TRIBOLOGICAL BEHAVIOUR OF SELF LUBRICATING COPPER BASED HYBRID COMPOSITES

A thesis submitted in partial fulfilment

for the Award of Degree

Doctor of Philosophy

By

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MANISH KUMAR

I would love to dedicate

This

Thesis

To my

Beloved father

Late. Baijnath Rav

ABSTRACT

In the present study, while considering several applications in the fields of sliding contact bearings, electronics, automotive, thermal management, heat exchangers, aircraft, rail transportation, and sliding contact between mating surfaces without lubrication, novel materials are always needed for a wide range of technical applications.

In the past few years, copper metal matrix composites have become more demanding and popular from an engineering and technological point of view. Copper is an incredibly valuable material for the application above due to its wide variety of engineering applications. Copper is employed in numerous technological domains because of its exceptional resistance to corrosion, excellent ductility, and great conductivity of heat and electricity when used as a matrix material.

To consider the ongoing demand for these materials, low-density copper metal is chosen as the matrix for the development of various copper-based hybrid metal matrix composites with improved performance. For this scientific and technical purpose, the powder metallurgy technique is considered the most suitable for developing copper-based hybrid metal matrix composites in the current investigation. This technique offers several advantages, including higher precision in forming the product and homogeneous distribution of particles. Additionally, it utilizes lower temperatures compared to other processing methods. The selection of hard ceramics reinforcing phases, such as (B_4C) and (SiC) particles, is supported by their ability to refine grain, their hardness, chemical stability, specific strength, good corrosion and wear resistance, and the presence of solid lubricant (Gr), which enhances wear resistance qualities. Adding chromium (Cr) to the matrix will improve the bonding between the matrix and the reinforcements and boost the wettability between copper and ceramic reinforcement. This investigation aims to make copper matrix composites by utilizing B4C,

SiC, and graphite in the form of carbon reinforced through the powder metallurgy technique. Two different composites Cu-B_4C -Gr and Cu-SiC-Gr assigned as Cu-0Cr-0Gr-0B_4C (CU01/C1), Cu-2Cr-1.5Gr-1.5B_4C (CU02/C2), Cu-2Cr-3Gr-3B_4C (CU03/C3), and Cu-2Cr-4.5Gr-4.5B_4C (CU04/C4), and Cu-0Cr-0Gr-0SiC (S1), Cu-2Cr-1.5 Gr-1.5SiC (S2), Cu-2Cr-3Gr-3SiC (S3), and Cu-2Cr-4.5Gr-4.5SiC (S4) respectively, have been developed by powder metallurgy technique.

Following several characterizations, it is found that the produced hybrid metal matrix composites based on copper have superior mechanical and tribological characteristics than copper matrix because of uniform dispersion and high wettability of the reinforcing Phases found in the matrix of copper. Therefore, a variety of engineering applications, including sliding contact bearings, the automotive and electronic industries, heat exchangers, thermal management, aerospace, and rail transportation, as well as sliding contact between mating surfaces without lubrication, can successfully use the developed copper-based hybrid metal matrix composites.

The copper-based hybrid metal matrix composites have undergone many characterizations to investigate their microstructural, physical, mechanical, wear, and friction characteristics. The microstructural observations have been conducted using a range of characterizing and analytical techniques, including X-ray diffraction (XRD), scanning electron microscopy (SEM), high-resolution transmission electron microscopy (HRTEM), energy dispersive analysis of X-ray (EDAX), and high-resolution scanning electron microscope (HR-SEM). The effect of the reinforcements was analysed by conducting a detailed investigation of the hardness, compressive strength, density, porosity, friction, and wear characteristics. Parametric analysis is crucial in the research of metal matrix composite materials since it allows for the examination of different behaviours exhibited by these materials during their evolution. Friction and wear tests were performed in accordance with the ASTM G99-05 standard. A pin-on-disc tribometer was used, with the pin rubbing against a counter face made from EN31 steel that had been hardened to a hardness of 62 HRC. The tests were completed at ambient temperature. An evaluation of the deteriorated materials deteriorated surface was carried out during the wear test. The deteriorated surface of all the composites analyzed in the present study was evaluated using a scanning electron microscope (SEM) equipped with energy-dispersive X-ray spectroscopy (EDS) equipment. The purpose was to investigate the mechanisms of wear. In order to further scientifically and technically understand wear mechanisms, the deteriorated surfaces of the specimens were examined using a Scanning Probe Microscope (SPM).

