Tribological Characterization of Cu-Ni Metal Matrix Composites Using MoS2 Nano-lubricant

Md Firdos Ali¹, M.F.Wani¹,*, Summera Banday¹, Bisma Parveez¹, M. Junaid Mir¹, S. Mushtaq¹

¹Tribology Laboratory, Department of Mechanical Engineering, National Institute of Technology Hazratbal, Srinagar, J&K, India-190006  
*Corresponding author. Tel.: +918803824243; E-mail address: mfwani@nitsri.net

Abstract: In this study, Cu-5wt.%Ni and Cu-10wt.%Ni two metal matrix of copper having 5%, 10% of Ni, reinforced with divergent percentages of titanium carbide (0, 3, 6, and 9wt.%) were synthesized with the help of high-energy ball milling, compaction, sintering. The coefficient of friction and wear characteristic were examined at various normal loads of 30N, 60N, 90N, and 120N, at fixed sliding speed of 0.25 m/s against a harder counter face made of steel, EN8 (HRC 46 - 48) ball under boundary lubrication using a ball-on-disk test equipment. The Cu10wt.%Ni-3TiC composite has a higher value of micro-hardness of 117(HV) and sintered density of 8.036gm/cm³ at 3wt% of TiC. The wear rate and coefficient of friction have been elaborated on the basis of micro-hardness and presence of nano MoS₂ in lubricant. At 3wt% TiC in metal matrix have optimum performance of friction and wear caused. The wear mechanism of the Cu5Ni and Cu10Ni metal matrix was a combination of adhesive and oxidative wear and composites had mainly abrasive wear.

Keywords: copper-based MMCs; TiC particles; boundary lubrication; Friction; Wear.

1. Introduction
Copper and its alloys contribute as a major part of industrial metals. They are very well-known substance that are versatile used in commercial applications specially in sporting goods automobile, aerospace, and its engineering industrial application is mainly due to their excellent thermal conductivity and electrical property, easier to fabrication, and high strength and resistance to fatigue [1]. Copper-based MMCs are auspicious materials for the reason of their excellent thermo-physical feature. It is also used in various industrial applications, such as, brush materials and torch nozzle, electrical sliding contact (railway overhead current collector systems). Strength of copper can be increased by alloying copper with other elements or by reinforced fine particles in copper based matrix. The reinforced particles may be a ceramic or metal, it may be non-metallic particles, for instance a stable oxide, carbide of metal, etc. added with the copper metal matrix. The benefits of the reinforced particles it work as a strengtheners, which depends on size of particles (finer size is better), particle distribution (good dispersed is preferred), density of particles (high per unit volume will be better), particles gap (closer is better) [2].
Various processing techniques, such as powder metallurgy, infiltration and casting methods have been grown and are being utilized for preparing the metal matrix composites [3]. Still, the powder metallurgy technique has an edge over liquid-processing technique because it succeed to deal with problems of non-uniform distribution of added reinforced particles, porosity, and useless chemical reactions, which are a chunk and package of the casting technique. The powder metallurgy technique also used for the fabrication of better products, specially, when the ceramic are used to reinforced into the metal matrix [4]. The problem of agglomeration of reinforcement particles in the metal matrix, specially, in order to deal with small-amount reinforcement particulates, it can be avoided by mechanical mixing that has repeated deformation, cold welding, and fragmentation of powder with the help of high-energy ball milling and that's lead to a homogeneous distribution of the reinforced particles in the metal matrix [5].

