Mechanical and tribological properties of composite made of marble dust-reinforced C93200 copper alloy

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Abstract
Composite materials are unique because reveal great physical, mechanical and thermal properties. However, there is yet huge potential to enhance their features by adding specific reinforcement in the matrix in order to reach the requirements of a particular application. This paper presents detailed research on the impact of marble dust reinforcement to mechanical and tribological features of copper based metal alloy (C93200 series) composites dedicated for bearing applications. The novel composites made with marble dust reinforcement (1.5 to 6.0 wt%) were manufactured using the liquid metal stir casting technique. A micro-hardness tester and universal testing machine (Instron-5967) were used to obtain the mechanical properties. While, the POD tribometer was engaged to detect the wear features by simulating various operating conditions by setting the temperature constant (35 °C). The Scanning Electron Microscope (SEM) was used to investigate the wear mechanisms produced at the hard contact between the composites with different marble content against an EN-31 hardened steel disc. The data gathered in this research proves an improvement in the mechanical properties, especially for a higher reinforcement ratio of novel composite, in respect to the matrix alloy. Furthermore, the novel marble dust reinforced composites reveal much better wear resistance in respect to un-reinforced composite that make it suitable for bearing application.

1. Introduction

Copper and copper based alloys (i.e. brass, bronze etc) are known as engineering materials used in large amount of friction-prone components. They are parts of the machines such as bushing, liners, bearing, sleeves etc due to their good thermal conductivity, corrosion resistance and self-lubricating properties [1, 2]. The C93200 is a commercial copper based alloy used widely as bearing material. This material owns multiple properties from which the most important are lower coefficient of friction, higher load carrying capacity, good thermal conductivity, and greater resistance to wear along good corrosion performances. However, these features can entail some challenges during long terms durability and wear activity when are used in bearing [3, 4]. Journal or plain bearing is a machine element that supports and radially positions a rotating part known as shaft. Therefore, the performance and efficiency of bearing can influence the functionality of the systems/mechanisms. The bearing materials must be carefully selected, in order to make these systems/mechanisms successfully that meet the desired performance efficiently. Rolling contact bearings offer the lower friction as compared to sliding contact bearings. However, in some cases, the use of sliding contact bearing cannot be replaced by another bearing because they have their specific benefits and are frequently in use [5]. Journal bearings are employed in industrial machineries, engines, automobile industries, turbines (steam, hydraulic and gas), electric generators, compressors and/or other machineries of oil and petrochemical industries [6]. The research trend is to replace the most widely used conventional bearing materials with novel materials that have superior properties in respect to the conventional bearing materials [7].
Table 1. Matrix alloy (wt%) chemical composition.

| Elements | Cu | Sn | Pb | Zn | Ni | Fe | Sb | Si |
|----------|----|----|----|----|----|----|----|----|
| Content  | Bal| 7.0| 6  | 2  | 1  | 0.2| 0.35| 0.005 |

Nowadays, ceramic reinforced composites (i.e. Al₂O₃, SiC, MgO along whiskers/fibers) are developed in order to enhance the main mechanical features and/or tribological characteristics of a ‘parent’ material [8]. Poddar et al [9] studied the microstructure evolution associated to mechanical properties of SiC as reinforcement for AZ91D (a composite based of magnesium) composites synthesized through rheocasting routine. There, the SiC presence improves the young’s modulus and yield strength. Yu et al [10] evaluated the reinforcement size over mechanical properties developed by the SiCp/5210 Al as composite. They indicate an improvement for the bending strength of the composites by decreasing reinforcement size. Abdizadeh et al [11] investigated the mechanical behaviour of zircon reinforced composites, in which was observed that the rise in zircon content and sintering temperature allows to increase the composite hardness. Yilmaz et al [12] worked on Cu-10Sn matrix composites and reported superior mechanical properties by addition of filler materials but the toughness of composites is adversely affected. Gangwar et al [13] developed quick lime (CaO) reinforced silicon-bronze alloy composites vacuum casting technique. They observation indicates the wear resistance increases considerable in respect to the base alloy. On the other hand, the mechanical properties developed by a cast composite may substantially enhance when the weight percentage of quick lime particulates is changed. The volume/weight percentage of ceramic plays a vital role for improving the mechanical as well as tribological features of materials made as composite.

