Research Article

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Synthesis and mechanical characterization of Si_3N_4 reinforced copper-tin matrix composites

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Abstract: The present research reports improvement in mechanical performance of Copper-Tin (Cu-Sn) composites due to the addition of hard Si_3N_4 particles as reinforcement. Two-step stir casting technique was adopted to prepare Si_3N_4 -reinforced Cu-Sn based composites. The uniform distribution of the reinforcement in the matrix was ensured by SEM morphology and energy-dispersive X-ray (EDAX) spectrum of the developed composites. Brinell hardness test and tension test were used to estimate the hardness and tensile strength of the composite. Si_3N_4 reinforcing materials added to the extent of 7.5% by wt. to Cu-Sn matrix enhanced hardness by 41% and UTS by 33%, YS by 29% and showed 23% decrement in ductility. Thus a comparison of Si_3N_4 -reinforced Cu-Sn based composites with base metal has shown that considerable improvement in tensile properties such as UTS and YS and hardness if reinforcing material is added to the extent of 7.5%.

Keywords: Cu-Sn alloy, stir casting, mechanical properties, microstructure, Si_3N_4

1 Introduction

Copper has exceptionally useful properties such as good electrical and thermal conductivity, corrosion resistance and can be readily alloyed. These properties make it an

attractive candidate for matrix material in thermal and electronic applications [1]. Conversely, its poor mechanical performance inhibits its usage in strength requirement applications. However, this drawback can be compensated by reinforcing it with the hard and fine ceramic materials such as carbides/borides/oxides/nitrides. These inclusions enhance hardness and strength at the expense of a relatively little loss in electrical conductivity [2]. Numerous evidences are found in literature which showed the improvement of the mechanical strength of copper matrix by the addition of reinforcements of TiC, Cr_2O_3 , Al_2O_3 , TiO_2 , B_4C , carbon nano tube, SiC, graphite, carbon fibre, rice husk ash etc. [3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13]. Recently addition of another ceramic silicon nitride (Si_3N_4) has gained interest in research due to its excellent wear, corrosion resistance, high-temperature mechanical properties, exceptional thermal shock resistance owing to its low coefficient of thermal expansion and high thermal conductivity. The mechanical and chemical stability at elevated temperatures made Si_3N_4 to be attractive and promising candidate for high temperature engineering applications. Very few studies are found in literature on investigations connected to influence of Si_3N_4 on copper matrix. These countable investigations majorly deal with the effect of Si_3N_4 on the electrical and thermal conductivity [14] of composites and mechanical characterization and wear behavior of composites. Robin *et al.* [15] have developed soft copper coatings with well-distributed Si_3N_4 particles on carbon steel by novel coelectro-deposition method and reported improved microhardness and strength compared to pure copper coating and no difference in corrosion resistance. Solderability properties of Si_3N_4 reinforced in Sn-0.7Cu solder was systematically investigated by MAA *et al.* [16]. They showed that an improvement in solderability was achieved with optimum wettability when 1.0 wt.% Si_3N_4 was reinforced in Sn-0.7Cu solder. Further, MAA *et al.* [17] studied the influences of the Si_3N_4 particulates on the mechanical properties of the same composite solder and reported improved microhardness value and lap-shear strength when higher reinforcements of Si_3N_4 particulates were present in

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the monolithic matrix solder. Using Modulus of elasticity-porosity relationships, empirical equations were proposed for indicating the porosity dependence of the strength of Si_3N_4 ceramics [18]. Effect of reinforcement content on the hardness, strength [19] and adhesive wear behaviour of $\text{Cu}_{10}\text{Sn}_{5}\text{Ni}/\text{Si}_3\text{N}_4$ composites were studied and results reported the improved strength and toughness when compared to pure Cu [20, 21].

Copper alloys are majorly employed in machine parts subjected to high-friction such as bearings. Copper–tin (Cu–Sn) alloys used in bearings need solid lubricants such as grease which have minor effects on the environment compared to Copper–lead alloys which release harmful gases at high temperatures when continuously used [22]. The desirable properties of bearings are high thermal conductivity to dissipate heat generated due to friction, high corrosion resistance, high fatigue, tensile, shear, compressive strength and wear resistance and be able to bear shocks and vibrations. Higher tensile and compressive strengths increase the ability of the bearing material, to resist formation and propagation of cracks under high contact pressures and to avoid extrusion or other permanent deformation of the bearing [23]. Hence in this study, an attempt is made to evaluate the influence of addition of Si_3N_4 as reinforcement to Cu–Sn matrix Composites on mechanical properties of the composite.

