

**Fabrication, Characterization and Testing Of Copper Reinforced Metal Matrix
Composite and Investigation of the Deformation Behavior through Hot Extrusion
Process**



Talile Chirkena Bali

A Thesis Submitted to Department of Mechanical Engineering
School of Mechanical, Chemical and Materials Engineering

Presented in Partial Fulfillment of the Requirement for the Degree of Master's in
Manufacturing Engineering

Office of Graduate Studies
Adama Science and Technology University

June, 2024

Adama, Ethiopia

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Advisor: Dr. Devendra Kumar Sinha

Co-advisor: Mr. Mengistu Gelaw

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DECLARATION

I hereby declare that this Master Thesis entitled “**Fabrication, Characterization and Testing of Copper Reinforced Metal Matrix Composite and Investigation of the Deformation Behavior through Hot Extrusion Process**” is my own work and has not been submitted to any university for similar purpose. The references used in this thesis are duly recognized by proper citations.

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We, the advisors of this thesis, hereby certify that I have closely advised the student while developing this thesis and read the draft thesis entitled “**Fabrication, Characterization and Testing of Copper Reinforced Metal Matrix Composite and Investigation of The Deformation Behavior through Hot Extrusion Process**” prepared under my guidance by Talile Chirkena. Therefore, I recommend the submission of the thesis to the department for further review and evaluation.

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APPROVAL PAGE

We, the advisors of the thesis entitled “**Fabrication, Characterization and Testing of Copper Reinforced Metal Matrix Composite and Investigation of the Deformation Behavior through Hot Extrusion Process**” and developed by Talile Chirkena, hereby certify that the recommendation and suggestion made the board of examiners are appropriately incorporated into version of then thesis.

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APPROVAL BY BOARD OF EXAMINERS

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ACKNOWLEDGMENT

First of all I would like to thank God for this opportunity and who has granted countless blessing, knowledge, and to finishes the thesis work for helping me in every situation. Next, my mentor, I am very grateful to Dr, Devendra Kumar Sihna (Associate Professor) for telling me the research guidelines, constructive suggestions and facilitating the necessary things for the study. And also, I would like to say my co-advisor Mr. Mengistu Gelaw (MSc) Mechanical Design and Manufacturing for his giving continuous support and supported laboratory directions.

Finally, I would also like to thank Mr. Kanate Sanbaba and Mr. Biruk Sisay from material science and engineering department for their technical support and encouragement during experimental work. I would also like to thank my very close friends who shared their helpful ideas and cooperation in completing this thesis: Bayush Lemma, Hanna Tadessa and Sisay Diriba. I would like to thank the people I did not mention above, but who participated in my research work.

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LIST OF ACRONYMS AND ABBREVIATIONS

ANOVA	Analysis of variance
ASTM	American Society for Testing and Materials
BBD	Box Behnek Design
CeO ₂	Cerium dioxide
CMMC	Copper Metal Matrix composite
DOE	Design of Experiment
GA	Genetic Algorithm
GRA	Grey Relational Analysis
Gr	Graphene
HCL	Hydrochloric acid
MMC	Metal Matrix Composite
RSM	Response Surface Methodology
S/N	Signal to Noise Ratio
SEM	Scanning Electron Microscopy
SiC	Silicon Carbide
TiC	Titanium carbide
TiO ₂	Titanium oxide
OM	Optical Microscope
WC	Tungsten Carbide
XRD	X-ray diffraction