The literature survey proves that particulate filled metal alloy composites offer superior properties in respect to monolithic materials, which impose a continuum research development to identify the best combination between different matrix and filler materials. Recently, Cu-BN [14], Cu-MoS2 [15], Cu-Sn-Fe₃Al [16], Cu-CNT [17], Cu-graphene [18] have been the most studied composite materials. However, the studies about copper based alloy (C93200) are very poor, especially, on the marble dust (CaCO₃) reinforced composites. In this respect, this research work is dedicated to manufacture the marble dust (CaCO₃) reinforced copper based alloy (C93200) composites that enable superior mechanical and tribological feature for materials used in bearing applications. In previous studies, copper based composites were obtained mostly by powder metallurgy [12] that presents some weakness in terms of mechanical properties. Here, we propose a better alternative by manufacturing the copper based (C93200) alloy composites through the liquid metal stir casting technique in which were added vary amount of filler contents (0, 1.5, 3, 4.5 and 6.0 percent by weight). The novel manufactured composites were then evaluated in terms of mechanical properties and wear performance. Further, the post mortem samples were investigated by an advanced Electron Microscopy based on Field Emission (FESEM) and x-ray diffraction (XRD) technique in order to elucidate the wear mechanism, structural crystallography and reinforcement status. The outcomes achieved are promising and they can be used as a new platform to enhance the performances of bearing composite for automotive applications.

2. Materials and methods

2.1. Test Materials and composite manufacturing

The commercial grade of C93200 copper alloy were procured from Mittal Industries (Jaipur, India) in rod form, from which were generated the marble dust as powder form with the size of < 44 μm. Table 1 offers the detail regarding elemental composition of C93200 alloy. The marble dust contains following elements in wt%: LoI (40.84), CaO (32.23), MgO (18.94), SiO₂ (4.99), and the rest of three is formed by different oxides (i.e. K₂O, Fe₂O₃, Al₂O₃, N₂O and SO₃). Marble dust (CaCO₃) reinforced copper (C93200) alloy composites were developed by stir casting process. Before starting the casting routine, both C93200 alloy and marble dust were preheated separately up to 400 °C. A graphite crucible linked with an induction furnace, was used to further heat up the preheated C93200 alloy. When the liquid temperature (1150 °C) of C93200 alloy was achieved, the required quantity of preheated reinforcement (0, 1.5, 3, 4.5, 6 wt%) was slowly poured into the molten metal. This composition was continuum mixed to obtain a homogenous composition by a mechanical stirrer. This routine were made for 1–2 min by applying a rotation of 300 rpm constant speed that also promote a proper bonding between the ceramic reinforcement and matrix alloy. In this routine, magnesium in a small quantity (2 wt%) was added which enable the superior wettability between reinforcements and melt alloy matrix.

Wettability is the ability of a liquid metal to spread over solid reinforcement particles. Several approaches have been used to promote the wettability of a liquid matrix alloy with reinforcement particles, decreasing the surface tension of liquid metal is one of them. Magnesium (Mg) acts like a surfactant element, which has been used
widely as well as wetting agent to promote the wettability of different ceramic particles, such as SiC and mica [19, 20]. The obtained mixed melt was discharged into the graphite permanent mould (size 100 × 65 × 10 mm³), and then followed by air cooling for about 15 min. After solidification, the samples were prepared for physical, mechanical and tribological characterization as per ASTM standards.

2.2. Physical and mechanical studies
The composites densities were evaluated by a standard immersion process, in which the material is immersed in a classical manner in a fluid. As per ASTM standard this is so-called Archimedes method, whereas the theoretical densities were calculated through the mixture formula [21] introduced in (1).

\[ \rho_{th} = \frac{1}{\frac{w_m}{\rho_m} + \frac{w_{wm}}{\rho_{wm}}} \]  

(1)

Where, \( \rho_{th} \) and \( \rho_{in} \) represent base alloy and marble densities, respectively; \( w_m \) and \( w_{wm} \) represent base alloy and marble weight fraction, respectively. Here, the value of void fraction \( (v_f) \) or amount of porosity of manufactured composites samples is deduced by applying the following equation (2).

\[ v_f = \frac{\rho_{in} - \rho_{exp}}{\rho_{in}} \]  

(2)

The composite micro hardness was measured under a Vickers-hardness tester device considering scale ‘C’. This was performed on a Walter Uhl testing machine as per ASTM E92 standards [22] collecting the average of five test trials. An applied load of 100gf per capita unity was imposed with a 5 s period.

The mechanical characteristics (i.e. tensile, compression and flexural strength, respectively) were determined under an INSTRON-5967 testing machine. Here, the performances in terms of tensile strength were measured accordingly to the ASTM E8 standard [23]. A nominal value of 2.0 mm min⁻¹ was used as crosshead speed. Each type of composition was tested at least five times in order to prove the repeatability and average value of measurement is reported. The compression strength of specimens having dimension of 25 mm × 10 mm × 10 mm were evaluated by applying a displacement control, a value of 2 mm min⁻¹, as per ASTM E9–09 standard [24]. Each trial was repeated at least three times in order to obtain a consistent mean. Material flexural strength with a nominal dimension of 60 × 10 × 10 mm³ were measured under a UTM-Instron-5967, it had a span length = 40 mm and was activated by a displacement speed of 1 mm min⁻¹ in conformity to the ASTM-E290 [25] standard. As earlier indicated each trial was repeated at least three times in order to obtain a consistent mean value.

