

CHAPTER 3

EXPERIMENTAL PROCEDURE

This chapter deals with the experimental procedures used in conducting investigation for development of copper-based hybrid composites and characterized for microstructural, mechanical, electrical and tribological properties. As received commercial pure copper has been also cast and characterized for the same properties for comparison with developed copper-based hybrid composites.

3.1. Materials selection for development of copper-based hybrid composites

In the present study, commercial pure copper having purity of 98 % has been selected as the matrix material for the development of copper-based hybrid composites using stir-casting (solidification processing) technique. Commercial copper was procured from New India Metal Pvt. Ltd., Varanasi, India. The compositional details of the commercial copper ingot in weight percent are given in Tables 3.1.

Table 3.1. Compositional details of commercial copper

Elements	As	Pb	Fe	Ni	Cd	Cr	Cu
Wt. %	0.015	0.05	0.005	0.032	0.048	0.076	Balance

Hard ceramic powders of, tungsten carbide (WC), alumina (Al_2O_3), zirconia (ZrO_2), boron carbide (B_4C), and boron nitride (BN) have been selected as the reinforcing particles to impart high strength, good wear resistance and low contact adhesion in the copper matrix.

These are supplied by Sigma Aldrich, Germany. To promote wetting reaction of these reinforcing particles, a small amount of chromium (Cr) is added that is also supplied by Sigma Aldrich. The particle size and other specifications of reinforcing particles are given in Table 3.2

Table 3.2. Specification of reinforcing powders used in development of copper-based hybrid composites

Reinforcing particles	Purity (%)	Density (g.cm ⁻³)	Particle size (µm)
Tungsten carbide (WC)	98.0	15.63	< 40
Alumina (Al ₂ O ₃)	99.0	3.95	< 40
Zirconia (ZrO ₂)	99.5	5.68	< 40
Boron nitride (BN)	99.0	2.1	< 40
Boron carbide (B ₄ C)	98.0	2.52	< 40
Chromium (Cr)	99.0	7.19	< 40

3.2. Development of copper-based hybrid composites

The technique stir-casting was used to develop the copper-based hybrid composites; its schematic diagram and pictorial view and image of cast composite are depicted in Fig. 3.1.(a), Fig. 3.1.(b) and Fig. 3.1.(c) respectively. The compositional details of composites are given in Table 3.3. The commercially copper was kept in a graphite crucible and placed in a muffle furnace. When commercial copper melted, it was stirred using the graphite blade stirrer and cleaned by skimming. It was followed by addition of weighed quantity of preheated (at 100 °C) reinforcing particles at a controlled rate through a vibratory feeder into the copper melt. Simultaneously, it was stirring by graphite blade stirrer at 500 rpm driven by a 0.5 HP motor, having a maximum rated speed of 2000 rpm. The position of stirrer inside

the crucible was always kept constant at a given level. During process of stirring, the slurry temperature was maintained within ± 50 °C more or less the temperature of processing (1200°C). After 10 minutes, the stirring was stopped. At any stage of processing, there was no practice carried out for degassing of the slurry or melts. Then, the slurry or melts in the crucible was poured into a preheated (at 150 °C) permanent steel mould and allowed to cool in open air.

