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<td>Article Type:</td>
<td>Original Research</td>
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<td>Keywords:</td>
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Processing and properties of Cu based micro- and nano-composites

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Abstract

Nanocomposites of 1, 3, 5 and 7 vol. % Al₂O₃ (average size<50nm) and microcomposites having compositions 5, 10, 15, 20 vol. % of Al₂O₃ (average size~10µm) reinforced in copper matrix were fabricated by powder metallurgy route. All the specimens were sintered at different sintering temperatures (850°C, 900°C, 1000°C) to study the effect of temperature on the process and progress of sinterability of the reinforced micro- and nano-particles 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 the ultimate flexural strength decreases and flexural modulus increases with increase in reinforcement content.

Keywords: Cu- Al₂O₃, Nanocomposite; Sintering; 3-point bend test.

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1. Introduction

Metal matrix composites (MMCs) integrate the ductility of metal and the toughness of ceramic particles which makes it an excellent candidate material for advanced engineering system (Kelly et al 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. Al₂O₃ increases the recrystallization temperature and strength at elevated service temperature by pinning the grain and sub- grain boundaries of the Cu matrix (Korac et al 2008). Orowan strengthening mechanism, Hall Petch relation, 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 inhomogenity misfit (Kelly et al 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 et al 1988). The wide applications of Cu–Al₂O₃(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) (Dash et al 2012; Motta et al 2001; Rajkovic et al 2009; Rajkovic et al 2010).

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; Lu et al 1996; Lloyd et al 1994). Particle reinforced composites have isotropic properties as compared to fiber or whisker reinforced composites (Ayyar et al 2006). In case of particle reinforced MMCs the service life of the material is strongly influenced by 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 stresses on reinforcement (Agrawal et al 2004). The thermal strain $\varepsilon$, 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 the matrix and $\delta T$ is 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, de-bonding 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 (Kelly et al 2000; Clyne et al 1993; Whitehouse et al 1993). Characterization of damaged particles will be valuable in improving processing procedures and in understanding 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 (Ayyar et al 2006; Tong et al 1994). A clean metallurgical interface (free from voids) is desired for effective reinforcement strengthening (Ye et al 2005). (Kapoor et al 1995) have studied the deformation behavior and failure mechanism of particle reinforced aluminium metal matrix composite. It is necessary to evaluate the fracture micro mechanics of the composite for the critical applications. Micromechanics have proved to be a better tool to estimate the micro-structural integrity and homogeneity which gives the blueprint for the mechanical index of the composite.

(Dash et al 2012) has 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 that 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 argon atmosphere sintering. (Karak et al 2011) 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 et al 1995) have investigated the physical properties such as densification parameter, nature of porosity and electrical conductivity of the Cu–Al₂O₃ composites with the variation in sintering temperatures. (Bera et al 2012) (Bera et al 2012) (Bera et al 2010) have fabricated nanocomposites 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 nanocomposites where the alumina rearranges in columns suggesting good electrical conductivity.

The understanding of the 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 predicting desired applications for micro- as well as nano-composites according to their respective advantageous properties.

In the present study, the interfacial integrity and sinterability of reinforcement particle in the matrix at different sintering temperatures was 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~11.09 μm), Al₂O₃ powder (Sigma Aldrich, average particle size~10 μm) and Al₂O₃ nanopowder (Sigma Aldrich, average particle size<50 nm) were used to fabricate the Cu- Al₂O₃ micro- and nano-composites. Cu and Al₂O₃ (5, 10, 15 and 20 vol. %) micro-powders were blended using agate mortar followed by compaction to 15 mm diameter cylindrical pellets in an electrically operated uni-axial 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°C, 900°C, 1000°C) for 1 hour in argon atmosphere. The above procedure was also followed for Cu and Al₂O₃ (1, 3, 5, 7 vol. %) nanopowder to fabricate nanocomposites with the variation of sintering temperature.

2.2. Characterization of the composites

The specimens were characterized using X-Ray diffraction (PANalytical model: DY-1656) CuKα and scanning electron microscopy (JEOL 6480 LV). The SEM micrographs of the specimen were obtained by treating the samples with ferric chloride solution (5 g FeCl₃ and 50
ml HCl in 100 ml distilled water) as an etchant. Sintered density of the pellets was determined by Archimedes method.