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List of Abbreviations and Symbols

Abbreviation	Description
MMCs	Metal matrix composites
pMMCs	Particle-reinforced metal matrix composites
PM	Powder metallurgy
PMCs	Polymer matrix composites
CFRP	Carbon fiber-reinforced polymer
GFRP	Glass fiber-reinforced polymer
CMCs	Ceramic matrix composites
PVD	Physical vapor deposition
CVD	Chemical vapor deposition
SEM	Scanning electron microscope
HRSEM	High resolution scanning electron microscope
TEM	Transmission electron microscope
CNTs	Carbon nano tubes
EDS	Energy dispersive spectroscope
P/M	Powder metallurgy
GPa	Giga pascal
MPa	Mega Pascal
ASTM	American society for testing and materials
m/s or $m.s^{-1}$	Meter per second
N	Newton
nm	Nano meter
wt%	Weight percentage
vol.%	Volume percentage
R_a	Average roughness
R_q	Root mean square roughness

R_z	Average maximum height from individual peak to valley
g	gram
kg	Kilo-gram
mm	millimetre
d	The inter planner spacing
JCPDS	Joint committee on powder diffraction standards
θ	The incident Bragg's angle
λ	The wavelength of the X-ray
n	An integer representing the order of the diffraction
ϵ	Lattice strain
k	The shape factor
hkl	Miller indices
EDAX	Energy dispersive analysis X-ray
VHN	Vicker hardness number
P	Load
D	Diameter of the ball
d	The average impression diameter of indentation
BN	Boron nitride
B_4C	Boron carbide
Cr	Chromium
TiC	Titanium carbide
SiO_2	Silicon oxide or silica
SiC	Silicon carbide
Al_2O_3	Alumina
h-BN	Hexagonal boron nitride
Gr	Graphite

PREFACE

The development of irregularly reinforced metal matrix hybrid composites has attracted more attention recently because of their isotropic characteristics and satisfactory effectiveness, all at comparatively inexpensive production costs. Applications for copper-based hybrid metal matrix composites continue to expand in general engineering, automotive, and manufacturing. Due to its outstanding electrical and thermal conductivity and malleability, copper is widely used in various industries. It is employed in applications such as sliding contact bearings and motor brushes and as a lubrication-free sliding contact between mating surfaces. Even so, the commercial application of copper and its alloys is limited due to their inadequate mechanical and tribological characteristics. Hard Ceramic and self-lubricating particles are used as reinforcement for copper-based hybrid metal matrix composites to improve specific strength, reduce wear and friction, and increase hardness. Copper-based metal matrix composites can be manufactured using various reinforcements, including graphite, graphene, molybdenum disulfide, and others. The composite benefits from including graphite, a kind of carbon, due to its exceptional self-lubricating properties, low friction, and high heat stability. The composite material's enhanced mechanical, thermal, and tribological properties are achieved by mixing copper with graphite. Copper graphite composites are used in electrical engineering due to their better conductivity and in sliding electrical contacts and bearings requiring strong lubricating properties. The unique combination of copper and graphite in these composites provides a well-balanced set of characteristics suitable for specific engineering applications.

By using hard ceramic particles (B_4C) and (SiC) as well as solid lubricant (Gr), the wear resistance and hardness of copper composite can be enhanced. However, adding Cr to the matrix will improve the bonding between the matrix and the reinforcements and increase the wettability between copper and ceramic support. This investigation aims to create copper-based hybrid metal matrix composites by utilizing B_4C, SiC, and carbon in graphite through the powder metallurgy route. This study aims to identify the most suitable copper-based hybrid metal matrix composites for various engineering applications, such as electrical contacts, bushings, electronic and automotive sectors, and motor brushes. Copper graphite composites are used in sliding electrical contacts and bearings, which require high wear resistance properties, and high hardness, where their better conductivity is helpful. The carefully balanced mix of copper, boron carbide, silicon carbide, and graphite in these composites provides a set of properties suitable for certain engineering applications.