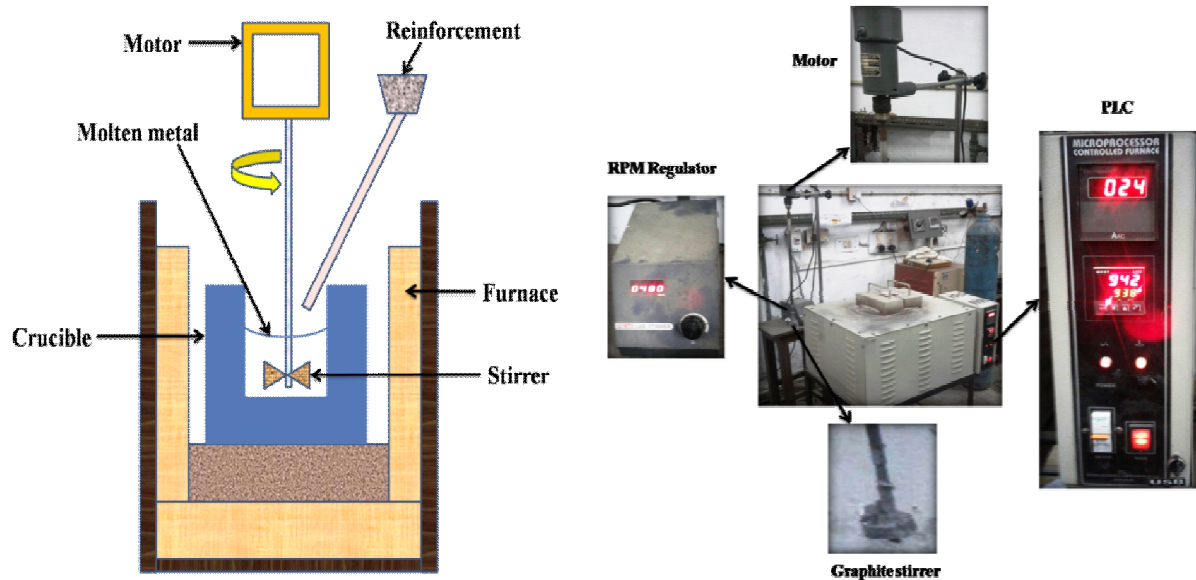


Fig.3.1.(a) Schematic diagram of stir-casting technique

Fig.3.1.(b) Pictorial view of stir-casting technique



Fig.3.1.(c) Photographic image of cast composite

Table 3.3. The compositional details of copper-based hybrid composites

BINARY REINFORCED COPPER HYBRID COMPOSITES						
Composition	Designation	Cu (wt. %[*])	Cr (wt. %[*])	WC (wt. %[*])	ZrO₂ (wt. %[*])	Al₂O₃ (wt. %[*])
Cast copper	CC	100.0	---	---	---	---
Cu-2Cr-1WC- 1ZrO ₂	HC-1	96.0	2.0	1.0	1.0	---
Cu-2Cr-1WC- 1Al ₂ O ₃	HC-2	96.0	2.0	1.0	---	1.0
Cu-2Cr-1WC- 2ZrO ₂	HC-3	95.0	2.0	1.0	2.0	---
Cu-2Cr-1WC- 2Al ₂ O ₃	HC-4	95.0	2.0	1.0	---	2.0
TERTIARY REINFORCED COPPER HYBRID COMPOSITES						
Composition	Designation	Cu (wt. %[*])	Cr (wt. %[*])	WC (wt. %[*])	BN (wt. %[*])	B₄C (wt. %[*])
Cu-2Cr-1.5WC- 1BN-0B ₄ C	HC-5	95.5	2.0	1.5	1.0	0.0
Cu-2Cr-1.5WC- 1BN-0.5B ₄ C	HC-6	95.0	2.0	1.5	1.0	0.5
Cu-2Cr-1.5WC- 1BN-1B ₄ C	HC-7	94.5	2.0	1.5	1.0	1.0
Cu-2Cr-1.5WC- 1BN- 1.5B ₄ C	HC-8	94.0	2.0	1.5	1.0	1.5

(*) All compositions are in percent of total weight.

