed materials of the microwave sintered Al–Cu composite in this investigation could be successfully used for industrial applications due to improved mechanical properties.

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1. Introduction

Metal matrix composites (MMCs) have the distinctiveness that is superior to those of the conventional monolithic metallic alloys, mandatory for aerospace and automotive industries [1,2]. Metal matrix composites have a variety of applications in aerospace, defense and automotive industries due to their superior mechanical properties. Among various MMCs, aluminum metal matrix composites (AMMCs) gains the most attention in the automobile and industry products due to their enhanced properties like low density, high elastic modulus, tensile strength, excellent mechanical strength, good wear resistance and high toughness [3–7].

One of the important keys in manufacturing AMMCs is uniform distribution of reinforcements in aluminum matrix (Al). Many methods have been engaged to produce aluminum based composites like casting routs, extrusion, and
2. Experimental procedures

Al (99.5% purity, 10 μm average particle size) and Cu particles (99.5% purity, 15 μm average particle size) were used as starting materials. Pure Al and Cu (3, 6 and 9 vol%) particles were weighed carefully and blended for 1 hr using high-energy ball mill with the rotation speed of 200 rotations per minute (rpm). No balls were used in this stage. The blended powders (~2.0 g) were then compacted into cylindrical pellets by applying uniaxial pressure of 30 MPa. The green pellets were then subjected to two different sintering techniques namely, vacuum sintering carried out at 550 °C for 2 hr, and microwave sintering at 550 °C for 30 min. Microwave sintering (MS) was carried out in a microwave furnace with a silicon carbide ceramic crucible and alumina insulation in it (VB Ceramic Consultants, Chennai, India). The green samples were placed in the center of the cavity and sintered in a microwave furnace (multimode cavity) at 2.45 GHz. SiC was selected as a microwave susceptor to assist heating and sintering of the green samples. The sintering temperature was set at 550 °C ± 5 °C with a holding time of 30 min and an approximately heating rate of 25 °C/min. Fig. 1 shows the schematic of vacuum and microwave sintering setup and sintering profile.

The phase identification of the amorphous powders and prepared Al-composites were examined using panalytical X’pert Pro diffractometer with CuKα radiation (λ = 0.15406 nm). The XRD patterns were recorded in the 2θ range of 30–90°.
Table 1 – Density and porosity measurements of vacuum and microwave sintered Al–Cu composites.

<table>
<thead>
<tr>
<th>Composition</th>
<th>Theoretical density (g/cc)</th>
<th>Vacuum sintering</th>
<th>Microwave sintering</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Experimental density (g/cc)</td>
<td>Porosity (%)</td>
</tr>
<tr>
<td>Pure Al</td>
<td>2.70</td>
<td>2.688</td>
<td>0.4</td>
</tr>
<tr>
<td>Al–3 vol.% Cu</td>
<td>2.886</td>
<td>2.725</td>
<td>5.5</td>
</tr>
<tr>
<td>Al–6 vol.% Cu</td>
<td>3.073</td>
<td>2.874</td>
<td>6.8</td>
</tr>
<tr>
<td>Al–9 vol.% Cu</td>
<td>3.253</td>
<td>3.016</td>
<td>7.5</td>
</tr>
</tbody>
</table>

with step size of 0.02° and a scanning rate of 1.5°/min. The morphology of the sintered composites was observed using scanning electron microscopy (SEM, JeolNeoscope JSM 6000) equipped with an energy dispersive X-ray spectroscopy (EDX). The microhardness values of the samples were measured using a Vicker’s microhardness tester (MKV-h21, with applied load of 100 gf for 15 s), with an average of at least five successive indentations for each sample. Universal testing machine-Lloyd 50KN at a strain rate $10^{-4}$/s was used to measure the compressive properties of the cylindrical samples with a diameter of 10 mm and a height of 3 mm.