The current work consists of seven chapters.

CHAPTER-1: This chapter addresses copper and copper-based hybrid metal matrix composites, which are classified depending on their matrix and reinforcement. The study additionally examines composite materials' mechanical, physical, and tribological properties and subsequently explores the practical applications of these materials.

CHAPTER-2: The literature review in the following section of the thesis provides a comprehensive understanding of recent improvements in the fabrication of copper-based hybrid metal matrix composites using powder-based techniques. The metallurgical route and strengthening impact within the copper matrix. The chapter discusses several kinds of composites, with a particular concentration on hybrid metal matrix composites that contain copper. The discussion covers the benefits of composites, their various uses, and the methods used for developing composites using different matrices and materials. The powder metallurgy approach has been thoroughly examined, including a detailed discussion of the processes involved. In addition, the chapter explores the complexity of many forms of wear and friction, as well as their fundamental mechanisms. Developing materials suitable for use in electrical contacts, bearing materials, automotive industries, and motor brush applications requires careful consideration of the matrix, reinforcement materials, and fabrication procedure. The selection process is vital to guarantee that the finished materials exhibit exceptional strength and efficient resistance to wear.

Hence, for the current investigation, Boron carbide (B_4C), silicon carbide SiC, and graphite have been selected as the reinforcing elements for the copper matrix composite. Boron carbide (B_4C) and silicon carbide (SiC), which are highly resistant ceramic materials, and graphite, serve as solid lubricants in copper matrix composites. The powder metallurgy process has been adopted for the production of composites. This work aims to create copper-graphite- Boron carbide and copper-graphite-silicon carbide composites with enhanced tribological properties to improve the durability and effectiveness of particular materials.

CHAPTER-3: This section of the thesis comprehensively explains the experimental processes carried out during this phase of studies. The current work utilized industrial copper as its main constituent and incorporated chromium along with hard ceramic materials and self-lubricating particles as reinforcements to create copper-based hybrid metal matrix composites through the powder metallurgy approach. The characterization of composites was accomplished using X-ray diffraction (XRD). An X-ray diffraction study was performed on both copper matrix and composite specimens to ascertain the present lattice strain, crystallite size, and phases. The Rigaku Desktop Miniflex II X-ray diffractometer, located in Tokyo, Japan, was utilized to analyze X-ray diffraction (XRD). The X-ray diffraction

(XRD) analysis was performed using a Cu-Kα radiation source filtered with a nickel (Ni) filter. The X-ray wavelength used was 1.5406Å. The XRD was operated at a voltage of 40 kilovolts (kV) and a current of 30 milliamperes (mA). Scanning electron microscope (SEM) and energy-dispersive X-ray spectroscopy (EDX) are often used for elemental analysis. By identifying and examining the unique X-rays produced by the specimen, EDX can map the elements throughout the composites' surface and ascertain their elemental composition. high-resolution Transmission Electron Microscope (HRTEM) investigation combined with selected area electron diffraction (SAED) and energy-dispersive X-ray spectroscopy (EDS: TEAM EDS SYSTEM with Octane Plus SDD Detector Company: EDAX Inc.). SAED makes it simple to identify and determine crystallographic orientations and crystal structures. The American Society for Testing and Materials (ASTM) B328 standard was followed in measuring the density of sintered specimens using the Archimedes principle.

The hardness of the created composite specimens was determined by utilizing a Vickers hardness tester (Model HTA RVM 50) with an applied load of 5 kgf and a dwell time of 10s. Compression tests were conducted in accordance with ASTM standard E9 to investigate the composite material's behaviour to compressive force. The screw-driven InstronTMTM Universal Testing Machine was used to perform the compression tests, and the initial strain rate was set to 10 s-1 at room temperature. Using pin-on-disc tribo-testing equipment (as specified by DUCOM Instruments India), the tribological behaviours of produced composites and unreinforced copper were examined under dry sliding conditions. To investigate the wear mechanisms, the samples' deteriorated surfaces were examined using a Atomic Force Microscopy (AFM) that NT-MDT Service & Logistics Ltd. supplied.

