Processing and properties of Cu based micro- and nano-composites

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Abstract. Nano-composites of 1, 3, 5 and 7 vol% Al_2O_3 (average size <50 nm) and microcomposites having compositions 5, 10, 15, 20 vol% of Al_2O_3 (average size $\sim 10 \mu\text{m}$) reinforced in copper matrix were fabricated by powder metallurgy route. All the specimens were sintered at different sintering temperatures (850, 900 and 1000 °C) to study the effect of temperature on the process and progress of sinterability of the reinforced micro- and nanoparticles in the matrix. These micro- and nano-composites were characterized using X-ray diffraction and scanning electron microscopy followed by density, microhardness and wear measurements. The compression and flexural tests were also carried out in order to investigate the mechanical behaviour of the micro- and nano-composites for a fixed optimum sintering temperature. Fractography of the 3-point bend specimens was performed to investigate the fracture behaviour of the micro- and nano-composites. The flexural test results showed that the ultimate flexural strength decreases and flexural modulus increases with the increase in reinforcement content.

Keywords. Cu– Al_2O_3 ; nano-composite; sintering; 3-point bend test.

1. Introduction

Metal matrix composites (MMCs) integrate the ductility of metal and the toughness of ceramic particles which makes it an excellent material for advanced engineering system (Kelly and Zweben 2000). The unique features of MMCs are high strength to weight ratio, high stiffness per unit density as a result of which the service performance increases. The decrease in structural weight, increase in creep strength, high fatigue strength, high thermal stability, enhancement in wear resistance and electrical conductivity, (Suresh *et al* 1993; Shon *et al* 2009) further make it a potent engineering material.

Copper has been extensively used as matrix because of its superior thermal and electrical conductivity. On the contrary copper has inadequate mechanical properties from the structural application point of view. Incorporation of fine ceramic particles as oxides or carbides in copper strengthens the matrix which enhances the strength and hardness causing a quantifiable degradation in electrical conductivity. Al_2O_3 increases the recrystallization temperature and strength at elevated service temperature by pinning the grain and sub-grain boundaries of Cu matrix (Korac *et al* 2008). Orowan strengthening mechanism, Hall–Petch relation and Taylor work hardening equation are some of the best functional tools to explain the strengthening mechanisms of MMCs (Dash *et al* 2012).

The driving phenomena for generation of dislocations in MMCs are misfit strain, thermal misfit, allotropic misfit,

lattice parameter misfit and elastic inhomogeneity misfit (Kelly and Zweben 2000). In case of solid solution strengthening the dislocation motion gets impinged principally by elastic interaction between the dislocation and solute atoms as well as by alteration of the energy of stacking fault (Dieter 1988). The wide applications of Cu– $\text{Al}_2\text{O}_3(p)$ composites include lead wires, resistance welding electrodes, relay blades, electrical connectors, continuous casting mould and first wall of nuclear reactors such as ITER (international thermonuclear experimental reactor) (Motta *et al* 2001; Rajkovic *et al* 2009, 2010; Dash *et al* 2012).

Powder metallurgy route is one of the versatile routes for fabrication of MMCs because of less probability of particle segregation, undesirable brittle phase formation, free from cast defects, consistent distribution of reinforcement and superior mechanical properties (Suresh *et al* 1993; Lloyd 1994; Lu *et al* 1996). Particle reinforced composites have isotropic properties as compared to fibre or whisker reinforced composites (Ayyar and Chawla 2006). In case of particle reinforced MMCs, the service life of the material is strongly influenced by the nature of matrix materials, individuality of the reinforcement, interface characteristics, reinforcement clustering and formation of interfacial products.

Thermal stress or phase stress in composites develops due to mismatch of coefficient of thermal expansion of matrix and reinforcement. The phase stress depends on reinforcement volume fraction, morphology of the reinforcement, matrix crystallographic texture, porosity, possible voids, loading conditions and state of stress. The thermal stress generates tensile stress on matrix and compressive stress on reinforcement (Agrawal and Sun 2004). The thermal strain

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ε , developed at the interface of discontinuously reinforced MMCs due to thermal stress is expressed as

$$\varepsilon = \delta\alpha\delta T,$$

where $\delta\alpha$ is the difference between the thermal expansion coefficient of the reinforcement and matrix and δT the range of temperature experienced during processing. The generated thermal strain may exceed the local yield strain of matrix–reinforcement interface which will lead to the damage accumulation at the interface. The resultant phenomena of thermal stress are particulate fracture, debonding and cracking in the matrix–reinforcement interface, failure in the matrix via micro-void coalescence and shear fracture of the matrix which deteriorate the composite properties. The above failure mechanisms act as relaxation phenomena and lower the internal strain energy and entropy of the composite (Clyne and Withers 1993; Whitehouse and Clyne 1993; Kelly and Zweben 2000). Characterization of damaged particles will be valuable in improving processing procedures and in understanding the deformation and failure of MMCs.