Copper-based hybrid composites are broadly classified as binary reinforced and tertiary reinforced hybrid composites depending on the number of reinforcement into the copper metal matrix. These binary reinforced hybrid composites have been designated as Cu-2Cr-1WC-1ZrO₂ (Hybrid composite-1 (HC-1)), Cu-2Cr-1WC-1Al₂O₃ (HC-2), Cu-2Cr-1WC-2ZrO₂ (HC-3) and Cu-2Cr-1WC-2Al₂O₃ (HC-4). Whereas, tertiary reinforced hybrid composites are designated as Cu-2Cr-1.5WC-1BN-0B₄C (HC-5), Cu-2Cr-1.5WC-1BN-0.5B₄C (HC-6), Cu-2Cr-1.5WC-1BN-1B₄C (HC-7) and Cu-2Cr-1.5WC-1BN-1.5B₄C (HC-8). The process flow chart of development of hybrid composites based on copper is depicted in Fig. 3.2.

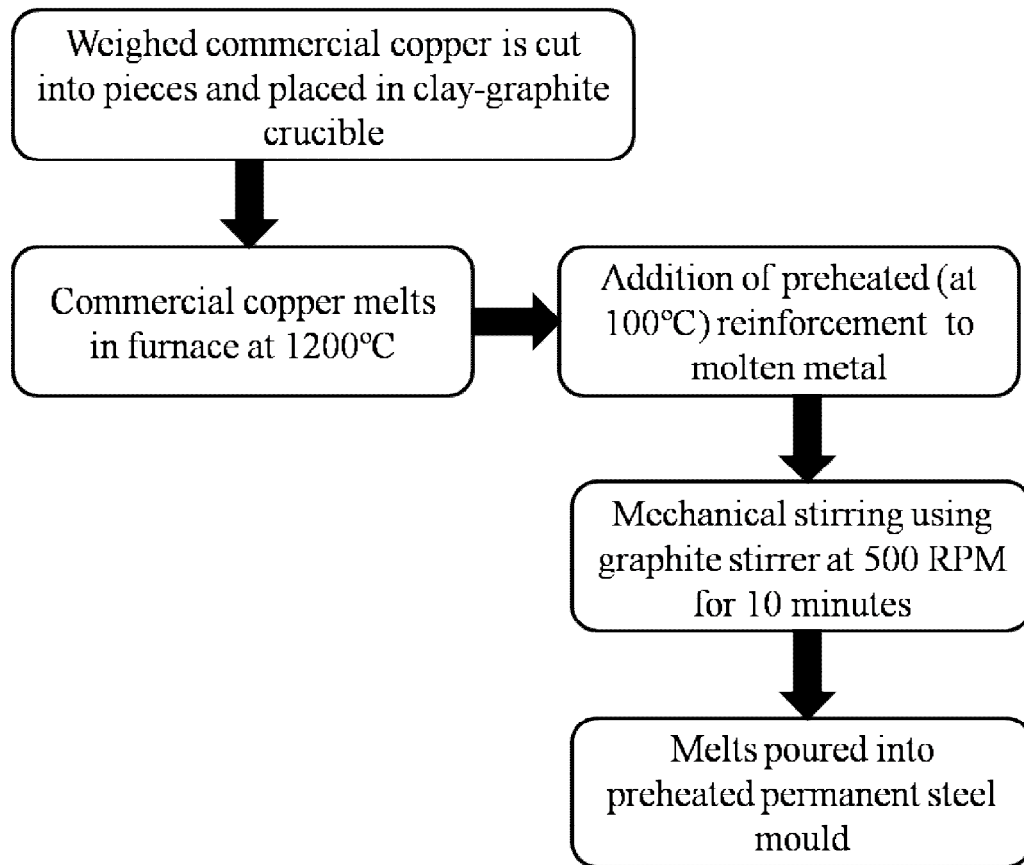


Fig.3.2. Process flow chart of development of copper-based hybrid composites

3.3. Characterizations

The following sub-section describes the details of the techniques and procedures used to characterize the microstructural, mechanical, electrical and tribological behaviors of the both copper matrix and copper-based hybrid composites in the present investigation.

