PREFACE

Metal matrix composites (MMCs) have attained growing importance because MMCs are endowed with properties like low fabrication cost and utilization in a variety of industrial applications. The current time is witnessing a high demand for advanced materials that possess higher strength and better electrical as well as thermal conductivity. On account of having a very high electrical and thermal conductivity, ductility and ease of forming, copper lends itself to countless applications, such as automobile radiators, heat sink materials, rocket nozzle, electrodes for resistance welding, electric switches, etc. However, the application scope of copper and its alloys gets restricted due to its inferior mechanical properties. To achieve better mechanical strength, wear resistance, creep and fatigue, ceramic particles reinforced copper based metal matrix composites are produced.

MMCs can be synthesized by various processes, such as powder metallurgy, squeeze casting, compo casting, stir casting, etc. Among these, powder metallurgy (P/M) method is considered to be the most viable due to certain advantages, such as, restricting the unfavorable reaction among the matrix and reinforcement, chances of addition of more volume fraction of reinforcement, uniform distribution of reinforced particles etc. Powder metallurgy processed composites have better properties, provided the reinforcement particulates are dispersed homogeneously in the matrix. If the reinforcement is not properly distributed, the agglomeration of the reinforced particles occurs and that decreases the mechanical properties of the composites. For preventing the agglomeration of the reinforcement particles in the matrix, particularly when the reinforcement particulates are smaller in size, mechanical alloying (MA) method is used to achieve homogeneous distribution of the reinforcement particles.

Despite having high hardness, high chemical stability, high modulus, grain refining effect, better corrosion and wear resistance, TiC particles, as reinforcement in copper based alloys have attracted a little attention from the research community. The Cu-TiC composites can be used for various engineering applications such as resistance welding electrodes, electrical contacts, shipping hulls, etc. However, its potential as a tribological material has not yet been fully explored. Hence, the present investigation is aimed at analyzing the friction and wear behavior of the Cu based composites containing different amounts of TiC under dry sliding conditions using a pin-on-disk wear testing machine.

The thesis has the following six chapters.

Chapter-1 contains the introductory remarks highlighting the technological importance of the problem under investigation.

Chapter-2 begins with a critical review of the existing literature on the techniques of production of composites. It is followed by an exhaustive survey on the various aspects of the friction and wear behavior of the composites in general, and copper based composites in particular. The chapter discusses the processing of copper based composites containing different hard particles as reinforcement and their properties. The chapter further highlights the importance of TiC as reinforcement in copper based composites and illustrates their structural properties, compressibility behavior, tribological behavior with the help of existing literature. The chapter also includes the studies available in published literature showing the application of response surface methodology (RSM) in determining the wear behavior of composites.

Chapter-3 presents the details of experimental procedures followed in the current investigation. In the present study, elemental copper powder (Cu; 99% purity; particle size -200 mesh) and nickel powder (Ni; 99% purity; particle size -200 mesh) have been selected as the matrix alloy whereas titanium carbide (TiC) added in 2, 4, 6 and 8% by weight has been used as reinforcement. The powders were mixed using a high energy ball mill for 2, 4 and 6 h, respectively, in the presence of toluene which acted as process control agent and restricted the formation of intermetallic compounds during milling. Hardened stainless steel vial was used to seal the powders during milling. The BPR was 10:1 and the speed of the mill was set at 400 rpm. The high- energy wet ball milling was stopped after every 20 min and again resumed for 20 min to avoid overheating. The prepared powders were dried for 1 h at 100°C. The powders were consolidated by cold uniaxial press in a rigid cylindrical die at pressures of 250, 450, 650 and 850 MPa. For friction free punch movement stearic acid was used as the die lubricant. The green pressed specimens had 12 mm diameter and 11 mm height. These green pressed specimens were useful in analysing the compaction behavior of the powders. The compacted green specimens were sintered at a temperature of 800, 850 and 900 ^{o}C in an argon atmosphere. The soaking time for the samples was 1 h and heating rate was 5 °C/min. The maximum density was achieved at 850^{o}C and 650 MPa pressure. Hence, different composites, viz., Cu4Ni, Cu4Ni-2 wt.% TiC, Cu4Ni-4 wt.% TiC, Cu4Ni-6 wt.% TiC and Cu4Ni-8 wt.% TiC were finally prepared by sintering at 850 °C for 1 h holding time and the specimens are designated as Cu4Ni, Cu4Ni-2TiC, Cu4Ni-4TiC, Cu4Ni-6TiC and Cu4Ni-8TiC, respectively. The theoretical density of the compacts was estimated using rule of mixture. Density was also measured experimentally by using Archimedes method. Phase determination was done using X-ray diffraction (XRD) employing Rigaku Desktop Miniflex II X-ray diffractometer (Tokyo, Japan). Microstructure of the composites was examined using ZEISS (Model No. EVO/18) Scanning Electron Microscope (SEM) equipped with Energy Dispersive Spectroscopy (EDS). Average particle size of the powders was estimated by using tool IJ1.46. Morphological studies on sintered composite specimens were performed by high resolution-scanning electron microscope (HR-SEM) equipped with energy dispersive spectroscopy (Model No. NOVANANOSEM450). Vickers micro-hardness tester (Model: DHV-1000) was used to measure the micro-hardness at a constant load of 100 g and dwell time of 15 s. Electrical resistivity was measured using four probe technique. Friction and wear tests were conducted according to ASTM G99-05 standard using a pin-on-disk tribometer (Magnum Engineers, Bangalore, India) with a counterface of EN31 steel hardened to 60 HRC at ambient temperature. The worn surfaces of all the materials studied in the present investigation were examined under SEM to explore the operative mechanisms of wear.