2.3. Wear measurements
A pin-on-disc type tribometer model TR 20, made Ducom was used to simulate the wear activity under dry sliding condition. It allows simulate/record the wear performances in accordance to ASTM G99 standard [26] in ambient conditions. The tribometer used and depicted in figure 1 contain a pin as a holder, steel disc made of EN-31 hardened. The AC motor stimulates this device while the mechanical contact is established through a dead weight. The loading applied on the pin, made of composite, push the pin against the disc, which rotate continuously and is used as counter-face. The hardness measured from the disc steel was around 60–70 HRC while its surface roughness was ~0.6 μm. Before and after running the test, the weight of pins was evaluated using an electronic micro balance with high accuracy (± 0.001 mg). The simulated wear performances were conducted by varying the load (5, 10, 15, 20 and 25 N) and speed of sliding (1.57, 2.61, 3.65, 4.69 and 5.73 m s⁻¹) for a track radius of 50 mm. The amount of mass loss \( (\Delta m) \) during wear testes was determined by making the difference of initial pin weight against the final weight. Mathematically, the following relation describes the wear loss:

\[ \Delta m = W_1 - W_2 \]  

(3)

Where, \( W_1 \) and \( W_2 \) represent the pin weight prior and after wear test of composites and \( \Delta m \) represents the total mass loss.

The frictional forces for each individual test run were measured by using load cell attached in the setup, and the coefficient of friction were captured through a specific WINDCOM 2010 software acquired from DUCOM Pvt. Ltd. At the end, the mean value was considered.

2.4. Experimental design by Taguchi method (TM)
The main purpose of Taguchi experimental design is to reduce the number of iterations. Further, it allows to obtain the optimal results efficiently. This powerful tool analysis was used here to simulate experimental runs through the Minitab 16 software. In conventional full factorial technique, it needs \( 5^4 = 625 \) trials to study four input control variables. Table 2 presents each of the five levels. The Taguchi’s experimental design permits to
transforms it into only 25 trials (using orthogonal array L$_{25}$). The additional benefit of TM is to reduce the cost and experimental trials. The data gathered from experimental results are then converted as signal to noise ($S/N$) ratio. The performance characteristics were adopted as ‘smaller the better’ in order to interpret the wear progress. Mathematically, equation (4) describes the $S/N$ ratio [27].

$$\frac{S}{N} = -10 \log \left[ \frac{1}{n} \sum_{i=1}^{n} y_i^2 \right] \tag{4}$$

Here, $y_i$ denotes data observed i.e. wear loss, number of observations, $n$. The model proposed here enables a reduced sliding wear loss. Further, to validate the results statistically, in turn to obtain superior performances of output parameters (from the given input parameters) were implemented the analysis of variance (ANOVA).

2.5. Micro-morphological wear characterization

The fabricated samples were submitted to cleaning process by acetone followed by air dry. An advanced FESEM connected to EDAX (FEI NOVA NANO 450) were used to study the microstructure arrangement, phases constituent, and wear mechanisms (pattern) caused by the wear trails. The Panalytical X Pert Pro x-ray diffractometer were used for examination the entire region of the specimen (of marble dust compounds ($\text{CaCO}_3$)).

3. Results and discussion

3.1. Microstructure and phase analysis of materials

The XRD analysis was conducted to detect the amount of marble dust ($\text{CaCO}_3$) particulates (major constituents $\text{CaO}, \text{MgO}, \text{SiO}_2$) on each composite fabricated by liquid metal stir-casting technique as shown in figure 2. It was

| Table 2. Parameters and imposed levels in this research. |
|--------------------------------------------------------|
| Control Factor | Level | Units |
|----------------|------|-------|
| A: Normal Load | I: 5  | N    |
|                | II: 10 |      |
|                | III: 15 |     |
|                | IV: 20  |    |
|                | V: 25   |    |
| B: Sliding velocity | 1.57 | m s$^{-1}$ |
|                | II: 2.61 |   |
|                | III: 3.65 |  |
|                | IV: 4.69 |   |
|                | V: 5.73  |    |
| C: Filler content | 0 | wt% |
|                | II: 1.5 |    |
|                | III: 3  |    |
|                | IV: 4.5 |    |
|                | V: 6    |    |
| D: Sliding distance | 500 | m |
|                | II: 900 |   |
|                | III: 1300 | |
|                | IV: 1700 |   |
|                | V: 2100 |    |