The crucial parameters influencing the fracture of a particle reinforced composites are size, shape, concentration and spatial distribution of the reinforcement, the concentration of impurities present in the constituents phase of composites, thermal and chemical exposure environment and particular reinforcement size matrix alloy combination (Tong and Ravichandran 1994; Ayyar and Chawla 2006). A clean metallurgical interface (free from voids) is desired for effective reinforcement strengthening (Ye *et al* 2005). Kapoor and Vecchio (1995) have studied the deformation behaviour and failure mechanism of particle reinforced aluminum metal matrix composite. It is necessary to evaluate the fracture micromechanics of the composite for the critical applications. Micromechanics have proved to be a better tool to estimate the microstructural integrity and homogeneity which gives the blue print for the mechanical index of the composite.

Dash *et al* (2012) have studied the effect of sintering atmosphere on the sintering property of the composite. The selection of sintering atmosphere for a system is purely system specific. The metals require protection from oxidation since oxides hinder diffusion bonding and evolution of desired properties need an inert or reducing environment. An appreciable bonding has been observed in an argon atmosphere sintering. Karak *et al* (2012) studied the effect of sintering temperature on the grain growth of matrix and reinforcement particle of the composite and conclude that as the sintering temperature increases the grain coarsening phenomena predominates. Upadhyaya and Upadhyaya (1995) have investigated the physical properties such as densification parameter, nature of porosity and electrical conductivity of Cu–Al₂O₃ composites with the variation in sintering temperatures. Bera *et al* (2010, 2012), Bera and Manna (2012) have fabricated nano-composites by mechanical alloying followed by laser sintering, equal channel angular pressing and high pressure sintering which show improved dispersion of

alumina in Cu alloy and moreover possesses appreciable electrical conductivity and wear resistance. Wen *et al* (2012) adapted a novel chemical reduction method to synthesize Cu–Al₂O₃ nano-composites, where the alumina rearrange in columns suggesting good electrical conductivity.

The understanding of Cu–Al₂O₃ system is confined to the enhancement in mechanical properties by decreasing the alumina particle size to nanometer scale (by mechanical milling) till date. This investigation emphasizes on the comparative discussion of service reliability of the micro- and nano-composites by improvement in their mechanical properties, in terms of their microstructural physical integrity in the interfacial zone. The present study will enable in predicting desired applications for micro- as well as nano-composites according to their respective advantageous properties.

In present study, the interfacial integrity and sinterability of reinforcement particle in the matrix at different sintering temperatures were investigated by SEM. Status of the interface of Cu–Al₂O₃(*p*) composites with the variation of Al₂O₃ content as well as size and sintering temperature were investigated extensively. The microscopic deformation behaviour of Cu–Al₂O₃(*p*) composites were investigated by 3-point bend test followed by fractography. Subsequently phase analysis, percentage of theoretical density, microstructure, hardness, flexural strength and failure mode of the composites were elaborately investigated to arrive at a conclusive particle size – process parameter – microstructure – properties correlation.

2. Materials and methods

2.1 Fabrication of composites

Cu powder (Loba Chemie average particle size $\sim 11.09\ \mu\text{m}$), Al₂O₃ powder (Sigma Aldrich, average particle size $\sim 10\ \mu\text{m}$) and Al₂O₃ nanopowder (Sigma Aldrich, average particle size $< 50\ \text{nm}$) were used to fabricate Cu–Al₂O₃ micro- and nano-composites. Cu and Al₂O₃ (5, 10, 15 and 20 vol%) micropowders were blended using agate mortar followed by compaction to 15 mm diameter cylindrical pellets in an electrically operated uniaxial cold compaction machine (Soil Lab) at an applied pressure of 700 MPa. Three sets of specimen were prepared and sintered in a tubular furnace (Naskar) at three different sintering temperatures (850, 900 and 1000 °C) for 1 h in an argon atmosphere. The above procedure was also followed for Cu and Al₂O₃ (1, 3, 5, 7 vol%) nanopowder to fabricate nano-composites with the variation of sintering temperature.