In previous research, various type of ceramic particles were reinforced in copper to improve their wear and mechanical characteristic [6]. R. Sathiskumar et al, B4C particulate reinforced in copper matrix composite and get 26% more hardness than copper matrix. Issac Dinaharan et al, (SiC, Al2O3, B4C and TiC) particles were reinforced in copper matrix and microstructure, micro-hardness evaluated, and Cu/B4C CMC exhibited superior hardness. S.Harish et al, TiC reinforced particles reinforced in copper metal matrix by situ technique, composite exhibit a remarkable improvement in ultimate tensile strength and hardness compared with verging copper. Among these additives, TiC is more appealing because of it has better hardness, high modulus, and melting temperature [7]. TiC particles used as an additive with copper based composite because of its various industrial application in motor, switches, and electrode, etc [8]. In composite, Ni is act as a binding agent between TiC and Cu particles [9]. Few study were conducted to understanding the wear and friction properties of TiC reinforced with Cu-based composites. The aim of study is to examine the effects of Ni (act as binding agent) in composites having 5wt%Ni and 10wt%Ni and also include the effects of TiC reinforced in meta matrix by the help of wear and friction behavior of the synthesized composites, the wear test performed under boundary lubrication with 0.2wt% MoS2 (Nanopowder). Sliding wear test used aбал-on-disk tribometer at different loads of 30N, 60N, 90N, and 120 N and kept at constant sliding speed of 0.25 m/s. The equally investigation were made for study the wear mechanisms of metal matrix composites.

2. Experimental procedures

2.1. Material and sample fabrication

Copper powder and Nickel was used and has an average particle size of 200 mesh. TiC was used with an average grain size of 80nm. Different weight percentage of TiC (0%, 3%, 6%, and 9%) reinforce in Cu-5Ni and Cu-10Ni metal matrix. These composites were denoted as Cu5Ni, Cu5Ni-3TiC, Cu5Ni-6TiC, Cu5Ni-9TiC, and Cu10Ni, Cu10Ni-3TiC, Cu10Ni-6TiC, Cu10Ni-9TiC respectively. Formulated composition of powders was mixed in a high-energy ball mill at speed of 200rpm for 180min. Mixing of powders was done in presence of ethanol to avoid the formation of intermetallic compounds and the ball-to-powder ratio was 10:1. The dry mixed powder was compacted by pressing in a cylindrical die at an optimized load of 465KN (650MPa). Green compacted sample were sintered at sintering temperature 900°C in argon atmosphere and for soaking time of 60 min. Table 1 shows the batch identification of synthesized specimen of Cu5Ni and Cu10Ni metal matrix composites and percentage of TiC reinforced. Figure 1 shows the flow chart of fabrication process of specimen throw the root of powder metallurgy.
Table 1: Details of batch identification of Cu5Ni and Cu10Ni metal matrix and composites.

| SI no. | Batch id | Copper based MMCs | Percentage Of reinforced TiC nanopowder | Final Composition of powder |
|--------|----------|--------------------|----------------------------------------|-----------------------------|
| 1      | C11, C21 | Cu-5,10wt%Ni       | 0%                                     | Cu5wt%Ni, Cu10wt%Ni         |
| 2      | C12, C22 | Cu-5,10wt%Ni       | 03%                                    | Cu5wt%Ni-3TiC, Cu10wt%Ni-3TiC |
| 3      | C13, C23 | Cu-5,10wt%Ni       | 06%                                    | Cu5wt%Ni-6TiC, Cu10wt%Ni-6TiC |
| 4      | C14, C24 | Cu-5,10wt%Ni       | 09%                                    | Cu5wt%Ni-9TiC, Cu10wt%Ni-9TiC |

2.2. Characterization

The morphology and microstructure of metal matrix and composites was illustrated by SEM images. The analysis was also conducted by energy-dispersive X-ray spectrometer (EDS), the presence of different elements in the sintered samples was confirmed by taking EDS analysis. From SEM image it can be seen that the uniform distribution of TiC particles thorough the metal matrix. EDS, which was carried out at three different spots marked as spectrum 1-3, which shows in figure 2(a) and 2(b), the presence of all the constituents mixed with the base material Copper.