Figure 1. (a) Pin-on-disc tribometer configuration; (b) pin against disc arrangement.
observed that for zero weight percentage of marble (CaCO₃) particulates only the peaks of matrix alloy elements (Cu, Pb, Sn, and Zn) may appear. Whereas, in the composites having 1.5 to 6.0 weight percentage, the XRD analysis (see details in figure 2) indicates the presence of strong peaks of Ca, Cu, Zn, CuSiO₃ and Cu₂PbSiS₄ and weak peaks of CaCO₃, Sn, Pb, CuMgSiO₃O₆, and Ca₂CuO₃. Moreover, several other phases in all the composites such as FCC (111) (d = 2.087Å), Cu, FCC (111) (d = 2.855Å), Pb, a tetragonal (101) (d = 2.571Å), a cubic (220) (d = 2.288Å) and (110) (d = 2.324Å) Sn and Hexagonal (102) (d = 1.692Å) Zn can be highlighted. In addition, the cubic CuO (200) (d = 2.120Å), cubic Mg₅SiO₄ (112) (d = 2.458Å), BCC (011) Cu₂MgO₃, Ca₂O₃, CuSiO₃, and HCP (104) (d = 3.035Å) CaCO₃ were also detected. SEM micrographs of the microstructure of unreinforced and marble dust reinforced composites are shown in figures 3(a)–(e). It can be seen that the blackish particles so-called marble dust are present uniformly dispersed in the matrix. Moreover, the CaO, SiO₂, MgO and CaCO₃ particles are also observed in the corresponding of EDX results.

3.2. Density and void content characteristics of materials
Table 3 presents the experimental results of density (∼8.84–7.63 g cm⁻³) as well as the calculated theoretical density (∼8.91–7.75 g cm⁻³) of the unreinforced and reinforced composites. From this table it can be seen that a very small fluctuation on density of fabricated composites that varies in a small range from 8.84 to 7.63 g cm⁻³. The composite containing 6 wt% of marble dust had the minimum value of density whereas the density of
Figure 3. SEM micrographs and corresponding EDS results of (a) Pure Copper alloy (C93200), (b) Composite-1 (C93200-1.5 wt% marble dust), (c) Composite-2 (C93200-3wt% marble dust), (d) Composite-3 (C93200-4.5 wt% marble dust), (e) Composite-4 (C93200-6wt% marble dust).
composite with 0 wt% of marble dust was the maximum. It indicates the density diminished with the increase of marble dust content. As can be seen in table 3, the void content reduces from 0.785% to 0.497% for 0 wt% to 4.5 wt% of marble dust, but when the marble dust is formulated with highest weight percentage (6 wt%) the void content increases up to 1.548%. The reason of dropping in void content is related to superior wettability, which promote the nucleation behaviour and crystal growth resulting into lower shrinkage porosity [13].

### Table 3. Comparison of theoretical and experimental density.

| S.No. | Composition          | Theoretical density calculated (g cm⁻³) | Measured density (g cm⁻³) | Void Content (%) |
|-------|----------------------|----------------------------------------|---------------------------|------------------|
| 1     | 0 wt% marble dust + C93200 alloy | 8.91                                   | 8.84                       | 0.785            |
| 2     | 1.5 wt% marble dust + C93200 alloy | 8.61                                   | 8.55                       | 0.696            |
| 3     | 3 wt% marble dust + C93200 alloy  | 8.31                                   | 8.26                       | 0.601            |
| 4     | 4.5 wt% marble dust + C93200 alloy | 8.04                                   | 8.0                        | 0.497            |
| 5     | 6 wt% marble dust + C93200 alloy  | 7.75                                   | 7.63                       | 1.548            |

3.3. Mechanical properties

3.3.1. Micro-hardness

The composites hardness enhanced by the increase of the marble dust (CaCO₃) contents because the reinforced particles are harder in respect to the base matrix alloy. Such as, the value of hardness of novel composites increases from 115.49 Hv to 128.97 Hv when was added different amount of marble dust content, from 0 to 4.5 weight percentage, details provided in figure 4. However, by adding further amount of marble dust content i.e. 6.0wt-%, the composite hardness reduces to 120.94 Hv. The hardness measurement on the unreinforced matrix alloy confirms the lower hardness value as compared to reinforced composites. The reason for increase in hardness is the presence of CaO, SiO₂, MgO and Al₂O₃ in the chemical made up of particles and lower void content [8]. Reinforcement arrests the dislocation movement when the load is applied, resulting in growth in hardness. This result is in agreement with work done by Atuanya [28] and Hassan [29]. Further, when the weight percentage of marble dust increases at its maximum, there is a possibility of agglomeration of marble dust particle which reduces the interfacial bonding between reinforcement and matrix material that generate a decrease of hardness value for the composites. This result is in agreement with the work done by Swati Gangwar [13] and Shanmughasundaram [30].

3.3.2. Tensile strength

Figure 4 shows the variations in the tensile strength for both unreinforced and marble dust reinforced composites. It was detected that, the composites tensile strength enhances when is used a higher weight percent of marble dust. There, the maximum value of tensile strength (278.99 MPa) was achieved at 4.5 weight percentage of marble dust. The most probable reason for this enhancement of tensile strength is attributed to hard particulates that can resist better to a higher load when are uniformly distributed in the matrix phase. Here,
the applied load may get transferred to the embedded hard particle which improves the loading capacity for the fabricated composites. However, the tensile strength of composites decreases in case of marble dust content more than 4.5 weight percentage. This decrease in tensile strength may be attributed to segregation of marble dust particulates at higher weight percentage of marble dust. The segregation may generate accumulation of damage-density that can activate a premature composite fracture. In addition, the higher weight percentage of marble dust content can produce an increase of brittleness for the composites that result in a reduced tensile strength of composites.

