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HAITER LENIN A.0^{1*}, SRINIVASAN MURUGAN⁰², SUJIN JOSE ARUL⁰³, MARY VASANTHI S.⁰⁴

A NOVEL REAL-TIME ASSESSMENT OF THE WEAR ANALYSIS OF Cu-Ni-Sn HYBRID COMPOSITE FOR MULTIFUNCTIONAL APPLICATIONS STRENGTHENED BY NANO B₄C

The hybrid composite is fabricated by powder metallurgy technique. The addition of ceramic reinforcements to the matrix phase helps in attaining tribological properties. Initially, the metal powders are prepared by atomization and reduction process. To reduce oxidation of pure metals, electrolytic process is carried out. The crushing, milling and sintering process are carried after atomization to obtain fine grain sized particles; these are then characterized using Scanning Electron Microscope (SEM), X-Ray Diffractometer (XRD). The obtained particles are then blended and compacted to attain better hardness and wear resistant of the materials. The hybrid composites prepared for the analysis by reinforcing 15%Ni-8%Sn-4%B₄C to the matrix phase Cu. The pinon-disc method is incorporated to study the wear rate, hardness and co-efficient of friction. By varying the contribution parameters such as load 10 N-25 N, sliding distance 400 mm-1000 mm and sliding speed, the matrix phase materials exhibited rigorous wear, whereas the reinforced hybrid composite (Cu-15%Ni-8%Sn-4%B₄C) provide good strength and wear resistant and also reduced volume loss of 25×10^{-5} cm³ and 110×10^{-5} cm³ for 10 N load with 400 mm and 1000 mm sliding distance even at sintering temperature of 900°C. The microstructure and morphology of the worn surface is analyzed using Scanning Electron Microscopy images. The sintered density of the hybrid composite is less due to reduced number of pores and so material defect is also condensed.

Keywords: Hybrid Composite; Wear; Characterization; Powder Metallurgy; Hardness

1. Introduction

The hybrid composites are novel material because of its increased demand in emerging field like space, defense, aircraft and automotive applications. The hybrid composite is mainly used due to its strength and light weight [1]. The composites are classified based on the matrix and material structure. The matrix composites are further classified as polymer, ceramic and metal matrix composites and material structure as particulate, laminate and fibrous composites [2].Different processes such as atomization, chemical reduction, electrolytic process, crushing, ball milling, condensation, blending, compaction and sintering of composite powders are involved in the preparation of composites [3]. Hence fine grain sized with homogeneous distribution of materials can be obtained. This leads to better bonding of matrix phase and reinforcement and hence strengthening effect is improved [4]. In this study, Copper, nickel and tin are used as primary matrix phase and boron carbide as secondary reinforcement phase. Boron carbide is a hard ceramic and covalent material used

in bulletproof vests, defense and numerous industrial applications [5]. KerimEmreOksuz studied the wear resistance on composite Cu-Sn-2%B₄C, at the applied load of 40 N. It maintained high strength and hardness and hence used in cutting tools and abrasives [6]. The wear behavior is unusual on copper-chromium alloy at room and moderate temperatures of about 300°C [7]. In the study of grain growth of Al-SiCnano-crystalline composites, grain growth was suppressed during sintering and hot processing [8]. A study explained that SiC, B₄C, and graphene reinforcements with aluminium matrix enhance the properties such as wear resistance, tensile strength of Al based materials [9]. Reinforcing of nano ceramic particles with Al, Cu and similar material enhance the physical and mechanical properties of the base materials [10].By considering all the literature, this research aims to develop unique combination of hybrid composites for enhancing the strength and lightweight properties by reinforcing of Ni, Sn and B₄C with Cu. This work mainly addresses on the effect of volume fraction on wear rate, hardness and sintered density of the composites with the addition of 4% B₄C reinforcement.