ABSTRACT

Copper Metal Matrix Composites (CMMCs) are a class of materials that are widely used in the automotive and aerospace industries due to their exceptional properties. This thesis work was synthesized copper reinforced composite materials through stir casting process as well as to examined numerically and experimentally investigated the deformation behavior for the reinforced composite material with different weight percentage of CeO_2 , SiC, and Gr nano particles. The investigation of distribution of the reinforced particles over the matrix as well as grain size determination has been carried out through Optical Microscopy (OM). The hardness of the developed samples has been measured through Vickers hardness tester and improvement of 51.73% has been found and wear rate has been investigated through Pin-on-Disc equipment. The wear morphology of the worn samples was done by Scanning Electron Microscopy (SEM) in order to determine the mechanism of wear behavior. The investigation of compressive strength has been carried out on Universal Testing machine and improvement to 393.758MPa has been observed. The Grey Relational Analysis method has been used for the selected (81%Cu-7.5%SiC- CeO_2 -4%) for secondary operation (hot extrusion process). The DEFORM 3D software was used for the numerical simulation of hot extrusion at varies parameters. The extrusion ratio of (2, 3, 4), ram speed (1, 2, 3) mm/s and deformation temperature of (850, 900, 950)°C have been chosen to investigate the output numerical responses like load, effective stresses and damage value. The Response Surface Methodology under Box Behnken method in Design expert was used to the predicted the combined damage, extrusion load and effective stress from simulation corresponding to input extrusion parameters. The global optimization of input extrusion parameters have been done through Genetic Algorithm for the minimum values of load, effective stresses and damage value. The post deformation characteristics like Hardness of the developed material were improved by 70.74% after extrusion and the porosity was reduced by 0.073%. The proposed research on Copper Metal Matrix Composites (CMMCs) aims to address the challenges faced by industries and researchers working in this field such as electronic packaging and contacts, resistance welding electrodes, heat exchangers.

Keyword: Copper Metal Matrix Composite, Casting, Extrusion, Evolutionary Algorithm, Box Behnken Design.

CHAPTER ONE

INTRODUCTION

1.1. Background of the Study

Composite materials are currently widely employed in the aerospace, automotive, road transportation, maritime, and medical areas for a range of applications due to their low weight, strength, high stiffness, and biocompatibility. Since they offer good mechanical qualities, a low coefficient of thermal expansion, and high thermal conductivity, composites with high reinforcement contents are being researched and employed in the electronics sector. Although metal matrix composite materials have higher specific strength, stiffness, and operating temperatures than monolithic materials, their production costs are still high, and they are only used in applications where weight reduction is crucial, such as in the aerospace and defense industries (Cuevas et al., 2018).

Metal matrix composites (MMCs) are a class of man-made materials that are created by mixing specific content reinforcements, like fibres, whiskers, particles, in to alloy matrix materials. These materials display excellent temperature performance, high compressive strength, the best conductivity of thermal property, good electrical conductivity, a controllable thermal expansion coefficient, and a high specific modulus, as well as designability and machinability (Lynch and Kershaw, 2018). Metal-matrix composite materials can be created using a variety of methods with desired the quantity of reinforcements and distribution of the reinforcements components (particles and fibres), the matrix alloy as well as the application are the main considerations when choosing an appropriate process engineer.

Copper (Cu) is frequently used in high-end application domains including integrated circuits, electric switches, and electronic packages due to its particular properties like strong electrical conductivity, fatigue resistance, workability, and corrosion resistance (Salvo et al., 2019). Copper alloy has good electrical resistance, corrosion properties but, poor mechanical properties which have low yield strength and soft material when compare relatively with other materials. That means it has limited mechanical property so, for improve mechanical property and to get benefit for modern application have to add reinforcement in order to form copper metal matrix composite. Copper alloy with reinforcement is far better than base alloy by mechanical properties like tensile strength, hardness, impact strength, wear strength and corrosion resistance is improved by adding

reinforcements (Gautam et al., 2018). Copper based metal matrix composite (CMMC) attracts the multiple applications due to their good electrical, thermal and mechanical properties (Somani et al., 2018). As a result, by incorporating hard ceramic particles like Al_2O_3 , SiC, TiC, and ZrO_2 into the metal matrix, materials' resistance to damage and their strength can be increased (Jamwal, Mittal, et al., 2020). When a material is needed for maritime applications, it is important to consider its resistance to wear and corrosion, particularly in the case of structural support elements like bearings, bushes, and blocks. This enhances mechanical qualities combination of copper alloy and graphene (Venkatesh and Rao, 2018). The outcomes show that the Cu-SiC composite's wear resistance capability is improved, and the SiC reinforcement lowers the coefficient of friction (Somani et al., 2018). The hardness and wear resistance of GO-Cu/CeO₂/Cr composites can be greatly increased by the addition of Cr and CeO₂ nanoparticles can be added to materials to correct dislocations, create dislocation entanglement, and increase material strength (Li et al., 2022).