3.3.3. Flexural strength
Components made of composite dedicated to automotive/structural application may suffer in terms of bending performances. In this respect we have dedicate attention to improve this aspect as well. Figure 3 presents details of composite fabricated flexural strength. The result from figure 4 indicates an increase of flexural strength of novel composite when the amount of marble dust is increased. The maximum flexural strength (413.34 MPa) were achieved by adding 4.5 wt% marble dust on the composite. But, by a further increase in the marble dust weight percentage (6 wt%) it was noted a slightly decreases in the flexural strength of composites. The casting defects such as void content, micro and macro cracks, some marble clustering formed from dust particulates along relative low interface bonding may represent potential cause of decreasing flexural strength of excessive (6 wt%) marble dust filled C93200 alloy composites. A similar study, reported by Reddy and Zitoun for alumina filled 7072Al aluminum alloy composites indicates the same trend in flexural strength.

3.3.4. Compressive strength
Figure 4 presents details regarding the compressive strength evolution of marble dust reinforced composites. As previously noted for the flexural strength, a relationship between compressive strength and percentage amount of marble dust exist also here, that seems the compressive strength increase proportionally with marble dust content added. Therefore, a higher compressive strength (40.60 MPa) was achieved by adding 3 wt% of marble dust. Once the marble dust weight percentage was increased further (6 wt%) to the matrix alloy were noted a reduction of composite compressive strength. This later behaviour is attributed to the segregation of marble dust particulates. A similar observation were reported by Jayalakshmi et al, when was studied Mg particle filled Ni60Nb40 alloy composite. They found that the composite have higher strength as compared to pure Mg alloy; it can increases up to 85% by adding ∼5 wt% of filler particles, however, when the filler is ∼10 wt% no further enhancement is noted.

3.4. Wear assessment
3.4.1. Effect of marble dust on wear loss at various sliding velocity
The wear behaviour of unreinforced C93200 copper alloy and marble dust reinforced composites with respect to different sliding velocities is reported in figure 5. The wear trials were conducted for each pin containing different amount of marble dust (0, 1.5, 3, 4.5, and 6 wt%) on a POD tribometer. Here, the tribo-routines were performed by imposing different sliding velocities (1.57, 2.61, 3.65, 4.69, 5.75 m s$^{-1}$) whereas a fixed sliding distance (1300 m) and a normal load (15 N) were settled, respectively. During wear sliding trials some important parameter were analysed carefully, such as, the weight percentage of filler material, geometry or size of filler
material, particles distribution and morphology of composites surface. These parameters can be used to calculate the wear loss of unfilled matrix alloy and reinforced alloy composites. Hence, they are suitable indicators to understand the performances of matrix alloy and reinforced alloy composites materials [13]. From figure 5 it can be seen that wear resistance of manufactured composites improved with the addition of marble dust content from 1.5 wt% to 4.5 wt%. However, further addition of marble dust (6 wt%) leads to decrease in wear resistance of composite, this might be the result of large clustered region of marble particle formed during the casting of composites. The decrease in wear loss of composites may be linked to improve in hardness of fabricated composites with the incorporation of harder phase, this wear behaviour with respect to hardness of materials has been proved by Archard [34]. Figure 5 also depict that wear loss is gradually increasing with increase in sliding velocity. At lower sliding velocity less wear loss was observed for all the samples but as sliding velocity increased, increase in wear loss was observed. When samples are subjected to higher sliding velocity and normal load there is a possibility of increase in temperature of composite pin due to frictional heat generated in between two mating surfaces (pin and disc) which results in thermal softening of pin. Hence at higher sliding velocity and normal load the bonding strength between Marble particles and copper matrix alloy decreases due to which hard reinforced particle easily pulled out from matrix alloy. Jin et al [35] carried out dry wear trials on Al matrix composites that contain Mg$_2$B$_2$O$_5$ whisker. They indicate that the wear loss is increased when the sliding velocity was varied for a fixed load of 15 N. The maximum value of wear loss is achieved on the unfilled matrix alloy (see figure 5). It was noted that the wear loss has a similar behaviour as compared with the hardness. Another observation indicates that the wear loss become accentuated when a higher sliding velocity is imposed. From our investigation, the amount of wear loss depends of imposed velocity. A slightly flat variation of wear loss were detected for the average amount of marble dust (4.5 wt%) added to the composite. The clustering and macro segregation are supposed to be responsible for this later behaviour. The mechanisms that drive the reduction of wear loss may be due to the incorporation of hard particulates and good bonding strength at the interface of matrix alloy with the hard particulates, which may promote a higher hardness features and good wear resistance of composites. A summary of wear loss can be stated as: 0 wt%MD > 1.5 wt% MD > 6 wt% MD > 3 wt%MD > 4.5 wt%MD, respectively.