¹ WOLLO UNIVERSITY, SCHOOL OF MECHANICAL AND CHEMICAL ENGINEERING, KOMBOLCHA INSTITUTE OF TECHNOLOGY, KOMBOLCHA, ETHIOPIA ² MAHENDRA ENGINEERING COLLEGE, DEPARTMENT OF MECHANICAL ENGINEERING, NAMAKKAL, TAMILNADU, INDIA

³ AUTOMOBILE ENGINEERING, NEW HORIZON COLLEGE OF ENGINEERING, BENGALURU. INDIA

⁴ ST XAVIER'S CATHOLIC COLLEGE OF ENGINEERING, DEPARTMENT OF ELECTRONICS AND COMMUNICATION ENGINEERING, NAGERCOIL, TAMILNADU, INDIA

* Corresponding author: drahlenin@kiot.edu.et



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2. Materials and methods

2.1. Materials

The materials used in this research are spinodal alloys i.e., Cu-Ni-Sn and boron carbide B4C. Cu is having high electrical conductivity, 8.96 g/cm³ density. Boron carbide is a ceramic having high strength, temperature withstanding material. To enhance the tribological properties, hard materials are incorporated as reinforcement to the matrix phase. The processes like milling, crushing and blending are performed to obtain coarse grain hybrid composite materials. The Powder Metallurgy technique is used and the materials are mechanically alloyed to micron range 1-100 µm. They are tested for size, morphology and phase transformation of particles using Scanning Electron Microscope and X-Ray Diffractometer with a diffraction angle 2θ and CuK_a radiation. The pin-on-disc experiment is carried out to determine the hardness and wear behavior of the hybrid composite by adjusting the variable parameters like load N, sliding distance m and sliding velocity m/s with different volume fraction % of the materials.

2.2. Scanning Electron Microscope (SEM)

The high-energy beam of electrons that interact with the sample produces backscattered electrons. It penetrates to a depth of microns, collects the signals andsend to detectors to form images and displayed on the screen visually. The maximum resolution obtained by SEM is below 1 nm [11].

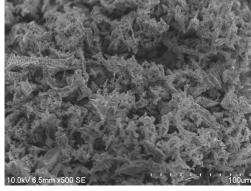
Fig. 1(a) shows the SEM image of Cu metal which is a short cylindrical tube like nature with less adhesion. The grain size is around 80-100 μ m. The nickel alloys are ductile and resistance to corrosion and heat with density of 8.9 g/cm³ at 20°C. The copper sample is the good conductor of heat with 8.9 g/cm³ density at 20°C. Fig. 1(b) shows the morphology and grain size of nickel alloy which appears as solid spherical balls of size 30 µm with reduced agglomeration.

The hybrid composites reinforced with 4%B₄C to copper alloy exhibits good morphology and coarse grain structure.

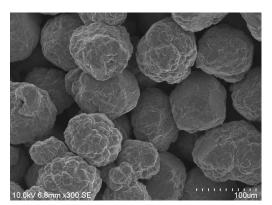
Fig. 3(a-b) proves that Cu &Ni and Cu &Sn are well impeded each other and appear as little clumsier and lathered nature with dimension ranges from 5-20 µm. Fig. 3(c-d) shows the compaction of each samples with diameter of about 1-10 μ m.

2.3. X-Ray Diffractometer (XRD) analysis

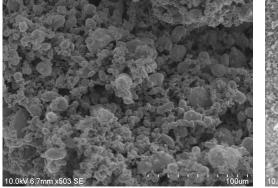
The X-ray powder diffraction method is used to determine the molecular structure, purity, lattice parameter and phase identification of the samples. The X-rays get scattered by the atoms present in the sample. From the scattered beams, the maximum intensity peaks for the particular sample is obtained [12].



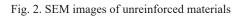
(a) SEM image of Copper (Cu) Fig. 1. SEM image of unreinforced materials



(b) SEM image of Nickel (Ni)

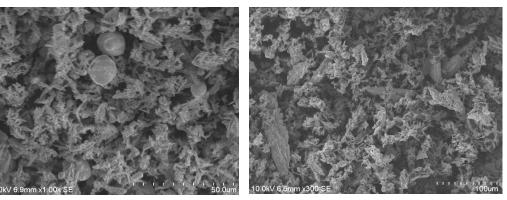


(a) SEM image of Tin (Sn)