2.2 Characterization of composites

The specimens were characterized using X-ray diffraction (PANalytical model: DY-1656) CuK α and scanning electron microscopy (JEOL 6480 LV). SEM micrographs of the specimen were obtained by treating the samples with ferric

chloride solution (5 g FeCl_3 and 50 ml HCl in 100 ml distilled water) as an etchant. Sintered density of the pellets was determined by the Archimedes method.

2.3 Mechanical testing

Microhardness of the specimen was measured by Vickers hardness tester (Leco Microhardness Tester LM248AT) at a load of 0.3 kgf for a dwell time of 5 s. The readings were recorded at four equivalent locations for each specimen. According to ASTM standard E9-89 compression test was conducted with the aid of universal testing machine (INSTRON SATEC series servo-hydraulic machine) with specimen dimensions of 10 mm diameter and 8 mm height at a strain rate of 1 min^{-1} . Graphite powder was used to minimize friction between the sample and fixture of compression machine. A reduction of 50% was maintained for all the specimens. The end surfaces were kept normal to the axis of the specimen. A set of specimens having dimensions of $31.5 \times 12.7 \times 6.3 \text{ mm}^3$, in accordance with ASTM standard B925-08 were fabricated following the above mentioned route for both micro- and nano-composites. A span length of 26 mm and strain rate of 0.5 mm/min were maintained during the flexural tests which were carried out in universal testing machine (INSTRON-5967). All the specimens for compression as well as flexural tests were sintered at 900°C for 1 h in an argon atmosphere. Fractography of the 3-point bend test specimens was carried out by scanning electron microscopy (JEOL 6480 LV). Calculations were performed to determine the strain hardening exponent of the composite from the compression test data. The micro- and nano-composites which were sintered at 850°C were subjected to wear test at a load of 20 N at a speed of 30 rpm for a period of 10 min. The wear-tested specimens were examined by scanning electron microscopy.

3. Results and discussion

3.1 X-ray diffraction

X-ray diffraction patterns of $\text{Cu-Al}_2\text{O}_3(p)$ microcomposites sintered at 900 and 1000°C sintering temperatures are illustrated in (figures 1a and b) respectively. X-ray peaks confirm the presence of Cu, Al_2O_3 and Cu_2O phases. As the vol% of Al_2O_3 increases the intensity of the Al_2O_3 peak increases. The formation of Cu_2O at 900 and 1000°C can be attributed to high sintering temperatures as well as high susceptibility of oxide formation of Cu at high temperatures. With the increase in Al_2O_3 content the intensity of Cu_2O peak increases. This can be attributed to the enhanced formation of interface with increasing Al_2O_3 content resulting in the generation of new surface which gets exposed to oxide formation (Ghasemi *et al* 2009). Figures 1(c) and 1(d) refer to X-ray diffraction patterns of $\text{Cu-Al}_2\text{O}_3(p)$ nano-composites sintered at 900 and 1000°C temperatures. The cuprous oxide peaks are prominent in the diffraction patterns

of nano-composites than in microcomposites. This may be due to the presence of Al_2O_3 nanoparticles in Cu matrix leading to higher interface area resulting higher amount of Cu matrix exposed for oxidation.