3.4.2. Effect of marble dust on coefficient of friction at various sliding velocity

Figure 6 shows friction coefficient evolution of marble dust (CaCO$_3$) reinforced composites in respect to different sliding velocities (1.57 m s$^{-1}$, 2.61 m s$^{-1}$, 3.65 m s$^{-1}$, 4.69 m s$^{-1}$ and 5.73 m s$^{-1}$) considering a fixed normal load (15 N). The initial rotation speed of the disc (1.57 m s$^{-1}$) reveals friction coefficients with lower values for the majority of composite, no matter of amount of marble dust added as wt%. It can be attributed to formation of an initial thin layer on top of counterface material. Such as, the formed layer as an oxide moisture may works as lubricant for the contacted surface leading to reduced friction coefficients. Gangwar et al [13] developed the CaO filled SnBr alloy composites. Further, they simulated the wear behaviour in dry condition and found the same phenomenon as in the present study. A little longer activity, of dry sliding condition, enable removal of the thin layer. Therefore, the counter surface become smoother when gets in contact with the pin causing higher friction coefficients (specifically at 2.61 m s$^{-1}$ velocity). The tribo process is followed by another friction reduction once the sliding velocity is increased (3.65 m s$^{-1}$) that probably is accompanied by high local temperature at the contacted interface. It permits to soften the matrix alloy and micro-welding of pin material
on the counterface may occurs; which prevents a direct contact between the pin and counterface generating lower friction coefficient. When the sliding velocity attains 4.69 m s\(^{-1}\) the micro-welded of pin material is sheared off (removed) leads again to slightly higher friction coefficients. But, at the maximum speed (5.73 m s\(^{-1}\)) applied in this research the friction coefficient decreases again.

3.4.3. Effect of marble dust on wear loss at various loading conditions

Figure 7 presents the wear behaviour of all the composites by varying the normal load. Here, the wear trials for each pin were carried out on a POD tribometer by varying the normal load (5 N, 10 N, 15 N, 20 N and 25 N) using a fixed distance (1300 m) and the sliding velocity of 3.65 m s\(^{-1}\), respectively. The amount of wear loss was noted to decrease almost proportionally in respect to the addition of marble dust weight percentage. Initially the reinforced composite showed a reduction in the wear loss by increasing the normal load from 5 N to 10 N due to lesser contact area between mating surfaces of pins and disc. Then, the wear loss starts increasing proportionally with the normal load, in the range of 10 N to 25 N. The possible reason for higher wear loss at 25 N normal load is related to physical phenomenon in which the shear forces may increases considerable, therefore, more interface material become removed. Elkady et al[14] evaluated the wear activity of Cu-based boron nitride composite materials by varying normal loading using a sliding speed of 0.2 m s\(^{-1}\). They results showed a gradual increase of wear loss corroborated to normal load variation.

3.4.4. Effect of marble dust on coefficient of friction at various loading condition

Figure 8 presents friction coefficients evolution for all the composites, by imposing different load (5 N, 10 N, 15 N, 20 N and 25 N). At the initial stage (5 N load), due the sharp asperities on the counterface (which acts like multiple cutting tool) that easily tear and penetrate the soft matrix alloy composites were noted the increase of the wear loss and friction coefficient. But when the same pin (novel pin with the same composition) is subjected to 10 N load, using the same distance, the friction coefficients decreases due to less effectiveness of sharp asperities of the counterface. A slightly increase on the load (15 N) generate a little increase in the friction coefficients. This occurs because of hard marble particles get fractured and entrapped between two sliding surfaces. Further, they form three body abrasion which results in removal of material from soft matrix alloy composites surface and increase the friction coefficient. By applying 20 N the temperature between sliding surfaces may increases. This helps to soften the matrix alloy, which can generate adhesion wear (micro-welding on counterface) that results in lower friction coefficients. However, when the maximum load (25 N) was applied were noted a gradual increase on the friction coefficient. This growth in friction coefficient could be related to a specific mixed layer formed at the contacting interface that act as third body material and favour the abrasion process.

3.5. Experimental design by Taguchi approach

The results gathered from the composite experimental trials with reinforcement and without reinforcements are presented in table 4 in terms of wear loss. Apart from this, this table contains details of equivalent signal-to-noise ratio (S/N) detected for each individual trial. By summing up all S/N ration, it was found an average of wear loss about 20.70 dB. Figure 9 presents the plot for respective responses in relationship to the control factors simulated on marble dust reinforced composites. There, the inclination angle line is an indicator of significance.
The higher inclination endorses a stronger significance; instead, lower angle proves a reduced importance. Such as, the normal load, sliding distance and filler content are more significant in respect to the other factor.