3. Results and discussion

The results of density and porosity measurements of pure aluminum and its composite materials synthesized at different sintering techniques are summarized in Table 1. The reference density of the Al–Cu composites is theoretically calculated using the rule of mixtures. A marginal increase in the experimental density value of pure Al was observed and microwave sintered Al–9 vol.% Cu composite exhibited a maximum of $\sim 3.241$ g/cm$^3$, which is greater than vacuum sintered samples. Further, the porosity value of the synthesized Al materials was found to increase with the addition of Cu particulates and Al–9 vol.% Cu composite exhibited a maximum porosity of $\sim 0.32\%$. The presence of porosity reduced the densification in sintered aluminum composites. Microwave heating rate also plays a role, in which the high heating rates will contribute to a low porosity [16].

Fig. 2(a and b) shows the XRD patterns and the phases present in the vacuum and microwave sintered Al–Cu composites. From the XRD phase analysis shows that the characteristic elemental peaks of Al and Cu are present in all samples. The intensity of the Cu diffraction peak increases with the increasing Cu content in both the vacuum and microwave sintered composites. Studies have shown that the addition of Cu to Al, a formation of AlCu and Al$_2$Cu phases is expected according to the binary Al–Cu phase diagram. However, the relatively short duration microwave sintering technique employed in this study allowed the retention of alloy structure of the reinforcement powder and thereby, prevents the formation of any intermetallic phase [17].

As the Cu content in the composites increases, the microwave sintered samples show less increase in the peak intensity as well as in sharpness than that of the specimens sintered by vacuum sintering. That is because the grain coarsening of the microwave sintered Al–Cu composites is lower than the vacuum sintered composite. As microwave sintering requires less sintering temperature and less soaking period than the conventional techniques. It is possible to produce bulk size nanocomposites without much grain coarsening [18].

Fig. 3 shows the SEM micrographs of the polished surface of both the microwave and vacuum sintered Al–Cu composites. Fairly uniform distribution of the reinforced Cu particles on the Al matrix can be observed with some clustering in some areas. Uniform distribution of the second phase particles is required to obtain proper mechanical properties. The dark region in the microscopic images contains mainly Al matrix and white region shows the Cu particles. The images also demonstrate proper interfacial bonding without any cracking between the Al matrix and Cu reinforcements similar to that observed by other researchers working on aluminum and magnesium based composites reinforced with nanoparticles [19–21]. However, vacuum sintered samples showed small pores in the microstructure, it seems that the slow heating
rate and long soaking times can cause porosity formation. In microwave sintered samples porosity is much reduced, due to fast heating and cooling rate. This fact results in a favorable sintering behavior with less grain growth, more densification and suppressed powder decomposition.

Fig. 4(a–d) shows the EDS spectra and quantitative analysis of elements present in Al–9 vol.% Cu composites sintered by vacuum and microwave sintering techniques. Elemental mapping was conducted to identify the distribution of elements in the observed composites. Mapping confirmed the uniform distribution of Al and Cu components over the field of analysis of the image. Note that no other peaks were found in the final product.

The microhardness of the fabricated composites was evaluated on Vickers microhardness tester (MKV-h21) for a duration of 15 s at an applied load of 100 gf. Fig. 5 shows the microhardness of the vacuum and microwave sintered Al–Cu composites. The hardness values for both the microwave and
vacuum sintered composites increased with the increasing of Cu content in the Al matrix. This can be attributed to the: (i) presence of harder copper reinforcement (ii) higher constraint to the localized matrix deformation during indentation as a result of the presence of reinforcement and (iii) reduced porosity with the increasing Cu content [22].

This increase trend was observed in vacuum and microwave sintered composites from 40 ± 3 to 65 ± 2 Hv and 43 ± 3 to 102 ± 3 for Al matrix materials to 9 vol.% of Cu reinforced composite, respectively. Comparatively higher hardness values were obtained for the microwave sintered samples as compared to the vacuum sintered. The lower hardness of the vacuum sintered composites is possibly due to the grain coarsening and longer soaking period. As microwave sintered samples contain less amount of voids and less grain growth occurred than conventional sintered specimens, the hardness values are expected to be higher than that of the composites sintered by other sintering methods.