2.3. Mechanical testing

Micro-hardness of the specimen was measured by Vickers hardness tester (Leco Microhardness Tester LM248AT) at a load of 0.3kgf for a dwell time of 5 seconds. The readings were recorded here 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 1min⁻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 x12.7 x 6.3 mm³, in accordance with ASTM standard B925-08 were fabricated following the above mentioned route for both micro- and nano-composites. A span length of 26mm and strain rate of 0.5mm/min was 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 hour in 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 20N at a speed of 30 rpm for a time period of 10 minutes. The wear tested specimens were examined by scanning electron microscopy.
3. Results and discussion

3.1. X-Ray Diffraction

The X-ray diffraction patterns of Cu-Al$_2$O$_3$(p) microcomposites sintered at 900°C and 1000°C sintering temperature are illustrate in (figure 1(a) and (b)) respectively. The X-ray peaks confirm the presence of Cu and Al$_2$O$_3$ and Cu$_2$O phases. As the vol. % of Al$_2$O$_3$ increases the intensity of the Al$_2$O$_3$ peaks increases. The formation of Cu$_2$O at 900°C 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$_2$O$_3$ content the intensity of Cu$_2$O peak increases. This can be attributed to the enhanced formation of interface with increasing Al$_2$O$_3$ content resulting in generation of new surface which gets exposed to oxide formation (Ghasemi et al. 2009). (Figure 1 (c) and (d)) refer to the X-ray diffraction patterns of the Cu- Al$_2$O$_3$(p) nanocomposites sintered at 900°C and 1000°C temperatures. The cuprous oxide peaks are prominent in the diffraction patterns of nanocomposites than in microcomposites. This may be due to the presence of Al$_2$O$_3$ nanoparticles in Cu matrix leading to higher interface area resulting higher amount of Cu matrix exposed for oxidation.

3.2. Scanning electron microscopy

The SEM micrographs give abundant information about the reinforcement distribution, status of physical intimacy between Cu and Al$_2$O$_3$, clustering and mechanical phenomena like twinning. The black regions in the SEM micrographs indicate the Al$_2$O$_3$ particles while the white portion corresponds to the Cu matrix captured in back scattered electron mode. (figure 2(a)-(c)) illustrate the distribution of Al$_2$O$_3$ in Cu matrix in the micro-composites sintered at various sintering
temperatures. With the increase in volume fraction of Al₂O₃ the efficiency of distribution becomes remarkably better (figure 2(d)-(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 2(b)) of Cu- 10% Al₂O₃ composite sintered at 900˚ C. Twin is a predominant phenomenon in Cu composites which gives a good indication in terms of mechanical value (Hertzberg et al 1996). The presence of twins (figure 3(a)) in the composites signifies the reduction in dislocations mobility or dislocation structure stabilization which is the 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 2(c)) of Cu- 10% Al₂O₃ composite sintered at 1000˚ C. The matrix grain growth is quite pronounced at 1000˚C than at 900˚C and 850˚C. The physical contact between matrix and reinforcement seemingly improves with increasing sintering temperature (figure 3(c)). Figure 3(d) illustrates the damage accumulation in the Cu matrix due to cluster formation of Al₂O₃ microparticles. The EDS analysis (the whole micrograph was selected for EDS analysis) in (figure 3(e)) showing the Cu, Al and O wt. % in the Cu-Al₂O₃ microcomposite. The SEM micrographs of Cu-Al₂O₃ nanocomposites illustrate improved distribution of Al₂O₃ in the matrix compared to that in microcomposites (figure 4(a), 4(b) and 4(c)) (Dash et al 2012). The agglomeration of Al₂O₃ nanoparticles is quite vigilant in the (figure 5(a)). The embedment of Al₂O₃ nanoparticles in Cu matrix is appreciably intimate than in the microcomposites (figure 5(b)). The enhanced physical contact of Al₂O₃ nanoparticles with the matrix can be attributed to the high atomic diffusivity of the nanoparticles (Upadhaya et al 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 Al₂O₃ is better in nanocomposites.
3.4. Density