1.2.Fabrication of Copper Metal Matrix Composite through Stir Casting

Copper matrix composites can be made in various ways and some of these methods are composite casting, composite casting, rio casting, ultrasonic assisted casting, stir casting, in-situ, powdered iron techniques are produced for various applications (Sridhar et al., 2020). A flowchart of the stir casting methodology has been created to provide a general overview of the metal matrix composites casting process (Sharma et al., 2015). Thus, the effect of the stir casting parameter on the uniform distribution of particles and the stir casting process in a crucible (Kumar et al., 2019). Stir casting is a practical and affordable way to make metal-matrix hybrid composites with a copper basis (Singh and Gautam, 2017).

1.3.Simulation and optimization the parameters of hot extrusion

Simulation is the process of creating a virtual model of some system, process, and phenomenon to make informed decision without the need for physical experimental. DEFORM 3D software was used to simulate for FEA in order to obtain die geometry and actual extrusion parameters (Jadhav and Dabade, 2016). In the extrusion process, modeling and optimization techniques are utilized to reduce the number of trial runs and obtain the ideal load and effective stress condition. In extrusion processes, a variety of modeling techniques are employed, including regression-based modeling, artificial neural networks

(ANN), fuzzy modeling, and response surface methodology (RSM), Taguchi methodology (TM), and genetic algorithms (GA).

1.4.Copper Metal Matrix Forming Through Hot Extrusion

Extrusion is a deformation process used to produce bars, tubes, and products from light and heavy metals, steel, as well as metallic composite materials. After fabricated copper metal matrix composite have to forming into desired shape for different application. The hot extrusion is certified as a valid method to disperse the particles due to the high shear stresses inherent in the extrusion process (Wang et al., 2019). The thermal conductivity of the extruded Cr-coated diamond/Cu composites was increased to with improved interfacial bonding (Zhang et al., 2018). It has been discovered that extruded composites show lower wear rates and friction coefficients when compared to both sintered and extruded composites. (Senthil Kumar et al., 2016). No extended cracks were seen at the worn subsurface of hot-extruded composites, despite the worn subsurface exhibiting severe plastic deformation. This could be attributed to the enhancement of the Ag-Cu/MoS₂ interface, the rearrangement of MoS₂, and the increased bonding strength between the silver particles following the hot extrusion process (W. Chen et al., 2016). The extrusion pressure is influenced by the die angle, cross-sectional reduction, extrusion speed, billet temperature, and lubrication (Yadav et al., 2018).

When compared to copper alloy with copper alloy metal composites, copper metal matrix composites are superior for improving mechanical and tribological behavior to boost strength, hardness, toughness, and corrosion resistance all of which are crucial for application areas. Because copper naturally combines with other alloys and reinforcement to improve metal properties, this composite offers the finest properties. Stir casting can be used to create copper composite. This composite can be formed into the desired shape after fabrication by hot extrusion. Improve material properties during the hot extrusion process. However, as temperature rises, ductility increases and strength decreases, therefore to boost strength, add SiC and minimize friction, by Gr.