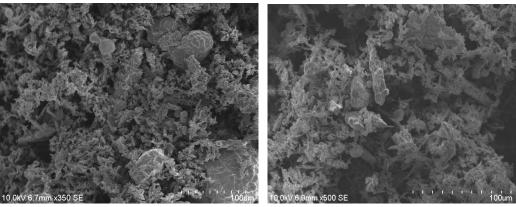


(b) SEM image of 2% Boron carbide



(a) SEM image of Cu-Ni

(b) SEM image of Cu-Sn



(c) SEM image of Cu-Ni-Sn

Fig. 3. SEM images of reinforced materials

(d) SEM image of Cu-Ni-Sn-2%B₄C

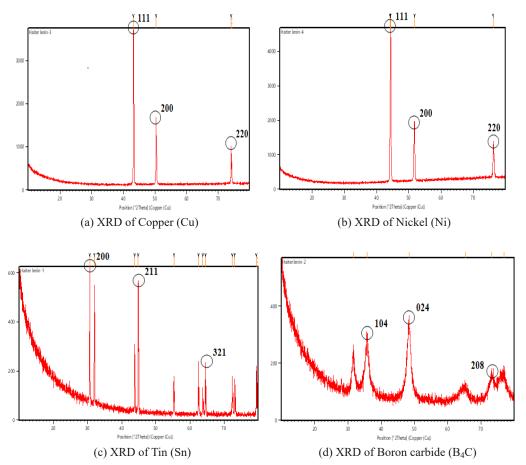


Fig. 4. XRD patterns for unreinforced materials before the experimentation

The data can be deduced using Bragg's law, inter-planar spacing d and spacing angle θ . The projector and detector are separated by an angle 2 θ with CuK_a radiation with wavelength 1.54 Å. Fig 4 shows the XRD patterns of the unreinforced materials. In Fig. 4(a), the intensity peaks reflected at $2\theta = 43.5^{\circ}$ and 51° at the lattice planes hkl as (111) and (200) respectively. The particle size is obtained from FWHM of the peaks at diffracted wavelength 1.54 Å. Fig. 4(b), shows the diffraction pattern of nickel with thin lines of intensity peaks and hkl lattice planes at (111) and (200) with the intensity reflected at $2\theta = 45^{\circ}$ and 52°.

The Fig. 5(a-d) illustrates the hkl lattice planes (200) and (211) of tin and boron carbide with the intensity at 31° and 45° , (104) and (024) at intensity of 36° and 49° respectively. The reinforcement of carbide material to the alloy is well mixed and tested for XRD results.

2.4. Wear Test

Wear test can be studied for composites after drilling, welding, cutting, straightening, stress relieving, annealing and heat treatment [13]. During these processes, oxidation of composite materials takes place, leading to the transition of material from brittle to ductile or vice versa [14]. The pin-on-disc experiment is carried out to study the wear behavior and hardness of the prepared material such as Cu, Ni, Sn, Cu-15%Ni-8%Sn and hybrid composite Cu-15%Ni-8%Sn-4%B₄C with the increase in load N and sliding distance 400 mm-1000 mm. The specifications of pin on disc setup are 165×80 mm disc size, 5-200 N normal load, 200-2000 rpm rotational speed .

3. Results and discussion

3.1. Effect of volume loss on sliding distance

The hybrid composites are experimented for wear rate, weight loss, specific wear rate, hardness, compaction and sintered density at 900°C with a sliding velocity of about 20.94 m/sec. From Fig. 6(a-e), one can notice that hybrid composites appear to be gradual reduction in volume loss compared to other unreinforced materials. The load 10-25 N applied along with the sliding distance of 400 mm-1000 mm is varied and the volume loss of the material can be determined. The Cu metal is a matrix and the primary phase 15%Ni-8%Sn are added to secondary phase material 4%B₄C to form hybrid composite. When the normal load 10 N is applied with 400 mm sliding distance, pure Cu exhibits very less volume loss. It may be attributed to its relatively low hardness, reduced adhesion and friction forces, and the early stages of wear development at this sliding distance. The gradual

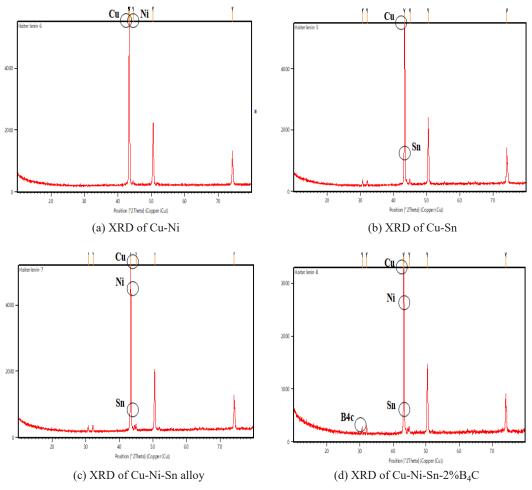


Fig. 5. XRD patterns of reinforced composites before the experimentation

increase in load up to 25 N and sliding distance upto 1000 mm, results in elevated volume loss.