Density measurements were carried out using the Archimedes water immersion method. The theoretical and sintered density values of all the specimens have been tabulated (Table.1). Figure 6(a)-6(b) demonstrate that the percentage of theoretical density (calculated by rule of mixtures) decreases with increasing content of Al₂O₃ micro- and nano-particles. This may be due to low density value of Al₂O₃ particles than that of Cu (Efe et al 2011). With the increase in sintering temperature from 900°C to 1000°C the % theoretical density also increases. Percentage of theoretical density of the microcomposites increases at a temperature of 1000°C as compared to the 900°C sintering temperature. This may be due to the enhanced viscosity of the Cu matrix at higher sintering temperature which results in efficient pore filling. In case of nanocomposites as the interfacial area is more, the detrimental effect of interfacial phenomenon (de-cohesion, 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 Cu₂O (figure 1(b)) representing that the matrix is more susceptible to oxide formation at higher sintering temperature. The pullout of cuprous oxide by evolution of O₂ 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 Al₂O₃ (Dash et al 2012; Ghasemi et al 2009). Hence, the interfacial area being more in nanocomposites 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.% Al₂O₃ at
950°C sintering temperature (with finer Al$_2$O$_3$) to be 93.9% and our results show 94.47% densification for the same composition at 1000°C sintering temperature.

### 3.5. Hardness

The hardness of the micro- and nano-composites increases with the increase in Al$_2$O$_3$ content in the matrix. As Al$_2$O$_3$ is inherently harder than Cu, its presence leads to a higher hardness in the composite. It was observed from (figure 7(a)) that by increasing the amount of Al$_2$O$_3$ 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-Al$_2$O$_3$ interface (Daoushb et al 2009). As the sintering temperature increases from 900°C 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 Al$_2$O$_3$ particles reduces due to clustering (figure 3(b)) which minimizes the effect of grain boundary pinning resulting in lower hardness value. In case of nano-composite nano-Al$_2$O$_3$ which have high hardness impedes the movement of dislocation during plastic deformation. Nano-Al$_2$O$_3$ restricts the grain growth of the 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 7(b)). This can be further complemented by the density values. The Al$_2$O$_3$ nanoparticles tend to agglomerate which decreases the Orowan strengthening effect. The hardness values trend represents better hardness of Cu-Al$_2$O$_3$ nanocomposites at low sintering temperature such as 850°C. The highest hardness values recorded are 108 HV for 7 vol. % reinforced Cu-Al$_2$O$_3$ nanocomposites. The density hierarchy
complements the hardness trends followed at different sintering temperatures. The lowest sintering temperature imparts highest density to the nanocomposites, 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 nanocomposites is high, leading to increased susceptibility for interfacial decohesion. Hence at higher sintering temperature exposed area for oxidation is higher, so the hardness values are lower due to subsequent decomposition of cuprous oxide and matrix expansion. (Tjong et al. 2000) have reported 101HV Vickers microhardness for Cu-20 vol.%SiC HIPed composites whereas in our results we have obtained 103HV Vickers microhardness for Cu-20 vol.%Al₂O₃ microcomposites with conventional sintering.

3.6. Mechanical testing

3.6.1. Compression Test

The compression test results of Cu-Al₂O₃ micro- and nano-composites have been illustrated in (figure 8(a) and 8(c)). The results indicate that the compression strength decreases with the increase in Al₂O₃ content. The compression strength of Cu-Al₂O₃ microcomposites decreases with increasing amount of Al₂O₃ at a strain rate of 1min⁻¹. With increase in reinforcement content the compressive strength decreases in case of microcomposites. Localized softening of the composite (difference in thermal conductivity of the Cu and Al₂O₃ 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-Al₂O₃ nanocomposites increases as the Al₂O₃ content increases till 3 vol. % of Al₂O₃ followed by downfall of compressive strength values. The decrease after 3 vol. % may be attributed to the agglomeration
of Al₂O₃ nanoparticles at higher vol. % of Al₂O₃. The agglomeration leads to increases in damage density and hence early fracture.