3.3.1. X-ray diffraction analysis of hybrid composites

X-ray diffraction investigation was carried out on samples of the both copper matrix and copper-based hybrid composites to find out the lattice strain, crystallite size, and phases present. X-ray diffraction (XRD) was performed by Rigaku Desktop Miniflex II X-ray diffractometer (Tokyo, Japan) with a Ni-filtered Cu-K α emission ($\lambda=1.5406\text{\AA}$) operated at 40 kV/30 mA with a scanning speed of 3 $^\circ$ /min in the angle (2θ) range between 20–100 $^\circ$. For all the intensity peaks and corresponding values of 2θ , the inter planner spacing, d , was evaluated using Bragg's law given by Eq. 3.1, which is ultimately utilized for detection of different phases with the help of X-ray diffraction statistics details (JCPDS)

$$2d \sin\theta = n\lambda \quad (3.1)$$

where, θ - Incident angle

λ - Wavelength of the X-ray

n - An integer representing the order of the diffraction.

The lattice strain and crystallite size were evaluated by the standard Williamson–Hall equation given below (Williamson et al., 1953).

$$\beta_{hkl} \cos\theta_{hkl} = \left[\frac{k\lambda}{t} \right] + 4\epsilon \sin\theta_{hkl} \quad (3.2)$$

where, k - Shape factor (0.9),

λ - Wavelength of X-rays (1.5406 \AA),

hkl - Miller indices,

θ - Bragg's angle,

t - Crystallite size normal to the reflecting planes and

ϵ - Lattice strain.

The first five Cu reflections (111), (200), (220), (311) and (222) were used to build a linear plot of $\beta_{hkl} \cos\theta_{hkl}$ against $2\sin\theta_{hkl}$. The crystallite size (t) was achieved from the intercept, c (i.e., $c = k\lambda/t$), and the strain (ϵ) was obtained from the slope (i.e., $m = \epsilon$). The results are presented in Chapter 4.

3.3.2. Microstructural evaluation of hybrid composites

For microstructural investigation, specimen of both copper matrix and hybrid composites were manually polished following the standard metallographic procedures as discussed below. The surface of the specimen that is to be examined was first made plane by means of a specially designed motor-driven emery belt. The sharp edges of the specimen were then beveled to avoid the tearing of the emery paper in the subsequent polishing. The specimens were then polished manually using the silicon carbide metallographic emery papers (400, 600, 800, 1200 and 1600 grit). During polishing on each emery paper the direction of grinding was keep changing to right angles from the previous grinding direction. The final polishing is carried out on a velvet-cloth using brasso and kerosene oil on a polishing machine (Bainpol, Metco, Chennai, India). After polishing, the specimens were etched using Ferric chloride (FeCl_3) etchant and followed by washed, dried. Finally, dried samples were examined using optical microscope (Dewinter Opticals Inc., New Delhi, India). Microstructural features of all these prepared samples are taken and analyzed.

The microstructural study of the samples were also performed by high resolution-scanning electron microscope (HR-SEM) equipped with energy dispersive analysis of X-ray (EDAX) to explore the compositional analysis (Model No. NOVANANOSEM450). HR-SEM helps to better understanding the bonding between the matrix and reinforcement phases at comparatively high resolution. The shape of the particles can also be observed through HR-SEM. These optical, HR-SEM microstructure and EDAX spectrum are presented and discussed in Chapter-4.

3.3.3. Density measurement of hybrid composites

Experimental density of copper matrix and developed copper-based hybrid composites was evaluated using Archimedes' principle (ASTM C 693) with the help of the calculated weight of hybrid composites in air (W_a) and in water (W_w) using an electronic balance having an accuracy of 0.001g. However, the theoretical density of the copper-based hybrid composites was evaluated using rule of mixture (ROM). Relative density and porosity of the copper-based hybrid composites were also evaluated by using the equations (3.3) and (3.4), respectively:

$$\text{Relative density (\%)} = \left[\frac{\text{Experimental density}}{\text{Theoretical density}} \right] \times 100 \quad (3.3)$$

$$\text{Porosity (\%)} = \left[1 - \frac{\text{Experimental density}}{\text{Theoretical density}} \right] \times 100 \quad (3.4)$$

3.3.4. Hardness measurement of hybrid composites

The hardness test is a kind of mechanical test for evaluating the property of materials which is used in structural analysis, engineering design and development of materials.