The pure copper, Cu-15%Ni and Cu-8%Sn provides volume loss of 35×10^{-5} cm³ at 10 N load and 400 mm sliding distance. These materials (Cu-15%Ni-8%Sn) are blended and experimented for wear. They exhibit slight increase in volume loss of about 40×10^{-5} cm³ and 80×10^{-5} cm³ at 10 N and 25 N load respectively with 400 mm and 1000 mm sliding distance, while the hybrid composites, Cu-15%Ni-8%Sn-4%B₄C appears to fall on 25×10^{-5} cm³ and 110×10^{-5} cm³ for 10 N load with 400 mm

and 1000 mm sliding distance respectively.

3.2. Effect of wear rate on sliding distance

The material specimens are tested for wear behavior for varying load and sliding distances with constant sliding velocity and sintering temperature. In our study, five specimens such as pure Cu, Cu-15%Ni, Cu-8%Sn, Cu-15%Ni-8%Sn and Cu-15%Ni-8%Sn-4%B₄C are tested to compare the wear behavior by varying the contributing factors. From the plots 7(a-d), before the addition of reinforcement (hybrid composite), all the four specimens exhibit wear of about 80×10^{-5} mm³/mat 10 N with 400 mm sliding distance and approximately 120×10^{-5} mm³/m for

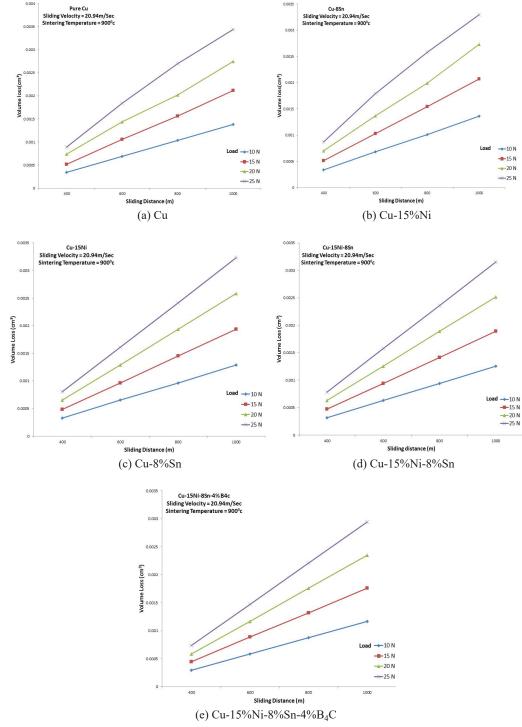


Fig. 6. Graph represents effect of wear rate on volume loss of metal, matrix and composites

the sliding distance of 1000 mm. The wear rate for the increase in load up to 25 N for the four specimens are $200 \times 10^5 \text{ mm}^3/\text{m}$ and $340 \times 10^{-5} \text{ mm}^3/\text{mat}$ sliding distance 400 mm and 1000 mm respectively.

The hybrid composite Cu-15%Ni-8%Sn-4%B₄C shown in Fig. 7(e) clearly proves that even at heavy load applied with increase in sliding distance, there is complete reduction of wear compared to unreinforced materials. When 10 N load is applied, the wear seems to be 75×10^{-5} mm³/m and 120×10^{-5} mm³/m at 400 m and 1000 m sliding distance respectively. When the load applied is raised to 25 N, the hybrid specimen appears to be deduced well and inferred from the plot as 180×10^{-5} mm³/m and 280×10^{-5} mm³/m for the sliding distance of 400 mm and 1000 mm correspondingly.