The strain-hardening exponent\( (n) \) was calculated from the true stress-strain curves (figure 8(b) and 8(d)) for micro- and nano-composites respectively. For elastically deformed composite the strain hardening exponent \( n \) can be estimated as \( \{ \text{where, } n = (\ln\sigma_1/\sigma_2)/ (\ln\varepsilon_1/\varepsilon_2) \} \) 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 \( \rho_G \) 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 nano particles the increased geometrically necessary dislocation density \( \rho_G \) leads to a higher work hardening in the matrix, thus leads to a higher composite flow stress (Fernandez et al 2005). With increases in reinforcement content nanocomposite 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 nanocomposite around 800 MPa whereas (Fathy et al 2012.) have reported around 600 MPa for 12.5 wt% Cu-Al₂O₃ nanocomposite at \( 10^{-2} \) sec\(^{-1} \) strain rate.

3.6.2. 3-point bend test

From the 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 (figure 9(a) and 9(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 hindres dislocation strengthening which decreases the ultimate flexural strength value. (figure 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 de-union promote premature failure and ultimate fracture occurs early. The flexural modulus values indicate the higher stiffness of the Cu-Al₂O₃ nanocomposites compared to microcomposites.

3.7. Fractography

Fracture surface of the three-point flexural test microcomposites specimens are shown in (figure 10(a) and 10(b)). The fracture mode of the micro- and nano-composites is seemingly mixed mode. The dimple fracture surface (figure 10(a)) 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 de-cohesion (10). 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 de-cohesion, 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 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 nanocomposites (figure 10(c) 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 nanocomposites exhibits more of void formation and matrix-reinforcement debonding. Crack formation and bridging is also visible in (figure 10(c) 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 nanocomposites indicate nearly flat surface, minimal plastic deformation preceding the fracture and rapid crack growth accompanied by loud noise (Fractography ASM handbook et al 1987). The microscopic characteristics include river lines, faceted fracture surface and transgranular fracture of Cu grains in the nanocomposites. The SEM fractographs indicate quasi-cleavage mode of fracture in Cu-Al₂O₃ nanocomposites. (Figure 10(e)) represents the fracture surface of micro- and nano-composites.

3.8. Wear test

The abrasive wear rate is greatly reduced as Al₂O₃ content in the composite increases (figure 11(a) and 11(b)). Al₂O₃ being inherently harder than Cu, hence the wear resistance of the composites are higher than native Cu. However, as the number of Al₂O₃ microparticles increase, the resistance to the penetration of abrasive particles increases (Hardness increases with increases 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 Al₂O₃ particles (Ramesh et al 2009) and (2) greater reduction of direct load contact between the Cu/Al₂O₃ composite surface and disk in comparison with pure Cu due to load bearing component action of hard Al₂O₃ particles. The worn out specimens’ SEM micrographs (figure 12(a), 12(b) and 12(c)) represents that with increase in reinforcement content the surface roughness decreases. As nano particles 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. The (figure 12(a) and 12(c)) represents narrow wear track in nanocomposites than in the microcomposites. The wear mechanisms operating 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 12(d)). The intensity of microploughing decreases as the Al₂O₃ content in the composites increases (Fathy et al 2012).

4. Conclusion

The 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 nanocomposites showed in other way around. The incorporation of Al₂O₃ nanoparticles strengthens the matrix to a greater extent resulting in increase of microhardness compared to Al₂O₃ microparticles. The microstructures of micro- as well as nano-composites demonstrate better distribution in the later. Compressive strength is higher in the nanocomposites. The flexural modulus of nanocomposites is higher as compared to the microcomposites. Fractography of the microcomposites and nanocomposites revealed mixed mode of fracture in both the cases. The wear resistance of nanocomposites 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.
Acknowledgement

The authors would like to thank the National Institute of Technology (NIT), Rourkela for providing the necessary financial and infrastructural supports.

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**Figure captions**

Figure 1. X-ray diffraction patterns of Cu – Al<sub>2</sub>O<sub>3</sub> (p) microcomposites sintered at (a) 900˚C, (b) 1000˚C and nanocomposites sintered at (c) 900˚C, (d) 1000˚C.

Figure 2. SEM micrographs representing the reinforcement particle distribution of Cu–10% Al<sub>2</sub>O<sub>3</sub> microcomposites sintered at different sintering temperature (a) 850˚C, (b) 900˚C and (c) 1000˚C, (d) Cu–15% Al<sub>2</sub>O<sub>3</sub> (e) Cu–20% Al<sub>2</sub>O<sub>3</sub> microcomposites sintered at 1000˚C temperature.