Hardness of any materials is a resistance against permanent deformation such as scratch, wear, indentation and abrasion. The hardness testing is more significant to established correlation between hardness and other material's properties.

The Brinell hardness tester was used to evaluate the hardness of the copper matrix and copper hybrid composites where a ball indenter of 10 mm diameter of hardened steel was used and 500 kg load was normally applied for 10 s on each and every specimen. A low powered microscope was used to measure the indentation diameter left on the test materials. Using the equation (3.5), the Brinell harness number (BHN) was evaluated where; the normally applied load was divided by the indentation's surface area. When the indenter was retracted two diameters of the indentation, d_1 and d_2 , were measured using a microscope with a calibrated graticule and then averaged. Five indentations were made for Brinell hardness measurement at different places and the average of the readings was reported. Hardness measurement was done as per the ASTM standards ASTM E10.

$$BHN = \frac{P}{\frac{\pi D}{2} [D - \sqrt{D^2 - d^2}]} \quad (3.5)$$

where, P – Normally applied load/Test load (kg)

D - Diameter of the ball (mm)

d - Average impression diameter of indentation (mm)

3.3.5. Compression strength measurement of hybrid composites

The compression tests of the copper matrix and copper-based hybrid composites were performed on a hydraulic machine at strain rate of 10^{-4} s^{-1} as per the standard ASTM E9. Compressive strength of the copper matrix and copper hybrid composites was performed on a specimen having length of 24 mm and diameter of 12 mm. To reduce the friction, grease

was placed in between the specimen and plate of the compression machine. The percentage reduction was maintained as 50%. The end surfaces of the specimen were maintained as normal to axis of the specimen.

3.3.6. Measurement of tensile strength of hybrid composites

Understanding the mechanics of material is important for engineers/scientists. The uniaxial tension test provides an easy and efficient method to characterize a response of material to loading. By subjecting a specimen to a controlled tensile movement along the one-axis, alter in dimensions and ensuing load can be recorded to evaluate a stress-strain profile. Then, the plastic and elastic properties of materials can be evaluated from the obtained curve. Therefore, to explore the mechanics of materials and get experience in uniaxial testing, the tensile test was executed on pure copper metal and copper-based hybrid composites, and calculated the ultimate tensile strength (UTS).

The uniaxial tensile test copper matrix and copper hybrid composites was executed on the Instron tensile testing instrument having model no. of 4206, USA and cross head speed of 1 mm/min. The test samples were developed as per the international standard ASTM E8, the schematic and dimensional details of tensile specimen is depicted in Fig. 3.3. The uniaxial tensile test was executed on each material thrice for repeatability of the results and reported the average values of them.