3.3. Effect of specific wear rate on sliding distance

The specific wear rate is given as wear volume per unit distance and load. The specimens are tested and found the incredible changes can be visited using these plots. The specimen pure Cu shown in Fig. 7(a) appears to be very high with sudden raise in results of applied load and sliding distance. It provides the data points of specific wear rate 68×10^{-6} mm³/Nm and

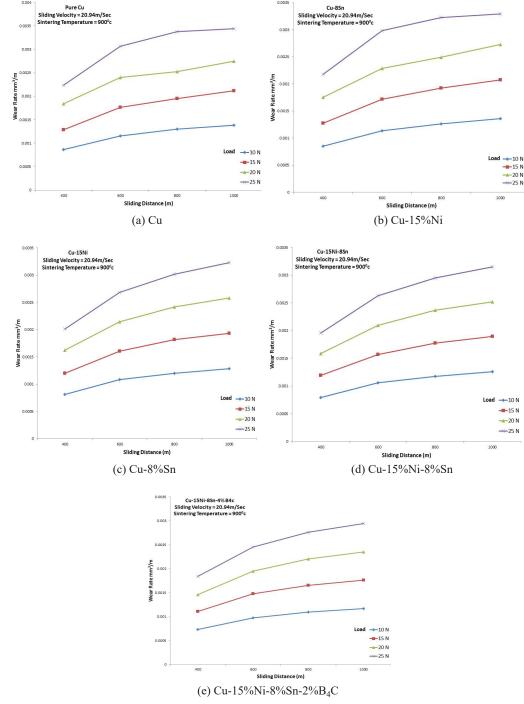


Fig. 7. Graph represents effect of wear rate on sliding distance of metal, matrix and composites

 118×10^{-6} mm³/Nm for the increase in sliding distance of 400 mm and 1000 mm during the load applied 10 N.

The load 25N applied to the sample Cu exhibit wear rate of 208×10^{-6} mm³/Nm and 318×10^{-6} mm³/Nm at 400 m and 1000 m sliding distance respectively.

The hybrid composite experimented for specific wear rate produce the values of 71×10^{-6} mm³/Nm and 117×10^{-6} mm³/Nm during 10 N and 25 N loads applied with sliding distance of 400 mm and 1000 mm respectively. This hybrid composite gave better performance of all other samples and the data lies in same line as shown in plots 8(a-e).

3.4. Effect of volume loss, wear rate and specific wear rate on composition

The compositions are taken and data values are compared for wear and volume loss to know about the reduction of loss %, wear and specific wear rate. From the graph 9(a-c), we can notice clearly that unreinforced material shows peak value at the sliding distance of 1000 mm. The samples are heated to high sintering temperature of 900°C and at constant sliding velocity 20.94 m/sec and are experimented by varying the parameters like sliding distance and load applied. The matrix phase reveals

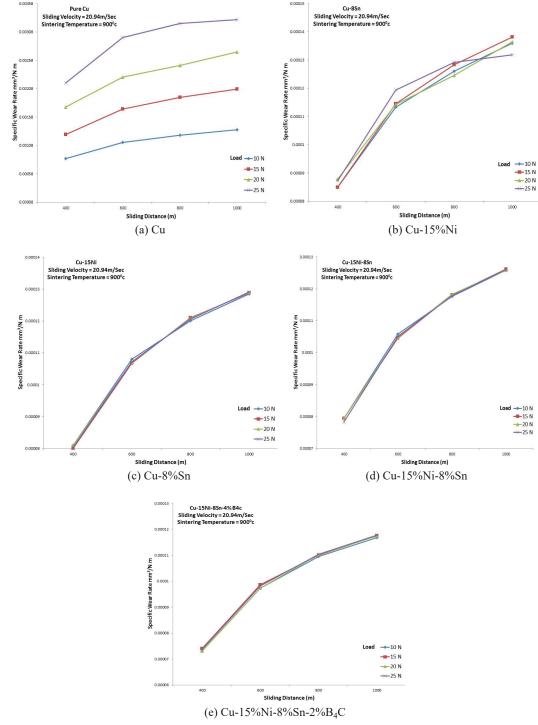


Fig. 8. Graph represents effect of sliding distance on specific wear rate of metal, matrix and composites