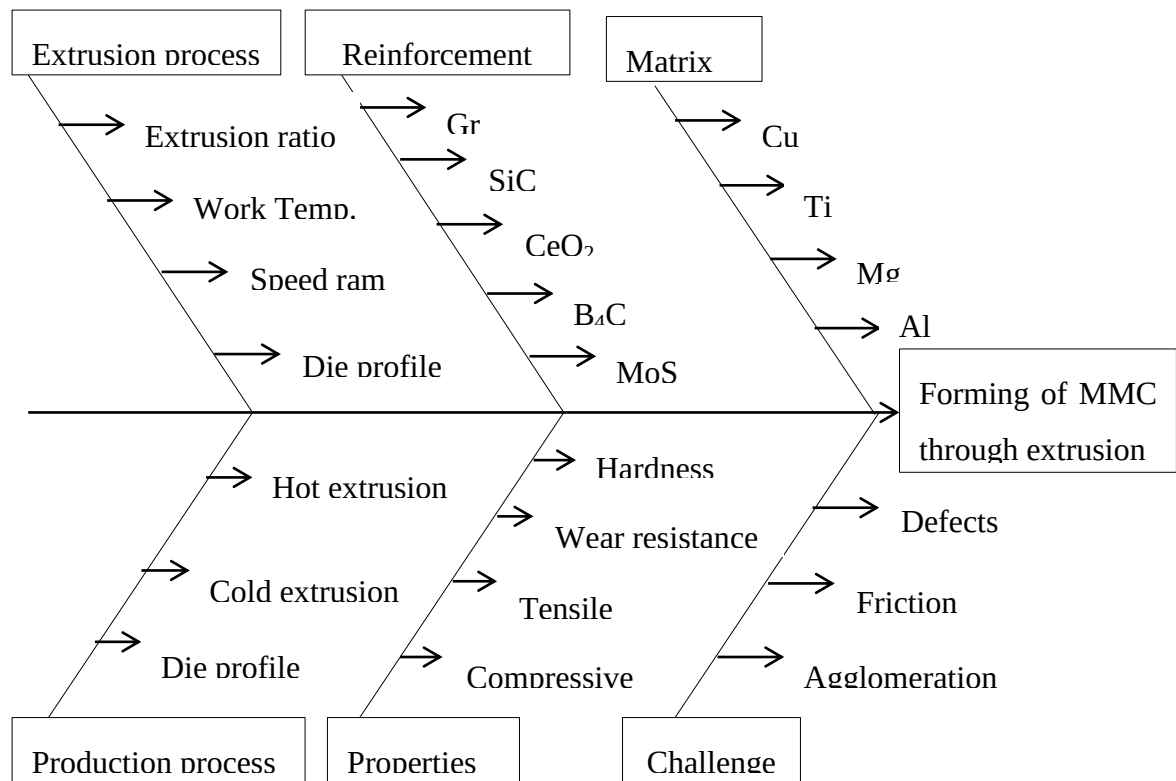


Figure 2.1 Fishbone diagram deform of MMC through extrusion process

1.5.Problem statement

For many engineering applications, including those in the automotive, medical, marine, and aerospace industries, there are many different engineering tools that are of great demand and profit. Circuit breakers, maritime, medical, aerospace, and electrical contact materials all heavily rely on copper. However, these sectors still face difficulties in terms of strength to weight ratio, ductility, resistance of corrosion with wear resistance. Hence it is very necessary for the scientific community to overcome these challenges and improve upon the properties of these alloys thereby making it suitable for various applications. Copper metal matrix has high ductility, hardness, corrosion resistance and thermal conductivity. Therefore, copper composite is one of the most essential engineering materials. Although copper has the best qualities, it does not have all the qualities to make one desirable as a specific environmental service. Although some works have been done to solve the above problem, further research is needed to address the above mentioned limitations especially the reinforced copper metal matrix and its manufacturing process requires more research. Copper often plays an important role in the desired areas, but pure copper alone is not sufficient because it does not have all the characteristics and sometimes does not provide quality service where it is needed. Furthermore, to convert these materials

into usable shapes and sizes, it is necessary to strengthen the processing techniques, strengthen the main service area by adding reinforcement to produce through stir casting and reduce the problem of damping through stir casting and naturally adding secondary stages such as extrusion. Therefore, a study on the fabrication and deformation behavior of CeO₂, SiC, and Gr (graphene) reinforced Cu alloy (OFC) was conducted to improve its properties through stir casting to produce and through extrusion process to solve the problem moved above.

1.6.Objectives

1.1.1. General Objectives

This study aims to fabricate, characterize, and test copper-based reinforced metal matrix composites and to evaluate the performance before and after deformation through hot extrusion process.

1.1.2. Specific Objectives

- To synthesis of reinforced Copper Metal Matrix composite at various weight percentages of reinforcement particles.
- To study phase, microstructural evolution and surface morphology of developed samples.
- To investigate physical, mechanical and tribological properties of developed samples.
- To investigate optimum load, effective stresses and damage value of developed material and conclude optimum input parameters extrusion process.
- To test hardness, compressive, corrosion and microstructural of extruded samples.