Figure 1: Flow chart for experimental procedure.
2.3. Density and hardness
Bulk density of sintered samples was measured by using well known Archimedes’ method. It becomes necessary to calculate relative density, which actually tells how much densification is achieved. Relative density is the ratio of the bulk density to theoretical density. Theoretical density of composite is calculated by rule of mixtures, based on the starting composition, assuming no reaction and no impurities in the sintered materials (ASTM, 2002).
Hardness tests were carried out using Vickers indenter. HV were measured to study influence of indentation load on the hardness values. The indentation load was 50 g (0.98 N) and indentation time was varied from 5s, 8s, 10s, 12s seconds. The hardness reported here was the mean value of five times tested.

2.4. Friction and Wear tests
The wear and friction tests were carried out under boundary lubrication conditions by ball-on-Disc test. The ball material chosen for testing is of EN8 mild steel, hardness (HV) 4.31 GPa, and surface roughness (Ra 0.01 μm). EN8 is widely used for many general engineering applications as shafts, studs, bolts etc. Nano lubricant made by mixing of Gear oil with 0.2wt.%MoS2 by the help probe sonicator for six hours. The wear loss was weighed by an electronic balance having accuracy of ±0.0001 g. Before each test, the samples were polished (mirror like finish) using various grades of abrasive papers, diamond paste of sizes 0.25μm and then cleaned with acetone followed by drying. Each specimen (Ra 0.30 μm) was weighed before and after the test. All wear and friction tests were performed three times to insure the repeatability.
of test data at the same experimental condition. Wear of disc and ball were calculated using Archard equation.

The specific wear coefficient is calculated as

\[ K_w = \frac{W_v}{F_n \times S_d} \text{ mm}^3\text{N}^{-1}\text{m}^{-1} \] (1)

Where \( W_v \) is wear volume, \( F_n \) is normal load and \( S_d \) is sliding distance.

The operating parameters for measurement of friction and wear were as follows:
- Normal load(s) (\( F_n \)) = 30 N, 60 N, 90 N and 120 N
- Sliding velocity = 0.0.25 m/s
- Sliding distance (\( S_d \)) = 200, 400, 600, 800 m
- Temperature (T) = 25\degree C and
- Relative humidity (RH) = 45 \%

2.5. Boundary lubrication condition

The following equation was used to determine whether the lubrication was under boundary lubrication or not:

\[ h_{\text{min}} = 7.43R \left( 1 - 0.85 \frac{1}{g_{1857}} + \frac{1}{g_{2879}} + \frac{1}{g_{2868}} + \frac{1}{g_{2871}} + \frac{1}{g_{2869}} + \frac{1}{g_{3038}} \right)(\eta \mu / E^* R)^{0.65} \left( L / R^2 E^* \right)^{-0.21} \] (2)

Where \( K \) is the elliptical parameter, \( R \) the composites radius (m), \( \eta \) absolute viscosity (Pa s), \( u \) the sliding velocity (m/s), \( E^* \) the composites elasticity modulus (Pa) and \( L \) the load (N)

\[ \frac{1}{E^*} = \frac{1}{E_a} + \frac{1}{E_b} \] (3)

\[ \frac{1}{R} = \frac{1}{R_a} + \frac{1}{R_b} \] (4)

\[ \lambda = \frac{h_{\text{min}}}{\sigma^*} \] (5)

\[ \sigma^* = \sqrt{(\sigma_a^2 + \sigma_b^2)} \] (6)

Where,
- \( R_a \) is the radius of ball, \( R_b \) is the radius of Flat disc (Infinite), \( k \) is the ellipticity parameter (2); \( \eta \) is the viscosity of oil (Pa s), \( u \) is the mean velocity (m/s), \( \nu_a \) is the Poisson’s ratio of steel ball; \( \nu_b \) is the Poisson’s ratio of disk; \( E_a \) is the elasticity modulus of ball; \( E_b \) is the elasticity modulus of disk; \( L \) is the normal load

For boundary lubrication the value of \( \lambda \) should be less than 1.