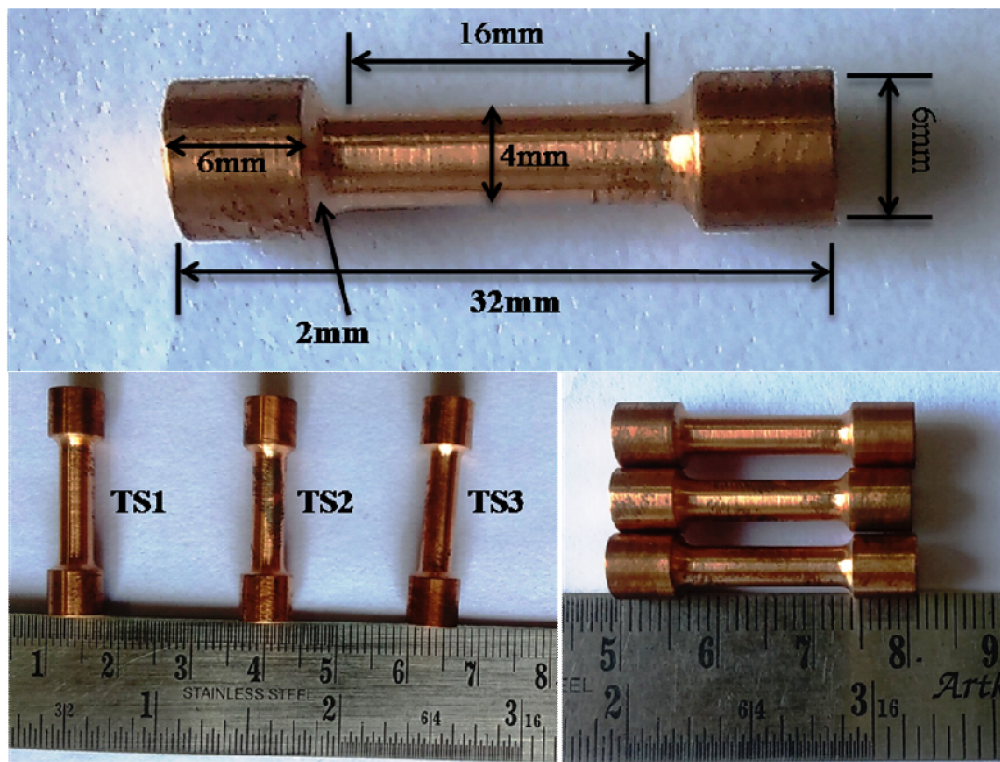
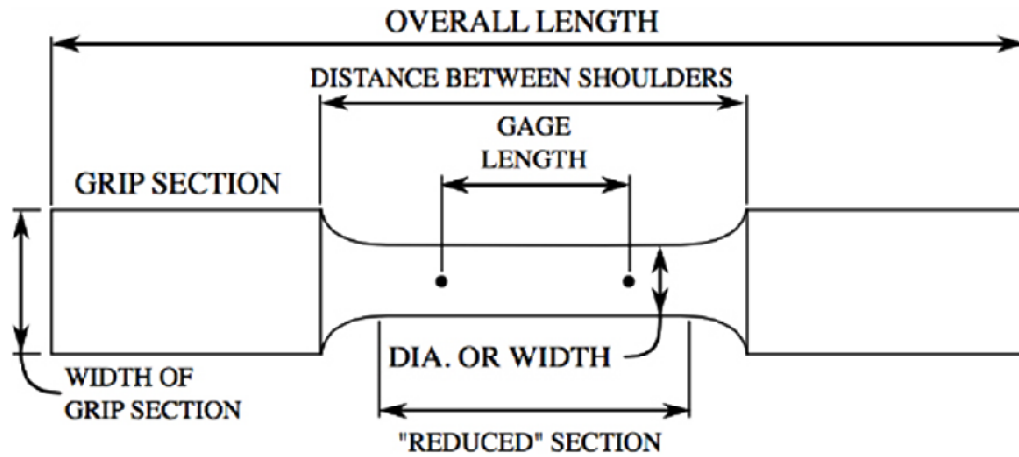


Fig.3.3. Schematic and dimensional measurement of tensile test specimen

3.3.7. Electrical resistivity measurement of hybrid composites

Electrical resistivity is an inherent characteristic which quantifies how strongly a given or particular material opposes the electric current flow, measured as per the standard ASTM B 193-95. A low resistivity infers a material that willingly allows the electric current flow. The electrical conductivity of copper matrix and its hybrid composites were determined