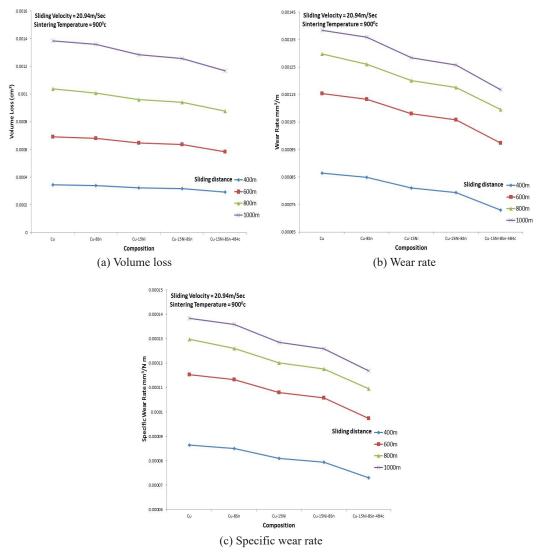


Fig. 9. Graph represents behaviors of different composition with respect to sliding distance

very less reduction in volume loss at basic load, while the carbide reinforcement added and sintering temperature

hybrid composite produce gradual decrease in wear rate and huge changes in specific wear rate. The result change is mainly due to the contributing factors of samples in different fraction % and variable parameters.

From Fig. 9(a) we infer that, there is an observable reduction in volume loss whereas the wear rate picked from Fig. 9(b) found to be deducing more and the hybrid composites hold specific wear rate performance. The reduction may be due to softer phases or altered microstructures, while increases could result from the incorporation of harder materials or improved alloying.

3.5. Worn surface and Microstructure

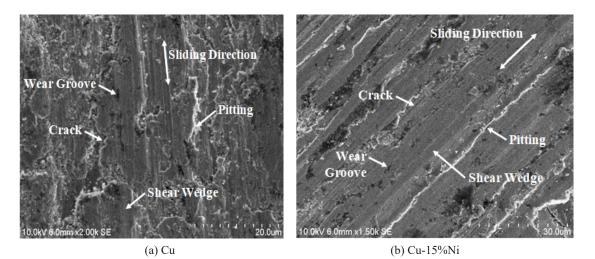
The wear surface can be analyzed metallo graphically, where the samples were placed inside a vacuum chamber. A holder keeps the sample for testing and the electron microscopic images were used to get the information about the specimen while the load is applied at high sintering temperature.

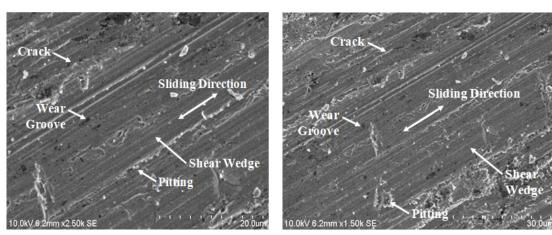
The material both withstand to its maximum and form wear grooves also [15]. The wear processes is carried out at argon, nitrogen and oxygen atmosphere which are resisted by formation of fatigues, shear edges, cracks and pitting. The wear of the specimen is decreased with addition of volume fraction % of the hard material which proved the scratch reduction with increased resistance of wear [16]. The plastic deformation is high for the matrix phase and less for the reinforced material because of good adherence to the tools and reduction in the abrasive wear [17]. The microstructures of unreinforced copper expose the variations in processing and mixing, which can be seen through the electron microscope and investigated that the dust powders disappear like ashes in few seconds [18]. The adhesion is poor while mixing the reinforcement to the copper matrix , resulting in fade away of the dust inside the evacuated chamber of the microscope and resulted in weak bonding between the interface of the Cu/Ni, Cu/Sn and hybrid composite Cu/Ni-Sn/B₄C.

The present study utilized carbide material which is hard and withstands the wear when the heavy load is applied even at high sintering temperature. The reinforcement added to the matrix provides better bonding and heterogeneous distribution [19]. The formation of grain boundaries at the center forms the long shear wedges in all specimens as shown in Fig. 10. The resulting images 10(a-c) shows the morphology of worn surface having smooth patches and less wear grooves. Fig. 10(e) shows that reinforced hybrid composite Cu-15%Ni-8%Sn-4%B₄C exhibit micro cracks with long shear wedges and continuous pitting. Since there is no breakage and brittle of specimen, it is capable of bearing load and provides strength and hardness to the composite material.