3.2 Scanning electron microscopy

SEM micrographs give abundant information about the reinforcement distribution, status of physical intimacy between Cu and Al_2O_3 , clustering and mechanical phenomena like twinning. The black regions in SEM micrographs indicate Al_2O_3 particles while the white portion corresponds to Cu matrix captured in back-scattered electron mode. Figures (2a–c) illustrate the distribution of Al_2O_3 in Cu matrix in the microcomposites sintered at various sintering temperatures. With the increase in volume fraction of Al_2O_3 , the efficiency of distribution becomes remarkably better (figures 2d and e). The density difference between the matrix and reinforcement also leads to the formation of clusters sometimes at high vol% of the reinforcement (Slipenyuk *et al* 2006). Annealed twin bands have been observed in (figure 2b) of Cu-10% Al_2O_3 composite sintered at 900°C . Twin is a predominant phenomenon in Cu composites which gives a good indication in terms of mechanical value (Hertzberg 1996). The presence of twins (figure 3a) in composites signifies the reduction in dislocation mobility or dislocation structure stabilization which is an important condition for improving the mechanical properties of the composites (Korac *et al* 2010). On the contrary no twin bands are to be seen in (figure 2c) of Cu-10% Al_2O_3 composite sintered at 1000°C . The matrix grain growth is quite pronounced at 1000°C than at 900 and 850°C . The physical contact between matrix and reinforcement seemingly improves with increasing sintering temperature (figure 3c). Figure 3(d) illustrates the damage accumulation in Cu matrix due to cluster formation of Al_2O_3 microparticles. EDS analysis (the whole micrograph was selected for EDS analysis) in (figure 3e) showing Cu, Al and O wt% in Cu- Al_2O_3 microcomposite. The SEM micrographs of Cu- Al_2O_3 nano-composites illustrate the improved distribution of Al_2O_3 in the matrix compared to that in microcomposites (figures 4a–c) (Dash *et al* 2012). The agglomeration of Al_2O_3 nanoparticles is quite vigilant in (figure 5a). The embedment of Al_2O_3 nanoparticles in Cu matrix is appreciably intimate than in the microcomposites (figure 5b). The enhanced physical contact of Al_2O_3 nanoparticles with the matrix can be attributed to the high atomic diffusivity of the nanoparticles (Upadhyaya 2011). The stabilization of the surface energy of nanoparticles is a thermodynamic driven phenomenon; hence it is quite obvious that the physical adherence of Cu with Al_2O_3 is better in nano-composites.

3.3 Density

Density measurements were carried out using the Archimedes water immersion method. The theoretical and sintered

Figure 1. X-ray diffraction patterns of Cu- Al_2O_3 (p) micro-composites sintered at (a) 900 °C, (b) 1000 °C and nano-composites sintered at (c) 900 °C and (d) 1000 °C.

density values of all the specimens have been tabulated (table 1). Figures 6(a) and 6(b) demonstrate that the percentage of theoretical density (calculated by the rule of mixtures) decreases with increasing content of Al_2O_3 micro- and nanoparticles. This may be due to low density value of Al_2O_3 particles than that of Cu (Celebi Efe *et al* 2011). With the increase in sintering temperature from 900 to 1000 °C the percentage theoretical density also increases. Percentage of theoretical density of the microcomposites increases at a temperature of 1000 °C as compared to 900 °C sintering temperature. This may be due to the enhanced viscosity of Cu matrix at higher sintering temperature which results in efficient pore filling. In case of nano-composites as the interfacial area is more, the detrimental effect of interfacial phenomenon (decohesion, void formation) are more likely to prevail at high sintering temperature as compared to lower sintering temperature. The maximum density value

was observed for the sintering temperature at 850 °C. The rise in sintering temperature triggers enhanced formation of Cu_2O (figure 1b) representing that the matrix is more susceptible to oxide formation at higher sintering temperature. The pullout of cuprous oxide by evolution of O_2 gas (decomposition of cuprous oxide into Cu and oxygen) creates voids in the matrix and interface expanding the matrix eventually. The creation of voids in the matrix hinders the densification and impedes the continuity in intimacy contact of Cu and Al_2O_3 (Ghasemi *et al* 2009; Dash *et al* 2012). Hence, the interfacial area being more in nano-composites the degree of oxidation is higher thus limiting densification at higher sintering temperatures. Shehata *et al* (2009) have investigated the densification% of Cu-5 vol% Al_2O_3 at 950 °C sintering temperature (with finer Al_2O_3) to be 93.9% and our results show 94.47% densification for the same composition at 1000 °C sintering temperature.

Figure 2. SEM micrographs representing the reinforcement particle distribution of Cu-10% Al₂O₃ microcomposites sintered at different sintering temperatures (a) 850 °C, (b) 900 °C, (c) 1000 °C, (d) Cu-15% Al₂O₃ and (e) Cu-20% Al₂O₃ microcomposites sintered at 1000 °C temperature.