by using the Keithley nano-voltmeter (Keithley Instruments, Cleveland, Ohio, U.S.A., Model no. 2182/2182A). For all the materials, the resistance was measured by using a four-probe method. Sample was mounted on sample holder and proper contact was established by using a silver paste. After making a proper contact, the sample was maintained under well dried conditions for at least 30 minutes. Out of four probes two were meant for current and another two for the voltage. A constant current of 1 A was applied by Keithley source meter (model 2400) and the corresponding voltage was noted by Keithley nano-voltmeter. From current, I , and voltage, V , the resistivity, ρ , and conductivity, σ , of the sample were determined utilizing the following relations:

$$V = IR \quad (3.6)$$

$$R = \rho \frac{l}{A} \quad (3.7)$$

Since, the resistivity was generally presented in terms of international annealed copper standard (IACS). Therefore, the measured resistivity of copper matrix and its hybrid composites was compared with IACS to evaluate the value of electrical conductivity corresponding to 100 % IACS.

3.3.8. Dry sliding friction and wear testing of materials

Dry sliding friction and wear tests of the copper matrix and developed copper-based hybrid composites were performed on pin-on-disc machine (Magnum Engineers, Bangalore, India) as per the standard ASTM G99. Friction and wear tests were performed using cylindrical specimen of 8 mm diameter and 30 mm length that had flat surfaces in the contacting region with rounded edge. The schematic diagram of wear test specimen with dimensional details is depicted in Fig. 3.5. The specimen was stationary against the rotating

counter disc of 100 mm diameter made up of EN-31 hardened steel with 60-62 HRC as provided in the pin-on-disc apparatus. The compositional details of the hardened steel counter disc are mention in Table 3.4. The EN-31 hardened steel is plain carbon steel which is case hardened to achieve a hardness of 60-62 HRC.

Table 3.4. Compositional details of EN-31 hardened steel counter disc

Elements	Si	Mn	C	P	Cr	S	Fe
Wt. %	0.10-0.35	0.30-0.75	0.90-1.20	0.05	1.0-1.6	0.05	Balance

The normal load was applied to the specimen through the lever ensuing in a continuous contact between the counter face and pin surface. The schematic diagram of the pin-on-disc apparatus is shown in Fig. 3.4. The contacting surface of specimens was polished with emery papers of 400, 600, 1200 and 1600 grit size and cleaned with acetone earlier to conducting the wear and friction tests. The tests were conducted under four different normal loads of 9.81, 19.62, 29.43 and 39.24 N and constant sliding speeds of 2.43 m.s⁻¹. Friction and wear tests were executed for a total time of 60 min and the loss of weight was calculated at the intervals of 10 min. To measure the weight loss, the specimen was uninvolved from the holder after 10 min of run, allowed to down to room temperature, cleaned with brush smoothly to remove loose wear debris, weighed and fixed again in exactly in the same manner and position in the holder to avoid the misorientation of its sliding. An analytical balance having an accuracy of 1 x 10⁻⁷ kg was used to weigh the specimen before and after the run. The results of weight loss were converted into volume loss using the density of respective composite as measured by Archimedes' principle. Each test at a particular load and constant sliding speed

was executed thrice for reproducibility of results and the average volume loss after each period of time was reported. The machine had a control panel showing the frictional force. The friction force was noted after every 60 sec and used to calculate the of friction coefficient dividing by normal load.