3. Result and discussion

3.1. Density and micro-hardness

The Effect of TiC reinforced on density shown in figure 3(a) and 3(b) of metal matrix. The density of composites increased; when percentage of TiC reinforced increasing from 0 to 3wt%, at 3wt% of TiC particles composites show the optimum value of sintered density, sample C12 has higher density of 8.001 gm/cm\(^3\). However, sample C22 has maximum value of density 8.036 gm/cm\(^3\). Afterward, density started
decreasing with the increased quantity of TiC particles: it may be due to the agglomeration of TiC particles in Cu-Ni metal matrix, which may contribute to the pore formation and lead to reduced in density of composites [10]. The composites C12 and C22 have approx same density: because Copper and Nickel have same density.

![Figure 3. Effect of TiC on density (a) Cu5Ni (b) Cu10Ni.](image)

![Figure 4. Effect of TiC on hardness (a) Cu5Ni (b) (Cu10N).](image)

The figure 4(a) and 4(b) shows the micro-hardness verses TiC reinforced on metal matrix. Micro-hardness as composite increased with the increase in the percentage of TiC reinforced particiles, till 3wt% of TiC. It may be due to the dispersion strengthening consequence of TiC particles. The thermal imbalance between the reinforce TiC particles and the metal matrix, which cause the internal stresses that generate dislocations, that’s lead to increase the dislocation density, which eventually contributed to the increment of the micro-hardness of composites [11]. Relatively higher value of micro-hardness were obtain at 3wt% of TiC, with a micro-hardness value of 112 (HV) of composite C12 and 117 (HV) for composite C22.. After 3 wt.% of the TiC particles, the micro-hardness of composites started decreasing, that’s the cause of increased in tendency of agglomeration of the TiC particle. It may also reduce the load carrying capacity of TiC. Another reason could be increase in gap of Cu particles that destructive affected the micro-hardness [12]. The composite C22 has 4.5 percent more micro-hardness in comparison to composites C12. It because of nickel has better hardnes than copper.

3.2 Friction and Wear behavior
3.2.1. Effect of loads on COF

Figure 5(a) and 5(b) represent the coefficient of friction of Cu5Ni and Cu10Ni metal matrix and its composites, at various normal loads of (30N, 60N, 90N and 120N) and constant sliding speed of 0.25m/s. It is clear that the friction coefficient of composites decreased with increasing the load on specimen. It’s may be due to the presence of nano MoS₂ in lubricant, which provide smooth layer between counter face and specimen. However, the coefficient of friction for the Cu5Ni metal matrix increased as the load increased from 30N to 60N for further loads its starts decreasing with loads. While Cu10Ni metal matrix shown that Coefficient of friction increased when the load increased from 30N to 60N, other composites of Cu10wt%Ni metal matrix decreasing coefficient of friction when the load increasing.

![Figure 5. Cof vs load at 800m (a) Cu5Ni (b) Cu10N.](image)

3.2.2. Effect of sliding distance on Wear rate

Figures 6(a) and 6(b) shows were rate of the Cu5Ni and Cu10Ni metal matrix’s composites at different sliding distance of (200m, 400m, 600m, and 800m) with fixed load of 120N and constant sliding speed of 0.25m/s. The wear rate of virgin samples C12 and C22 have higher than composites at every sliding distance, because of the metal matrixes have less micro-hardness than it’s composites. As the sliding distance increases wear rate of composites decreases. It is because of as sliding distances increases the TiC particles coming out from composites and it provide shield to the specimen, which reduce the wear rate of composites. The wear rate of metal matrix decreases with increasing the TiC reinforced particles. It observed that, at 3wt% of TiC, the wear rate was low at every sliding distance. Composite C12 has lowest wear rate (0.3x10⁻⁴mm³N⁻¹m⁻¹) sliding distance of 800m. But composite C22 has wear rate of (0.2x10⁻⁴mm³N⁻¹m⁻¹) at same sliding distance. The composites C22 has 33% low wear rate than the composite C12, Because of C22 composite has more binding agent (Ni), which not allow to the TiC particles to come out from specimen during sliding wear behavior test.
3.2.3. Effect of loads on Wear rate