Figs. 6(a) and 7(a) show the compressive stress–strain curves of the Al–Cu matrix composites with different Cu contents and sintered through vacuum and microwave

Fig. 5 – Microhardness of the vacuum and microwave sintered Al–Cu composites.

Fig. 6 – (a) Room temperature compression stress–strain curves for the vacuum sintered Al–Cu composites and (b) corresponding data of mechanical properties.

Fig. 7 – (a) Room temperature compression stress–strain curves for the microwave sintered Al–Cu composites and (b) corresponding data of mechanical properties.
sintering techniques, respectively. Figs. 6(b) and 7(b) represent both yield strength and ultimate compressive strength of the Al–Cu matrix composites. It can be seen that, yield strength and compressive strength are increasing with the increasing Cu content in Al matrix. The yield strength and compressive strength of microwave sintered composites are much higher than those vacuum sintered composites. These superior compressive properties are ascribed to the effect of three possible important strengthening mechanisms [23]: (i) the Orowan strengthening mechanism related to uniform distribution of Cu particles, (ii) enhanced dislocation density as a result of the large mismatch between the thermal expansion coefficient of copper particles and the aluminum matrix, and finally (iii) the dispersion hardening effect of the hard Cu particles into the soft aluminum matrix.

Fig. 8 – The fractured surface morphology of the (a–d) vacuum sintered and (e–h) microwave sintered Al–Cu composites.
Several theories and mechanisms have been recommended to explain the strengthening of MMCs. However, the strength of the composites does not depend on a unique mechanism but several mechanisms may act simultaneously. In the present study, strengthening effect is mainly due to the dispersion hardening effect of the hard Cu particles into the soft aluminum matrix [24]. The strength induced by the dispersion of the Cu particles in the aluminum matrix can be explained on the basis of Orowan strengthening mechanism. Actually, residual dislocation loops are formed around every Cu particle due to bending of dislocation passing by the hard Cu particles. This loop formation leads to high work hardening rates and helps to strengthen the material. The contribution to yield strength can be expressed as [25,26]:

\[ \sigma_{\text{Orowan}} = \frac{2Gb}{\lambda} \]

where \( G \) and \( b \) are the shear modulus of the matrix and Burger’s vector of the dislocation, and \( \lambda \) is the distance of dispersed particles.

In addition, effective load transfer from the relatively softer matrix to the harder reinforcement particles depends on the interfacial bonding between Al matrix and Cu reinforcement. SEM micrographs (see Fig. 3) indicate good interfacial bonding between Cu particles and the Al matrix. The microwave sintering might have helped to improve bonding of the Al matrix with Cu particles.

The surface morphologies of the fractures of the vacuum and microwave sintered Al–Cu composites after compressive loading are shown in Fig. 8. It is observed that failure in pure Al and Al–Cu composites occurred at 45° with respect to the compression loading axis and their representative fractographs indicate the presence of shear bands. The presence of a shear band in all the samples indicated that the mode of deformation of the matrix remained ductile irrespective of the presence of Cu particles and the difference in the sintering behavior. There was no observable difference in the fracture surface in the case of Al samples sintered at different techniques.

4. Conclusions

Al–Cu composites were successfully synthesized by using vacuum and microwave sintering techniques and their structural and compressive mechanical properties were studied. XRD analysis and SEM images confirm the presence of Cu reinforcement particles in the fabricated Al metal matrix composites. It is observed that microwave sintering leads to a more compact structure as compared to the vacuum sintering and thus more effective in consolidation of ball milled Al–Cu composite powders as compared to the vacuum sintering. The significant improvement in mechanical properties can be attributed to the dispersion hardening effect of hard Cu particles present in the soft Al matrix. It is further noticed that the mechanical properties of Al–Cu composite increases with the increasing amount of Cu. The microwave sintered Al–9 vol.% Cu composite exhibited the highest microhardness (102 ± 3 Hv), yield strength (136 ± 4 MPa) and compressive strength (384 ± 5 MPa) as compared to the vacuum sintered composite.

Conflicts of interest

The authors declare no conflicts of interest.

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