2 Experimental procedures

2.1 Materials used

The Cu–Sn alloys possess superior properties and hence are employed for various mechanical and electronics industrial applications. Cu-rich Cu–Sn alloys are employed as structural materials and Sn-rich alloys as lead-free solder materials. The latter application avoids toxicity of lead and hence has attracted researchers due to health and environmental concerns. [24, 25].

Commercially available Cu–Sn alloy was considered as the matrix for composite preparation. Table 1 presents its chemical composition. It is stronger and more ductile than red brasses and has high wear resistance. Despite having low solubility in Cu at room temperature the tin is a solid solution strengthening in Cu [26].

In present investigation, to enhance the mechanical performance of Cu–Sn alloy, Si_3N_4 particles of size $40\text{ }\mu\text{m}$ were used as reinforcement material. Liquid stir casting technique was adopted to develop Cu–Sn/ Si_3N_4 composites. Figures 1(a) and 2(a) show the SEM and EDAX pattern of Cu–

Sn base metal (A sample) and Figures 1(b) and 2(b) depict the SEM and EDAX pattern of Si_3N_4 particulates used to fabricate the composites. The intensity peaks of Cu and Sn are mainly observed in the EDAX spectrum of the A sample as they are the major elements present along with negligible amounts of Al, Si and O.

Table 1: Chemical composition of Cu–Sn alloy in weight percentage.

Element	Al	Si	Sn	O	Cu
Wt. %	0.47	0.06	7.85	0.2	Remaining

2.2 Fabrication procedure

The morphology and mechanical properties are established at the production stage of composite and hence the materials used and the preparation method have to be carefully planned and executed. The cost of production is also dependent on it. Though a number of difficulties are faced due to the formation of porosity and poor wettability between reinforcement phase and matrix, when compared to other MMC preparation routes, liquid state processing method is eye-catching one because of low cost and its simplicity. Under liquid state processing method, stir casting technique is more established and promising one, as it ensures the proper dispersion of reinforcements with in matrix due to mechanical stirring action [27].

Table 2: Composition of prepared samples.

Sample	Cu–Sn alloy	Si_3N_4 particles
Base alloy (A)	100%	0%
Composite (ASiN)	92.5%	7.5%

In this work, one of the variants of liquid stir casting called two step stir casting method is adopted for sample preparation. The set up used for casting is shown in Figure 3(a) and 3(b). Initially required amount of charge or matrix material was placed in a graphite crucible, and was placed in electric muffle furnace. It was then heated to a temperature of around 1150°C above the liquidus temperature of the matrix to melt completely. After complete melting of a Cu–Sn matrix, degassing was carried out by using solid hexachloro-ethane (C_2Cl_6) degassing tablet. It removed unwanted adsorbed gases from the melt. Once degassing got over, the preheated (to 500°C) ceramic reinforcement Si_3N_4

Figure 1: SEM of (a) Base metal 'A' and (b) Si_3N_4 particulates.

Figure 2: EDAX of (a) Base metal 'A' and (b) Si_3N_4 particulates.

particles were introduced into the matrix and mixed manually. Two-stage addition of reinforcement during stirring was adopted. This novel two stage additions of reinforcement into Cu-Sn matrix helped in increasing the wettability of the matrix and ceramic reinforcement, and ensured uniform distribution of the particles in matrix. Mechanical stirring was carried out for about 15 min at an average mixing speed of 250 rpm. The melted material was poured finally to a cast iron die with the dimensions of 120 mm length and 15mm diameter preheated to a temperature of 200°C and there it was allowed to cool to get the required casting. Table 2 gives the composition of the composite prepared.

Figure 3: (a) Schematic diagram of Stir casting set up and (b) Photo of stir casting set up.