3.6. Analysis of variances (ANOVA)

By using ANOVA simulation were possible to detect statistically the design parameters that influence the output responses i.e. wear loss. The wear loss results simulated by ANOVA were introduced in table 5. The ANOVA simulation was performed applying 5% as significance level with 95% confidence level. As expected, the normal load ($P = 0.006$), sliding distance ($P = 0.019$), filler content ($P = 0.063$) are acknowledged being responsible to drive the sliding wear loss. However, the effect of sliding velocity ($P = 0.547$) is relatively lesser as importance on the sliding wear loss.
3.7. Surface morphology

The micrographs obtained at higher magnification with the FESEM allow to observe the worn surface morphology for all the unreinforced and reinforced composites (see details in figures 10 to 12). Figure 10 shows the characteristics for worn surface of both base (C93200) metal alloy and marble dust reinforced composites. It describes the wear characteristics when was used a steady state condition by imposing different sliding velocity (between 1.57 m s\(^{-1}\) to 5.73 m s\(^{-1}\)) and keeping fixed the normal load (15 N) and distance (1300 m). These observations were corroborated to the maximum wear loss for all the composites at the highest sliding velocity i.e. 5.73 m s\(^{-1}\) (see details in figure 5).

The worn surface morphology of control (unfilled marble dust) condition (see figure 10(a)) shows an apparently unaffected surface. However, on depth surface of this unfilled base alloy we can note the ploughing (abrasion) signs responsibly for material removal. The local ductility of C93200 alloy can slightly rise during the mechanical loading in sliding routine. Therefore, the produced heat due to frictional forces can generate softener of matrix material which enable ploughed of hard asperities to the counterface. By increasing the amount of marble dust reinforced to 1.5 wt% the surface micrograph after loading condition reveals specific cracks along with continuous grooves [8] (see details on figure 10(b)). Probably, the continuous grooves are cause of hard penetration of the asperities. This can be driven by the abrasions process in relationship to the fragmented hard reinforced particles that act as third body abrasions. The surface of novel composite containing 3 wt% of marble dust indicates the formation of a complex wear process that cover macro cracks along with delamination wear patterns while some segregation particles were identified as well. This observed wear morphology was depicted in figure 10(c). Further, the surface of 4.5 wt% marble dust reinforced composites presents details of shallow grooves orientated on the sliding direction (see details in figure 10(d)). Accordingly, to figure 4 the 4.5 wt% marble dust reinforced composites has the smallest amount of wear loss. The decrease in the wear loss is attributed to a proper filler wt% content that offer a better sticking mechanism between base matrix and hard reinforcement particles. Hence, this composition demonstrates as well better hardness features (see figure 3). The removal of matrix material was observed for 6 wt% marble dust reinforced composites as shown in figure 10(e). The higher speed combined with the load, when the pin slides against the interface of counterface, is accompanied by significant frictional heat. It led to a reduced material matrix with much lower interfacial bonding performances at the interface of reinforced particles with the base matrix alloy. Such as, the hard reinforced particles are much easy pulled out during the sliding action [36].

| Source          | DoF | Adj SS  | Adj MS  | F-Value | P-Value |
|-----------------|-----|---------|---------|---------|---------|
| Normal Load     | 4   | 0.026386| 0.006597| 8.20    | 0.006   |
| Sliding velocity| 4   | 0.002640| 0.000660| 0.82    | 0.547   |
| Filler content  | 4   | 0.011161| 0.002790| 3.47    | 0.063   |
| Sliding distance| 4   | 0.017912| 0.004478| 5.57    | 0.019   |
| Error           | 8   | 0.006432| 0.000804|         |         |
| Total           | 24  | 0.064531|         |         |         |

**Figure 9.** Evolution of wear loss for the fabricated composite in respect to control factors.
The surface micrograph for both unreinforced (base matrix C93200 alloy) and reinforced composites submitted to different normal loads (from 5 N to 25 N) while fixing the sliding velocity (3.65 m s\(^{-1}\)) and distance (1300 m) was presented in figure 11. Accordingly, to figure 6 the amount of wear loss become higher when the normal load is gradually increased. Where the highest wear loss was observed at maximum applied normal load (25 N). Moreover, it is also found that wear loss decreases as the filler wt% of marble dust increases to a value of 4.5 wt% then is noted a slow decrease. The surface of matrix alloy without reinforcement is presented in figure 11(a). The worn surface morphology of unfilled alloy composite exhibits that the adhesive wear is the main cause of material removal. The bigger load and higher sliding velocity promote relatively huge amount of frictional heat at the two contacting surfaces, responsible for the localized bonding between pin and counterface (disc). Figure 11(b) shows the worn surface micrograph for 1.5 wt% of marble dust reinforced composites where
the delamination wear mechanism is noted. Figures 11(c) and (d) shows the worn surface micrographs for 3 wt% and 4.5 wt% marble dust reinforced composites, where several cracks & continuous parallel grooves [8] was highlighted. At 3 wt% marble dust reinforced composite shows surface with some micro cracks, continuous thin grooves and delamination wear mechanism, whereas at 4.5 wt% marble dust reinforced composites the surface morphology exhibits the general wear pattern of ploughing the softened matrix material from the composites surface. Figure 11(e) reveals specific details of composite surface with 6 wt% marble dust reinforced composite where the removal of matrix as well as filler material was seen. The reason behind this may be poor bonding strength between filler and matrix alloy [37]. Moreover, here the segregation or improper distribution