Meanwhile, Figure 7(a) and 7(b) show the wear rates of Cu5Ni and Cu10Ni metal matrix and its composites at different loads of (30N, 60N, 90N, and 120N) and fixed sliding distance of 800m, with constant sliding speed of 0.25m/s. The wear rate of composites was very low than virgin sample at load 120N. Wear rate increases with increase in loads. Moreover, wear rate was found maximum at the load 120N. In figure 9(a) the composites C12 has low wear rate than other composites and metal matrix at every applied normal loads. In figure 6(b) the C22 composites has less wear rate than virgin sample and other composites at every condition on normal loads, which due to credited of higher relatively micro-hardness of the composites than metal matrix and composites. If we compare the wear rate of composite C12 to the composite C22 at every normal load, then C22 has less wear rate than C12. Because of more amount of binding agent leads strong binding between copper and the TiC particle.
3.3. Worn surface analysis

The SEM image of worn surface has oxide layer and containing counter material element, which transferred from the counter body. The range of cover given by this layer can explain the non-appearance of the wear tracks. However, some deep groves are shown in micrograph. The layer occupied a metal to metal junction and supplied a low shearing strength at the contact, which leads to reduced in friction coefficient in metal matrix and their composites. The composite C22 shows few fine lines covered by the oxide layer and by wear debris at few points along with TiC particle, which pulled-out during sliding at some locations. The existence of Fe in the EDS shows that the transfer of metal took place from counter body to specimen. The existence of oxygen in the EDS result shows the probability of oxidation that may have come out during the sliding process.

4. Conclusions

1) The density of the composite increases with an increase in TiC reinforced in metal matrix till 3wt% of TiC, after that, it started decreasing. Composite C22 has best density of 8.036gm/cm³.
2) The micro-hardness increased with increase TiC reinforced in metal matrix, till 3wt% of TiC, beyond this it start decreasing. Sample C22 shows the best micro-hardness of 117HV.
3) The wear rate of composites and the Cu5Ni & Cu10Ni metal matrix increases with applied normal load. While the composites have less wear rate as compared to the metal matrix shows the effect of higher value of micro-hardness of composite.
4) The 3wt%TiC additions showed better results in terms of wear rate and coefficient of friction.
5) The sample C22 has low wear rate than C12, this is may be due to C22 has higher micro-hardness and ability to hold TiC particles towards specimen.
6) The average friction coefficient decreases with the increasing load, because of MoS₂ in lubricant, which provide smooth film.
7) The wear mechanism of the C22 and C12 composite was a combination of oxidative wear, adhesive and mainly abrasive wear found in investigation.

Reference

[1] Tyler D E, Black W T 1990 ASM International, Metals Park 216-240.
[2] Glidop 1994 SCM Metals Products, Research Triangle Park.
[3] Liu R, Song K, Jia S, Xu X, Gao J, Guo X 2008 Chinese Journal of Aeronautics 21(3) 281-288.
[4] Zhou G, Ding H, Zhang Y, Hui D, Liu A 2009 *Metalurgija – Journal of Metallurgy MJoM* 15(3) 169-179.

[5] Soleimanpour A M, Abachi P, Purazrang K 2009 *Tribology – Materials, Surfaces & Interfaces* 3(3), 125-131.

[6] Fathy A, Shehata F, Abdelhameed M, Elmahdy 2012 *Mater Des* 36 100–107.

[7] Li L, Wong Y S, Fuh J Y H, Lu L J 2001 *Mater Proces Techn* 113 563–567.

[8] Rathod S, Sharma M, Modi O P, Khare A K, Prasad B K 2013 *Inter J Mater Res* 104 666–674

[9] Rajkumar K, Aravindan S. 2011 *Tribo Inter* 44 347–358.

[10] Lee D W, Ha G H, Kim B K 2001 Sci Mater 44 2137–2140.

[11] Nemati N, Khosroshahi R, Emamy M, Zolriasatein A 2011 *Mater Des* 32 3718–3729.

[12] Arsenault R J, Shi N 1986 *Mater Sci Eng* 81 175–187.