2.3 Characterization procedure

2.3.1 Microstructural characterization

Microstructural characterization of the prepared composites was done using Scanning Electron Microscopy and EDAX analysis. The microstructure of the cast A and ASiN samples were obtained by scanning electron microscope (TESCAN VEGA, Czech Republic). The samples of cast A and ASiN composite for microstructural study were cut from cast rods. The surface of the specimen was first made flat by rough grinding using Abrasive paper of grit size 200-320 and subsequently with grit sizes of (600-1200) to replace coarse lines with finer ones. The final layer is removed chemically by etching with Keller's reagent. It was then examined under scanning electron microscope.

2.3.2 Mechanical characterisation

To investigate the mechanical performance of the composites the tensile and hardness tests were carried out. Tensile testing was conducted on round test specimens of diameter 9mm and gauge length of 36mm prepared from cast A and ASiN composite using a computerized universal testing machine of 400 kN capacity at ambient temperature.

Yield strength, ultimate tensile strength and elongation percentage were calculated based on three samples and result was obtained based on average value. Figure 4(a) and 4(b) depict the test coupons used for the tensile test according to the ASTM E8-18 standard [28]. To evaluate the influence of Si_3N_4 particulates in the matrix material hardness test was performed on cast A and ASiN composite. The polished specimens were tested for hardness, using Brinell hardness testing machine having ball indenter of 5mm diameter applied for 250kgf load and dwell time of 30sec as per the standard of ASTM 10-18 [29]. Nine sets of readings were taken at different places of the specimen and an average value was used for calculation.

Figure 4: (a) Dimensions of tensile test specimen in mm as per ASTM 10-18 and (b). Tensile test specimens as per ASTM 10-18.

3 Results and discussion

3.1 Microstructural characterization

EDAX characterization is one of the known methods to confirm the elemental presence in the developed composite. Figures 5a, 5b show the SEM and EDAX spectrum of the ASiN composite respectively. The EDAX spectrum of the developed composite ASiN has the intensity peaks of Cu, Sn of matrix and Si, N of reinforcing materials.

As seen in Figure 5a, the surface of the composites obtained after polishing and chemical etching has SiN rein-

forcements homogeneously dispersed in the Cu-Sn matrix without any noticeable large agglomerations. Also the sample matrix is continuous and no clear pores or cracks are visible at the interface of matrix and reinforcement particles on the surfaces of the composite. This is attributed to the two stage addition of SiN reinforcement into Cu-Sn matrix. This novel method helped in increasing the wettability of the matrix and ceramic reinforcement, and ensured uniform distribution of the particles in matrix [30].

3.2 Mechanical characterization

3.2.1 Tensile behaviour

Table 3: Tensile test results of A and ASiN composite.

Mechanical properties	As cast (0% Si_3N_4)	(7.5% Si_3N_4)	% change
Tensile strength (MPa)	170	226	33
% Elongation	3.704	2.852	-23
Yield stress (MPa)	145.5	187.5	29

The tensile properties such as yield strength (YS), ultimate tensile strength (UTS) and percentage elongation (PE) of base metal and developed copper based composite ASiN are evaluated and presented in Table 3. Compared to base metal, ASiN composite showed 33% increase in UTS, 29% increase in YS and 23% decrement in PE. From the tabulated values, it is evident that Si_3N_4 particles are very effective in improving the tensile strength of Cu-Sn matrix. This rise in UTS and YS can be attributed to the presence of hard Si_3N_4 reinforcing particles in the matrix. The application of uniaxial tensile load results in the movement of dislocations through the matrix. They pile up near the boundaries of reinforcement particles, giving rise to plastic zones. The size of this plastic zone depends on difference in coefficient of thermal expansion of the matrix and the reinforcement phase [31]. It is known that thermal expansion co-efficient of Si_3N_4 particulate is $1.4\text{--}3.7 \times 10^{-6}/\text{K}$ and that of Copper is $17 \times 10^{-6}/\text{K}$ [32, 33]. As this mismatch in thermal expansion coefficients of the composite constituent's increases, the size of the plastic zone also increases [34]. The piled up dislocations eventually propagate through the interface when a critical shear stress is induced at higher loads [35]. Thus the interface between reinforcement and copper matrix provides efficient barrier

Figure 5: ASiN composite samples (a) SEM and (b) EDAX pattern.

to the propagation of dislocations across the interface. That is how Si₃N₄ hard particles impart strength to the matrix and due to this strengthening mechanism, composite offers more resistance to tensile force applied.