Figure 11. SEM pattern observed for diverse marble content and normal load variation of fabricated composite (fixed speed 3.65 m s$^{-1}$ and distance: 1300 m).
of filler material that result in large amount of void content (see table 4) and lower hardness may be the possible reason for material removal.

The surface of composite in respect to the optimized parameters obtained as per L_{25} Taguchi approach was depicted in figure 12. It shows micrographs of reinforced/unreinforced composite submitted to different loading conditions. The worn micrographs for unfilled metal alloy composites are presented in figures 12(a) and (b). Parallel and continuous grooves were observed at lowest normal load and sliding velocity (Experiment No. 1) whereas some cracks and ploughing strip [13] along the wear track were observed when same specimen runs under bigger load and higher sliding velocity (Experiment No.18). Obviously, the bigger load corroborated to higher sliding velocity generate a substantial frictional heat at the pin and counter disc interface, which softens the material matrix. Then, finally induce softened matrix alloy, which is ploughed by repeatedly action of hard sharp asperities of counter body. The micrograph of figure 11(b) indicates that unfilled matrix alloy underwent severe plastic deformation at higher load and a higher sliding velocity. Figures 12(c) and (d) shows the worn surface micrographs of 1.5 wt% marble dust reinforced composites. Macro cracks [38] along the sliding direction were noticed at 15 N normal load and the highest sliding velocity (Experiment No. 15), whereas the ploughing phenomenon and delamination wear mechanism were observed at almost the maximum load (20 N) and sliding velocity (Experiment No.19). Figures 12(e) and (f) present details of composite surface containing 3 wt% marble dust. Continuous deep grooves [8] that follow the wear track are revealed (see figure 12(e)) at moderate applied normal load and lowest sliding velocity (Experiment No.11) whereas when the same weight percentage of reinforced composite runs at highest load and higher velocity (Experiment No. 24) the material become removed by ploughing mechanism due to high frictional heat. The composite surface containing 4.5 wt% marble dust reinforced were presented in figure 12(g) and 12 h. The surface show only smooth and continuous thin grooves orientated in the sliding direction when was applied lower load (Experiment No. 4). It endorses a good sticking progress at the interface of hard particle with the matrix alloy with superior hardness (details in figure 3) together with minimum wear loss. But when same weight percentage reinforced composite is subjected to highest normal load and sliding velocity (Experiment No. 25) severe plastic deformation of wearing surface occurs. At bigger applied normal load and higher sliding velocity, the matrix alloy gets softened. Hence, the hard reinforced particles and the material matrix is pulled out creating wear debris which further are blow-out forming the wear scar. Figures 12(i) and (j) shows the worn surface micrographs of 6 wt% marble dust reinforced composites. It shows the removal of material from the matrix in big portion (Experiment No. 13). Nevertheless, at highest load and lowest sliding velocity some cracks and deep grooves were observed as well (Experiment No. 21).

4. Conclusions

In this research work, marble dust reinforced C93200 copper based alloy composites were manufactured via liquid metal stir casting process. Then, the samples manufactured were characterized in terms of mechanical and tribological properties. The key conclusions were summarized as:

1. The mechanical properties of novel bearing material as composite significantly enhanced by varying the amount of marble dust weight percentage. It enables to reduce the void content by increasing the marble dust content. The void content decreases from 0.785% to 0.497% when is added up to 4.5 wt% of marble dust, however, beyond 4.5 wt% the void fraction increases to 1.548%.

2. The reinforcement of marble dust ceramic particulates enhances the mechanical properties (i.e. tensile strength, hardness, compressive, flexural strength, respectively) for the novel manufactured alloy composites when is added up to 4.5 wt%, but when a large amount of marble dust reinforcement is added (i.e. 6 wt%) a slightly reduction in the mechanical performances is noted.

3. Superior wear performances were achieved no matter either normal load and/or sliding velocity by introduction 4.5 wt% marble dust into matrix material.

4. The statistical simulation performed under ANOVA were successfully to detect the critical parameters that drive the sliding wear loss. The normal load was indicated as critical one, followed by filler content along with the sliding velocity as magnitude order to stimulate the sliding wear loss for prepared composites.

5. Wear micrographs of 4.5 wt% marble dust reinforced composite exhibit superior wear performance in respect to other combination analysed which make it preferable for bearing applications.
Figure 12. SEM patterns of unreinforced and reinforced composites optimized as per Taguchi Design of Experiment.
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