3.4 Hardness

The hardness of the micro- and nano-composites increases with the increase in Al₂O₃ content in the matrix. As Al₂O₃ is inherently harder than Cu, its presence leads to a higher hardness in the composite. It was observed from (figure 7a) that by increasing the amount of Al₂O₃ from 0 to 20% in microcomposites, the hardness value increases from 53.7 to 103 HV at a sintering temperature of 900 °C. The hardness enhancement is an indication to good physical bonding at Cu–Al₂O₃ interface (Daoushb *et al* 2009). As the sintering temperature increases from 900 to 1000 °C, the hardness value decreases which may be due to matrix grain coarsening (Rajkovic *et al* 2008). At high volume fraction of reinforcement, surface area of Al₂O₃ particles reduces due to clustering (figure 3b) which minimizes

the effect of grain boundary pinning resulting in lower hardness value. In case of nano-composite nano-Al₂O₃ which have high hardness impedes the movement of dislocation during plastic deformation. Nano-Al₂O₃ restricts the grain growth of Cu matrix by effective pinning (Tian *et al* 2006). The hardness of the composite gets improved with increase in the reinforcement content. The hardness values are maximum for the composites sintered at 850 °C temperature (figure 7b). This can be further complemented by the density values. Al₂O₃ nanoparticles tend to agglomerate which decreases the Orowan strengthening effect. The hardness values trend represents better hardness of Cu–Al₂O₃ nano-composites at low sintering temperature such as 850 °C. The highest hardness values recorded are 108 HV for 7 vol% reinforced Cu–Al₂O₃ nano-composites. The density hierarchy complements the hardness trends

Figure 3. (a) SEM micrograph illustrating twins in Cu-10% Al_2O_3 microcomposites sintered at 900 °C. (b) SEM micrograph illustrating the clustered region of microcomposites in Cu-15 vol% Al_2O_3 sintered at 900 °C. (c) SEM micrograph illustrating the improved physical contact in microcomposites in Cu-10 vol% Al_2O_3 sintered at 1000 °C. (d) SEM micrograph illustrating the particle cracking in the clustered region of microcomposites in Cu-10 vol% Al_2O_3 sintered at 900 °C. (e) EDS analysis showing Cu, Al and O wt% in Cu-5 vol% Al_2O_3 microcomposite sintered at 900 °C.

followed at different sintering temperatures. The lowest sintering temperature imparts highest density to the nano-composites and this trend is also followed for the rest of the temperatures. This can be ascribed to the fact that the interface dimension in the nano-composites is high, leading to increased susceptibility for interfacial de-cohesion. Hence, at higher sintering temperature exposed area for oxidation is also higher, the hardness values are lower due to subsequent decomposition of cuprous oxide and matrix expansion. Tjong and Lau (2000) have reported 101 HV Vickers microhardness for Cu-20 vol% SiC HIPed composites whereas in our results we have obtained 103 HV Vickers microhardness for Cu-20 vol% Al_2O_3 microcomposites with conventional sintering.

3.5 Mechanical testing

3.5a Compression test: The compression test results of Cu- Al_2O_3 micro- and nano-composites have been illustrated in (figures 8a and c). The results indicate that the compression strength decreases with the increase in Al_2O_3

content. The compression strength of Cu- Al_2O_3 microcomposites decreases with increasing the amount of Al_2O_3 at a strain rate of 1 min^{-1} . With the increase in reinforcement content, the compressive strength decreases in case of microcomposites. Localized softening of the composite (difference in thermal conductivity of Cu and Al_2O_3 constituents) at high volume fraction of reinforcement during compressive testing may possibly decrease the compressive strength (Tan *et al* 2008). The compressive strength for Cu- Al_2O_3 nano-composites increases as Al_2O_3 content increases till 3 vol% of Al_2O_3 followed by downfall of compressive strength values. The decrease after 3 vol% may be attributed to the agglomeration of Al_2O_3 nanoparticles at higher vol% of Al_2O_3 . The agglomeration leads to increase in damage density and hence early fracture.

The strain-hardening exponent(n) was calculated from the true stress-strain curves (figures 8b and d) for micro- and nano-composites respectively. For elastically deformed composite, the strain hardening exponent n can be estimated as

$$\{n = (\ln \sigma_1 / \sigma_2) / (\ln \varepsilon_1 / \varepsilon_2)\}.$$

Figure 4. SEM micrographs representing the reinforcement particle distribution of Cu-5% Al₂O₃ nano-composites sintered at different sintering temperatures (a) 850 °C, (b) 900 °C and (c) 1000 °C.

Figure 5. SEM micrographs representing the matrix-reinforcement compatibility in Cu-3% Al₂O₃ nano-composites sintered at different sintering temperatures (a) 900 °C and (b) 1000 °C.