Nevertheless, percentage elongation has reduced by 23 %, only to infer that higher tensile strength is achieved at the cost of ductility of copper Matrix. Reinforcing Si₃N₄ particles when added to Cu-Sn matrix, the matrix content gets reduced and the reinforcing particles being less ductile inhibit the flow-ability of Cu-Sn matrix. Similar results of improvement in tensile strength due to the addition of reinforcement to the matrix and reduction in strain at failure are documented in research regardless of the preparation method of Copper MMCs [15, 19, 21]. Figure 6 shows the fractured tensile specimens of both A and ASiN and Figure 7 presents stress-strain graphs for both Cu-Sn base material and ASiN composite.

Figure 7: Stress-Strain curve for (a) base metal 'A' and (b) ASiN reinforced composites.

3.2.2 Fractography

This deals with the study of fractured surface of the material. The SEM micrograph of the fractured surfaces after the tensile testing of the pure copper and ASiN composites are shown in Figure 8. The base metal exhibits ductile fracture as depicted in Figure 8(a). Characteristic feature of ductile fracture is the presence of dimples and can be observed all over the surface of the SEM micrograph. Voids nucleate at inclusions, due to plasticity they grow and then finally coalesce, to form cracks resulting in ductile fracture of the material [36]. The smaller dimples are observed in Figure 8(b) in the micrograph of the fractured surface of ASiN composite compared to its matrix. This confirms that the fracture behaviour of the ASiN composite is brittle in nature. Large voids and propagated cracks also can be seen in the micrograph. The reinforcing hard particles entrapped in the matrix material play a vital role in inhibiting the faster propagation of cracks in the materials. Hence these materials have better tensile strength compared to matrix material.

Figure 6: Fractured tensile specimens of (a) base metal 'A' and (b) ASiN composite.

Figure 8: Fractured surfaces of (a) base metal 'A' and (b) ASiN composite.

3.3 Hardness

Hardness is a mechanical property which signifies the resistance to permanent deformation. The hardness behaviour of both base metal 'A' sample and ASiN composite were evaluated using Brinell hardness test and values are presented in the Figure 9. The BHM of ASiN is higher than the base metal 'A' sample. The higher hardness of the ASiN is because of the presence of hard reinforcement of SiN particles. When an external load is applied, as per the composite loading rule, the hard SiN reinforcement share more load, when compared to soft matrix. Addition of 7.5 wt% of SiN to Cu-Sn matrix has resulted in increased hardness of matrix by 41%. This increment in the hardness signifies proper distribution of the reinforcing particles in the matrix and the good interfacial contact between the reinforcing particles and copper matrix. They strengthen the composite materials by restricting the grain growth [37].

Figure 9: Hardness values of Base metal 'A' and ASiN reinforced composites.

This is in consistence with already presented results which can be summarised as; hardness of MMC is dependent on the:

1. Amount and distribution of the reinforcing particles [36]
2. Interfacial contact between the composite constituents [37]
3. Hardness of the embedded particles [40]. For example addition of MoS₂ and graphite [41] reduce the hardness of the matrix whereas addition of hard particles such as Al₂O₃ [41, 40, 38], SiC [41], CeO₂ [42] and WC [39] etc. have opposite effect.

4 Conclusions

In the present investigation, SiN-reinforced Cu-Sn based composites were successfully prepared two-step stir casting technique. The focus of study was to evaluate the influence of addition of hard SiN reinforcing particles to soft Cu-Sn matrix on its mechanical performance. The SEM morphology and EDAX spectrum of the developed ASiN composites conclude the uniform distribution of the reinforcement in the matrix. Hardness and tensile behaviour of the composites were studied using Brinell hardness test and tension test. The addition of hard SiN particles is found to increase hardness and ultimate tensile strength of the composite. SiN reinforcing materials added to the extent of 7.5% wt to Cu-Sn matrix enhanced hardness by 41% and UTS by 33%, YS by 29% and showed 23% decrement in ductility. Thus a comparison of SiN-reinforced Cu-Sn based composites with base metal has shown improved hardness and tensile properties such as UTS and YS.

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