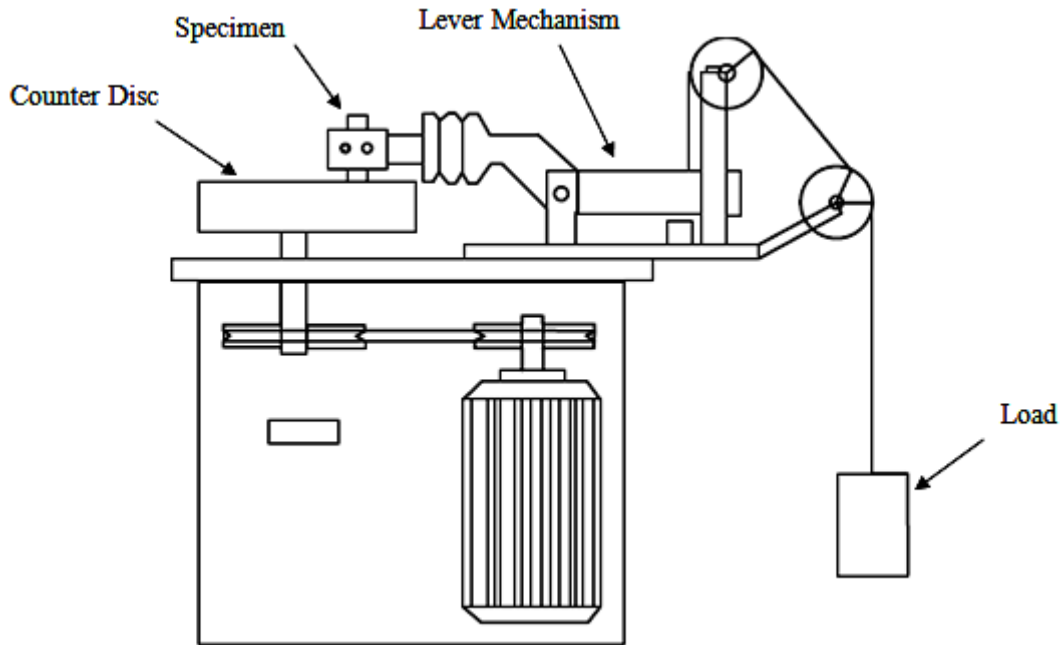


Fig.3.4. Schematic diagram of the pin-on-disc machine (ASTM G99)

Mathematically, friction coefficient can be obtained using the following formula

$$\mu = \frac{F}{N} \quad (3.8)$$

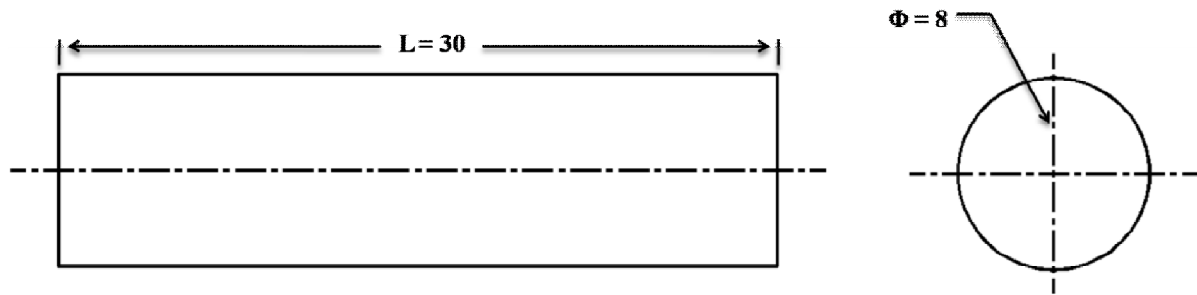
where, μ - Friction coefficient

F - Frictional force

N - Normal load.

This helped in plotting the variation of coefficient of friction with distance of sliding. The results of friction coefficient was then averaged over sliding distance to achieve an

average friction coefficient for a particular test and it has been utilized to analyze the variation of friction coefficient with either load.



All dimensions are in “mm”

Fig.3.5. Schematic diagram of wear test specimen

3.3.9. Examination of worn surfaces

In order to explore the prevailing mechanisms of friction and wear, the dry sliding surfaces of copper matrix and hybrid composites were performed for worn surface after dry sliding wear test. Worn surfaces under low load (9.81 N) and high load (39.24 N) condition were examined by scanning electron microscope (SEM) model no. EVO/18, made by ZEISS, Germany, equipped with energy dispersive analysis of X-ray (EDAX) made by Oxford instruments X-act. Atomic force microscope (AFM) model no. NTEGRA Prima, made by NT-MDT spectrum Instruments, Russia was used to explore the salient features and their micrographs are reported.