3.6. Sintered density test

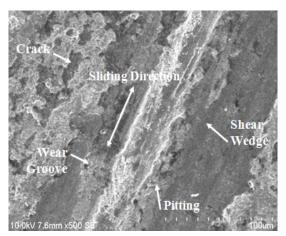
The sintered density is a crucial factor which plays an important role in maintaining the tribological properties of the specimen. If the material is affected by load and temperature changes, its density also changes [20]. The composite materials are of coarse grain, the number of pores is fewer, and thereby the material defect can be reduced due to the densely occupation of materials but no agglomeration. The concentration of





(c) Cu-8%Sn

(d) Cu-15%Ni-8%Sn



(e) Cu-15%Ni-8%Sn-4%B₄C

Fig. 10. SEM image of worn surface for unreinforced and reinforced composites

the agglomeration is deduced during sintering and increase in density results in boost up of material hardness.

The study on the sintered density of the hybrid composite reveal that the powders are taken and blended manually for few minutes. Then cold pressing is carried out with a pressure of 500 MPa and sintering at 900°C for 60 min. The collected experimental values are plotted and precede sintered density for all different compositions exposed in Fig 11 (a).

The copper metal matrix positioned at 7.62 g/cm³, Cu-15%Ni lie on 7.76 g/cm³ and the hybrid composite located at 7.3 g/cm³. The performance is good for 4%B₄C reinforced material compared to previous work 2%B₄C. The investigation is continuing to 6% boron carbide volume fraction and the upcoming result will be fine and make the material to be eco-friendly. The further efforts on this area will use critical material and employed in numerous applications.

3.7. Hardness test

The most commonly equipped method is Rockwell hardness test and ASTME-18. The metals and composites uses Rockwell hardness test for the accuracy comparison to other testing methods. The penetration depth and the impression on the material acting upon the composites are found out by the indenter in attendance with the equipment. The hardness density of the hybrid composite material is affected by the formation of phases under the reaction with the environment and can be reduced by increasing the time of mechanical alloying during the process [21]. The concentration of the hard carbide material is also an important contributing factor in affecting the hardness. It can be lift up by the mechanically alloying process. The hardness can be measured by Rockwell hardness or Vickers hardness test.

The 4% weight fraction of B_4C reinforced hybrid composite integrated with various compositions in different fraction weight are tested for strength using Rockwell hardness number. The powders were compacted and sintered to 900°C and the phases affecting environment are make off for good presentation. After that load applied on the composite which bear and sustain from delaminating of surface of the material and keep up the tribological properties for longer, the composite encompass good strength and can be applied for field purpose to function. The bar chart drawn for the hardness number data obtained for composition is shown in the Fig. 11(b). The copper metal alone shows 19 hardness numbers whereas the composition of Cu added to nickel and tin falls on 6-9. The hybrid composite Cu-15%Ni-8%Sn-4%B₄C appears on the hardness value 25. The higher the volume fractions of carbide material, better the hardness and strength of the material.

4. Conclusion

The primary matrix phase Cu-15%Ni-8%Sn and the secondary reinforcement phase Cu-15%Ni-8%Sn-4%B4C is a hybrid composite prepared by using powder metallurgy technique were characterized for morphology and size of the samples. The specimens are tested under pin-on-disc apparatus to study the wear behavior and compared the data for B₄C volume fraction % with matrix at variable load and sliding distance. The effect of composition 4%B4C on wear rate, volume loss and specific wear rate can be studied by the experimental data obtained while testing. The reinforced hybrid composite provide better diminish of wear rate and weight loss of the specimen compared to the unreinforced matrix material. The performance of the composite with the effect of specific wear rate on sliding distance is very much improved and so withstanding of wear and pitting is proved. While undergoing wear debris analysis, plastic deformation, cracks and shear wedges are diminished and withstand along their sliding direction.

In this study, even at high sintering temperatures and loads applied, 4% weight fraction of boron carbide results in superior coordination in wear rate. Even the samples prepared in micron range furnish good improvement in strength and wear resistance. In future, we will use the samples to be prepared in nano-scale and increase the weight fraction to 6% and greater performance will be achieved with less volume fraction of hard materials.

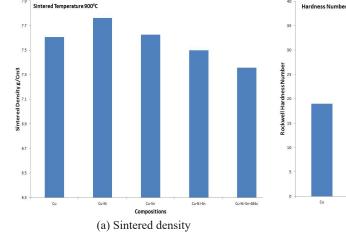
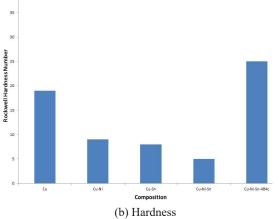


Fig. 11. Effect of composition on sintered density and hardness



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