Figure 3(a). SEM micrograph illustrating twins in the Cu–10% Al<sub>2</sub>O<sub>3</sub> microcomposites sintered at 900˚C.

Figure 3(b). SEM micrograph illustrating the clustered region of microcomposites in Cu-15 vol% Al<sub>2</sub>O<sub>3</sub> sintered at 900˚C.

Figure 3(c). SEM micrograph illustrating the improved physical contact in microcomposites in Cu-10 vol% Al<sub>2</sub>O<sub>3</sub> sintered at 1000˚C.

Figure 3(d). SEM micrograph illustrating the particle cracking in the clustered region of microcomposites in Cu-10 vol% Al<sub>2</sub>O<sub>3</sub> sintered at 900˚C.

Figure 3(e). EDS analysis showing the Cu, Al and O wt. % in Cu-5 vol% Al<sub>2</sub>O<sub>3</sub> microcomposite sintered at 900˚C.
Figure 4. SEM micrographs representing the reinforcement particle distribution of Cu–5% Al₂O₃ nanocomposites sintered at different sintering temperature (a) 850°C, (b) 900°C and (c) 1000°C.

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

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

Figure 7. Microhardness plot of (a) microcomposites and (b) nanocomposites with different vol. % of Al₂O₃ sintered at different sintering temperature.

Figure 8(a). True stress- true strain compressive plot for microcomposites.

Figure 8(b). Strain hardening exponent vs. vol. % of reinforcement content plot for microcomposites.

Figure 8(c). True stress- true strain compressive plot for nanocomposites.

Figure 8(d). Strain hardening exponent vs. vol. % of reinforcement content plot for nanocomposites.

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

Figure 10. SEM micrograph illustrating the 3-point bend test specimen fracture surface of having composition (a) Cu-5% Al₂O₃, microcomposite (b) Cu-10% Al₂O₃, microcomposite, (c) Cu-5% Al₂O₃ nanocomposites, (d) Cu-7% Al₂O₃ nanocomposites.
Figure 10(e). Fracture surface of micro- and nano-composites.

Figure 11. Plot for wear depth vs. time (a) microcomposites and (b) nanocomposites 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 (nanocomposite)

Figure 12(d). Scanning electron micrograph of worn-out surfaces of Cu-5 vol. % Al₂O₃ (microcomposite) representing the microgrooves.

**Table captions:**

Table 1: Theoretical and sintered density values of Cu-Al₂O₃ micro- and nano-composites processed at different sintering temperatures.
Response to reviewer’s comments

Reviewer #1: The author has modified the manuscript and satisfactorily answered the questions. Therefore, it can be considered for publication.

However, it still requires minor revision.

Query 1: In repose of query no 4 authors mention that "The theoretical and measured densities have been incorporated in a table." However, no such table is observed in the manuscript. The author should provide the table.

Response: The density table has been added to the revised manuscript.

Reviewer # 2:

Query 1: Table with density values is missing

Response: The density table has been added to the revised manuscript.
Figure

The graph shows the relationship between hardness (HV) and volume percentage of Al₂O₃ at different temperatures: 850°C, 900°C, and 1000°C. The hardness increases with increasing volume percentage of Al₂O₃ and temperature.

- At 850°C, the hardness starts at approximately 30 HV and increases to about 120 HV as the volume percentage of Al₂O₃ increases from 1% to 7%.
- At 900°C, the hardness range is similar but consistently higher than at 850°C.
- At 1000°C, the hardness range is the highest among the three temperatures, starting at 50 HV and reaching up to 110 HV.

Error bars indicate the variability in the hardness measurements.
Particle/matrix interface de-cohesion
<table>
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<th>Element</th>
<th>Weight%</th>
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<td>Al K</td>
<td>9.32</td>
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<td>Cu K</td>
<td>84.44</td>
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</table>
Al₂O₃ particles
Agglomerated alumina nanoparticles
Reinforcement embedded in the matrix
Table 1: Theoretical and sintered density values of Cu-Al$_2$O$_3$ micro- and nano-composites processed at different sintering temperatures.

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<th>Composition</th>
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<th>850°C</th>
<th>900°C</th>
<th>1000°C</th>
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