For composites, the presence of particles induces an inhomogeneous elastic strain due to the elastic modulus mismatch of matrix Cu (117 GPa) and reinforced Al₂O₃ particle (300 GPa). The elastic strain leads to the generation of high geometrically necessary dislocation density ρ_G in the composite matrix interface. The composite reinforced with microparticles tends to fail through particle fracture which leads to decrease in strain hardening exponent. For nanoparticles, the increased geometrically necessary dislocation density ρ_G leads to a higher work hardening in the matrix, thus leads to a higher composite flow stress (Fernandez *et al* 2005). With increase in reinforcement content nano-composite tends to fail through void nucleation, growth and coalescence in the matrix regions near particles. The above failure mechanisms lead to the decrease in strain hardening exponent. The compressive strength of 3 vol% alumina

reinforced copper nano-composite around 800 MPa whereas (Fathy *et al* 2012) have reported around 600 MPa for 12.5 wt% Cu–Al₂O₃ nano-composite at 10^{-2} s^{-1} strain rate.

3.5b 3-point bend test: From 3-point bend test result, it was found that the ultimate flexural strength value decreases with increase in volume fraction of the reinforcement illustrated in (figures 9a and b). The underlying reason could be the addition of ceramic particles which decreases its ductility component, hence lowering the ultimate flexural strength. This may be due to the enhancement in damage sites with increase in reinforcement content. Early fracture possibly occurs by linking the damage in clustered regions as a result ultimate flexural strength value decreases. According to Griffith criterion cracked particle in the clustered region hinders dislocation strengthening which decreases the

Table 1. Theoretical and sintered density values of Cu–Al₂O₃ micro- and nano-composites processed at different sintering temperatures.

Composition	Theoretical density	Sintering temperature		
		850 °C Sintered density	900 °C Sintered density	1000 °C Sintered density
Pure copper	8.9	8.05	8.4	8.49
Cu-1% nanoalumina	8.85	8.31	7.92	7.54
Cu-3% nanoalumina	8.75	8	7.55	7.51
Cu-5% nanoalumina	8.65	7.61	7.37	7.2
Cu-7% nanoalumina	8.55	7.26	7.27	6.97
Cu-5% microalumina	8.69	7.62	7.9	8.22
Cu-10% microalumina	8.49	7.36	7.64	7.75
Cu-15% microalumina	8.14	7.04	7.3	7.35
Cu-20% microalumina	7.9	6.7	7.03	6.88

Figure 6. Densification plot of (a) microcomposites and (b) nano-composites with different vol% of Al₂O₃ sintered at different sintering temperature.

ultimate flexural strength value. Figures 9(a) and (b) reveals a marginal increase in flexural modulus of the composite with an increase in reinforcement content. Defects such as porosity, macro-, micro-cracks and interface deunion promote premature failure and ultimate fracture occurs early. The flexural modulus values indicate the higher stiffness of the Cu–Al₂O₃ nano-composites compared to microcomposites.

3.6 Fractography

Fracture surface of the three-point flexural test microcomposites specimens are shown in (figures 10a and b). The fracture mode of the micro- and nano-composites is seemingly mixed mode. The dimple fracture surface (figure 10a) suggests ductile fracture as the principal mode of fracture in the microcomposite. Fracture surface of the microcomposite exhibit micro-void coalescence in the matrix and particle cracking or matrix-particles interface decohesion. No secondary cracks are observed in the brittle Al₂O₃ particle which indicates the premature failure of the composite (Akhtar *et al* 2009). Damage is possibly nucleated by

interfacial decohesion, particle cracking, void growth and finally coalescence of voids in the matrix around the reinforcement particles. The dimple size decreases with increase in Al₂O₃ content. Dimples growth hindered by the presence of Al₂O₃ particles. The sharp micro-cracks that develop due to particle fracture can enhance the localized plastic flow within the ductile matrix which aids in failure phenomena such as ductile separation of the matrix by void growth and shear bands (Rabiei *et al* 2008). The fracture mode for nano-composites (figures 10c and d) also depict mixed mode, brittle mode of fracture being the predominating one. The presence of river line indicates closeness of the fracture to brittle mode. The fracture surface of nano-composites exhibits more of void formation and matrix-reinforcement debonding. Crack formation and bridging is also visible in (figures 10c and d). For high volume fraction of reinforcement content, early fracture of material occurs with minimal plastic deformation. The macroscopic features of the fracture surface of Cu–Al₂O₃ nano-composites indicate nearly flat surface, minimal plastic deformation preceding the fracture and rapid crack growth accompanied by loud noise

Figure 7. Microhardness plot of (a) microcomposites and (b) nano-composites with different vol% of Al_2O_3 sintered at different sintering temperature.