Chapter-4 describes the effect of TiC content and technological parameters on the properties of Cu-4wt. % Ni- x wt. % TiC ($x = 2, 4, 6$ and 8 wt. %) composites. Effect of milling time and reinforcement on the morphology of the powders has been investigated by using SEM, EDS and XRD. The compressibility behavior of the milled powders has been determined using various compaction equations; Panelli and Ambrosio Filho, Heckel, and Ge compaction equations. Hardness was evaluated as a function of milling time and reinforcement. The effect of technological parameters on the properties of the composites presented in the chapter show that there is an optimum milling duration, an optimum temperature and pressure of sintering to achieve the optimum density and consequently the hardness in the P/M processed composites. The study also reveals that it is the uniform distribution of second phase rather than its amount which plays a vital role in attaining the desired properties in such composites. This has been confirmed by the higher density and hardness shown by the composite containing 4 wt. % of TiC rather than those containing 6 and 8 wt.% TiC, respectively, because of the agglomeration of particles in these composites.

Chapter-5 presents the results and discussion on the tribological behavior of Cu-4 wt.% Ni matrix composites containing different amounts of TiC (0, 2, 4, 6, and 8 wt.%). Friction and wear behavior has been determined at four different normal loads of 5, 10, 15, and 20 N and three sliding speeds of 0.75, 1 and 1.25 m/s by sliding against a hardened counter-face made of EN31 steel (HRC 60) under ambient conditions, using a pin-on-disk test rig. The surfaces of specimens worn under different loads and sliding speeds have been examined under SEM equipped with EDS to explore the operative mechanisms of wear. SEM studies have also been carried out to understand the nature of wear debris. The coefficient of friction of matrix alloy and composites showed a fluctuating behavior with respect to sliding distance and no certain trend is observed. However, coefficient of friction shown by the Cu4Ni matrix alloy is always less than the composites. Average coefficient of friction has been observed to increase with increasing load. However, composites have revealed the higher values of coefficient of friction in comparison to the matrix alloy. As sliding speed increases coefficient of friction of the matrix alloy as well composites increases. The cumulative volume loss of matrix alloy and composites investigated in this study increased almost linearly with increasing sliding distance. It is observed that cumulative volume loss for the composites is much lower than the matrix alloy. The wear rate, described as volume loss per unit of sliding distance has been estimated from the slope of the variation of cumulative volume loss vs. sliding distance with a linear least square and it has been found to increase linearly with increasing normal load for the composites as well as Cu4Ni matrix alloy, following Archard's law. However, the composites have shown a lower rate of wear than matrix alloy which has been correlated with the estimated hardness. Addition of 4 wt.% TiC has shown better wear performance, which has been attributed to its relatively higher hardness and ability to hold a transfer layer of relatively larger thickness in comparison to other materials. The wear rate of all the specimens is found to increase with increase in sliding speed (0.75, 1 and 1.25 m/s) at 5 N and 20 N. Better wear performance among all the materials investigated is shown by the Cu4Ni-4TiC composite. The value of coefficient of friction is the highest at a sliding speed of 1.25 m/s. The examination of the worn surface of matrix alloy as well composites under SEM showed that wear mechanism for Cu4Ni matrix alloy is a mix of adhesive and oxidative wear whereas it is primarily abrasive for the composites containing hard TiC particles. EDS analyses showed the presence of Fe in the spectra confirming thus the metal transfer from the counter face to the pin's surface. The presence of oxygen peak in the spectra pointed toward the possibility of occurrence of oxidation during the sliding wear. SEM analyses of the worn surfaces of matrix alloy and composites slid under different sliding speeds indicate that as sliding speed increases from 0.75 to 1.25 m/s the wear rate increases for both the matrix alloy as well as for the composites.

Chapter-6 presents and discusses the technique used for modeling the friction and wear behavior of composites using response surface methodology (RSM). The experiments were based on the plan of face centered central, composite design using RSM because face centered, central composite design finds importance over the entire design space due to its ability of high quality prediction. The design was developed and analyzed using MINITAB 16 statistical package. Analysis of variance (ANOVA) results were used to check the adequacy of the developed model and it showed that linear and interaction terms had significant influence on the volume loss while the linear and square terms affected the coefficient of friction of the composites. Mathematical models were developed to investigate the influence of process parameters on the responses at 95% confidence level. Response surface methodology was used for the optimization of the process parameters to reduce the wear and friction responses of composites. Significant linearity between the predicted and measured values confirmed that the developed mathematical models can be effectively used to predict the responses. Developed surface and contour plots depicted the effect of main parameters and their interactions on the responses. Response optimizer option of the MINITAB 16 was used to get the optimum output parameters (volume loss and coefficient of friction). The optimum predicted input parameters to obtain the minimum output of volume loss and the coefficient of friction (COF) are found to be 12.72 N, 2.82 wt. % TiC reinforcement and 1431.82 m sliding distance with a composite desirability of 0.82773.

Chapter-7 presents the major conclusions of the current study conducted to investigate the friction and wear behavior of Cu based composites containing varying amounts of TiC.