CHAPTER-4: It covers the fabrication of composites with different wt.% of graphite (Gr) and boron carbide (B_4C) particles in copper-based hybrid metal matrix composites, as well as the morphological analysis, XRD phase confirmation, and impact of the wt.% of the two particles on the hardness characteristic. Particles are distributed uniformly, according to Scanning electron microscope (SEM) analysis. With the addition of boron carbide (B_4C) and graphite, the hardness of the produced composites increased rapidly compared to their matrix. Hardness rises rapidly in the copper matrix up to 3 wt.%B_4C + 3wt.% Gr reinforcement, after which it falls but stays higher than the matrix.

As the applied load and sliding velocity rise, along with the wear rate and coefficient of friction (COF). As more boron carbide (B_4C) and graphite were added, wear rate and coefficient of friction (COF) decreased. the least amount of wear was seen in the 9 wt.% of reinforced composites. The deteriorated surface investigation was conducted using a Scanning electron microscope (SEM) coupled with EDS and AFM analysis. Delamination wear is seen at smaller sliding distances and applied loads, whereas abrasive, oxidative, and severe delamination modes of wear take place at higher loads and sliding velocities. The AFM analysis showed that the topography became smoother when the boron carbide (B_4C) and graphite content increased and the average surface roughness decreased.

CHAPTER-5: This chapter covers making composites in copper-based hybrid metal matrix composites with varying wt.% of graphite (Gr) and boron carbide (B_4C) particles. It also covers morphological analysis, XRD phase confirmation, and the two particles' weight percentage effect on the hardness characteristic. The characteristic lattice fringes of graphite and boron carbide are shown in the high-resolution Transmission Electron Microscope (HRTEM) image. From Scanning electron microscope (SEM) analysis, particles are uniformly dispersed. Compared to its matrix, the hardness of the resulting composites quickly increased with the addition of boron carbide (B_4C) and graphite. Up to 6wt.% reinforcement, hardness in the copper matrix increases quickly. After that, it declines but remains greater than the matrix. The coefficient of friction (COF), wear rate, and sliding velocity increase with the applied load. Wear rate and COF dropped with increasing boron carbide (B_4C) and graphite additions. The 9w.%t of reinforced composites showed the least degree of wear. The deteriorated surface was evaluated with a SEM in conjunction with AFM and EDS analysis. Whereas abrasive, oxidative, and severe delamination modes of wear occur at higher loads and sliding velocities, delamination wear is observed at smaller sliding distances and applied stresses. According to the AFM research, the average surface roughness decreased, and the topography got smoother as the B_4C and graphite content increased.

CHAPTER-6: This chapter discusses the fabrication of composites using varying wt.% of graphite (Gr) and silicon carbide (SiC) particles in copper-based hybrid metal matrix composites. It also includes an analysis of the morphology, confirmation of XRD phases, and the influence of the wt.% of each of the particles on the hardness characteristics. The SEM analysis reveals that particles are evenly distributed. The incorporation of SiC and graphite resulted in a significant enhancement in the hardness of the generated composites compared to their matrix. The hardness of the copper matrix increases significantly up to a concentration of 6wt.% for both reinforcing materials. Beyond this point, the hardness decreases but remains higher than that of the matrix alone. As the applied load and sliding velocity increase, so do the wear rate and coefficient of friction (COF). The addition of additional SiC and graphite resulted in a decrease in both the wear rate and the coefficient of friction (COF). The composites are reinforced with 4.5wt.% SiC and 4.5wt.% Gr showed the lowest level of wear.

An examination of the deteriorated surface was performed utilizing a Scanning Electron Microscope (SEM) coupled with Energy Dispersive Spectroscopy (EDS) and Atomic Force Microscopy (AFM) analysis. Delamination wear occurs at lower sliding distances and applied forces, while adhesive, abrasive, oxidative, and severe delamination wear modes occur at greater loads and sliding velocities. The AFM research revealed that the topography exhibited a smoother texture when the SiC and graphite content increased and the average surface roughness reduced.

CHAPTER-7: The primary findings on the morphological, mechanical, and tribological characteristics of the Copper-Graphite- boron carbide and Copper-Graphite- silicon carbide composites, which were covered in various thesis chapters, are provided in this section. Furthermore, the future scope has been further outlined.