Figure 8. (a) True stress–true strain compressive plot for microcomposites, (b) strain hardening exponent vs vol% of reinforcement content plot for microcomposites, (c) true stress–true strain compressive plot for nano-composites and (d) strain hardening exponent vs vol% of reinforcement content plot for nano-composites.

Figure 9. Plot for ultimate flexural stress (MPa) and flexural modulus (GPa) vs vol% of reinforcement content for (a) microcomposites and (b) nano-composites.

Figure 10. SEM micrograph illustrating the 3-point bend test specimen fracture surface having composition (a) Cu-5% Al_2O_3 microcomposite, (b) Cu-10% Al_2O_3 microcomposite, (c) Cu-5% Al_2O_3 nano-composites, (d) Cu-7% Al_2O_3 nano-composites and (e) fracture surface of micro- and nano-composites.

(ASM handbooks Fractography, 1987). The microscopic characteristics include river lines, faceted fracture surface and transgranular fracture of Cu grains in the nano-composites. SEM fractographs indicate quasi-cleavage mode of fracture in Cu- Al_2O_3 nano-composites. Figure 10(e) represents the fracture surface of micro- and nano-composites.

3.7 Wear test

The abrasive wear rate is greatly reduced as Al_2O_3 content in the composite increases (figures 11a and b). Al_2O_3

being inherently harder than Cu, hence the wear resistance of the composites are higher than native Cu. However, as the number of Al_2O_3 microparticles increase, the resistance to the penetration of abrasive particles also increases (hardness increases with increase in the reinforcement content) and hence the wear depth decreases (Shehata *et al* 2009). The drastic reduction in wear rate may be attributed by (1) enhancement in hardness of the composite reinforced by Al_2O_3 particles (Ramesh *et al* 2009) and (2) greater reduction of direct load contact between Cu/ Al_2O_3 composite surface and disk in comparison with

Figure 11. Plot for wear depth vs time (a) microcomposites and (b) nano-composites with different vol% of Al₂O₃ sintered at 850 °C temperature.

Figure 12. SEM images of worn-out surfaces of (a) Cu-5 vol% Al₂O₃ (microcomposite), (b) Cu-10 vol% Al₂O₃ (microcomposite), (c) Cu-1 vol% Al₂O₃ (nano-composite) and (d) scanning electron micrograph of worn-out surfaces of Cu-5 vol% Al₂O₃ (microcomposite) representing the microgrooves.

pure Cu due to load bearing component action of hard Al₂O₃ particles. The worn out specimens' SEM micrographs (figures 12a–c) represents that, with increase in reinforcement content the surface roughness decreases. As nanoparticles act as obstacle for the smooth removal of material the abrasion resistance also increases. The wear track width decreases with the rise in Al₂O₃ content in both micro- and nano-composites. Figures 12(a) and (c) represents narrow wear track in nano-composites than in the microcomposites. The wear operating mechanisms can be enlisted as grooving and micro-plastic ploughing. The microploughing has eventually

created long grooves and the removed material has been pushed to the ridges of the grooves (figure 12d). The intensity of microploughing decreases as Al₂O₃ content in the composites increases (Fathy *et al* 2012).

4. Conclusions

Cu–Al₂O₃ micro- and nano-composites were fabricated by blend-compact-sinter powder metallurgy route at different sintering temperatures. The microcomposites possess

better properties (density and hardness) at higher sintering temperatures. This behaviour in nano-composites showed in other way round. The incorporation of Al_2O_3 nanoparticles strengthens the matrix to a greater extent resulting in the increase of microhardness compared to Al_2O_3 microparticles. The microstructures of micro- as well as nano-composites demonstrate better distribution in the latter. Compressive strength is higher in the nano-composites. The flexural modulus of nano-composites is higher as compared to the microcomposites. Fractography of the microcomposites and nano-composites revealed mixed mode of fracture in both the cases. The wear resistance of nano-composites is higher than the microcomposites under the same experimental conditions. The rise in reinforcement content imparts higher microhardness, low density and high wear resistance values for both the systems.

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