1.7.Significant of the study

- To gain an understanding of how a copper metal matrix composite is made, as well as to improve the material's tribological behavior and improved with mechanical properties of developed samples by increase hardness, compressive strength and reduced wear rate.
- When made using an enhance stir casting method and a final extrusion step to produce the composite with the optimum properties.

- Copper metal matrix composite can be extruded into any shape to be supplied to businesses in the necessary application area.
- To help other researchers understand this work and use it as a resource for their own research.

1.8. Scope of the study

This study focuses on analyzing the deformation behavior of an extruded copper metal matrix composite and optimizing the process parameters to improve the material's characteristics and develop a composite that is both light and strong. In this research, a copper composite is produced and its mechanical characteristics such as hardness, tensile strength, impact resistance, and extrusion ratio are evaluated. Also find its tribological behavior, including friction coefficient and wear rate.

1.9. Limitation of the study

There are many challenges to overcome when completing this thesis. Stir casting process good process for homogenous distribution of reinforcement particles mixed into metal matrix but, it has challenge of mixing process may tend to agglomerate due to Van der Waals forces or interfacial tensions. Also lacks the necessary tools for producing composite materials, performing tests, and manufacturing products. No available standard machines/equipment's for test specimen, especially for composite materials properties and those equipment's can't occur at one place, so its delay long time.

1.10. Research motivation

The motivation behind this research project is the desire to fabricate sample with superior properties and performance. The corrosion resistance and high strength application of Copper matrix composites makes it attention application area of the heat exchanger, bearing. Based on the above motivation, the study aimed to copper composite matrix with SiC, CeO₂, and Gr by stir casting techniques. The study also focus to study the combined effects of these multiple reinforcements on the composite's performance. Additionally, the study is further motivated by the benefit of incorporating secondary operations like deformation to improve microstructure, and performance characteristics.

1.11. Thesis organization

This thesis has five chapters. First chapter discussed introduction of composite material, objective, scope, limitation of this thesis. Second chapter discussed literature review which

related with this thesis. Third chapter, studied methodology about fabrication of copper metal matrix composite, raw material, method of fabrication, parameters, and testing samples with use of equipment type. Fourth chapter discussed experimental result of the developed sample through stir casting process and experimental result of mechanical properties of samples through hot extrusion process. Fifth chapter discussed conclusions of experimental results and recommendation for future works.

CHAPTER TWO

LITERATURE REVIEW

2.1. Introduction

Many studies have been conducted on composite fabricated through extrusion process; results for industrial purposes have been done to the extent that it is still achievable. The large amount of research studies of copper metal matrix composite are explained in this chapter. In order to get an improved understanding of some of the areas of this thesis work, a review of various academic works was helpful. The results several investigations on the composition, production, and properties of composite materials conducted by foreign researchers. In the subtitle of composite material behaviors, advantages, matrix and reinforcement with its fabrication selected parameters; this chapter primarily discussed previous research related to metal matrix composite. The fabrication technique for metal matrix composites (MMCs) suggests stir casting process parameters and recent developments in copper metal matrix composites. This chapter also covers extrusion as a supplementary procedure used after stir casting to further enhance mechanical properties. Discussion of parameters and their impact on output occurred during the casting process.

2.2. Composite

A material that is composed of two or more constituent materials and each of which has unique chemical or physical properties is referred to be a composite material. In order to give the completed product qualities that are distinct from those of the individual constituents, these constituent parts are mixed. Composite materials are divided into two categories: base materials and filler materials. Due to their excellent wear and corrosion resistance, high strength, weight-to-strength ratio, and hardness, composite materials are widely used in the construction of automobile, medical device, and aircraft constructions. A composite's properties are significantly influenced by the distribution, interactions, and quality of the individual materials that make up the composite. The composite properties may be the volume percent sum of the separate characteristics of the pieces. The filler material is present as sheets, bits, particles, fibres, or whiskers of natural or synthetic material, and the foundation substance that binds or holds the filler material in structures is referred to as a matrix or binder material. (D. K. Rajak et al., 2019).

Composite materials are categorized according to the matrix and reinforcing particles.

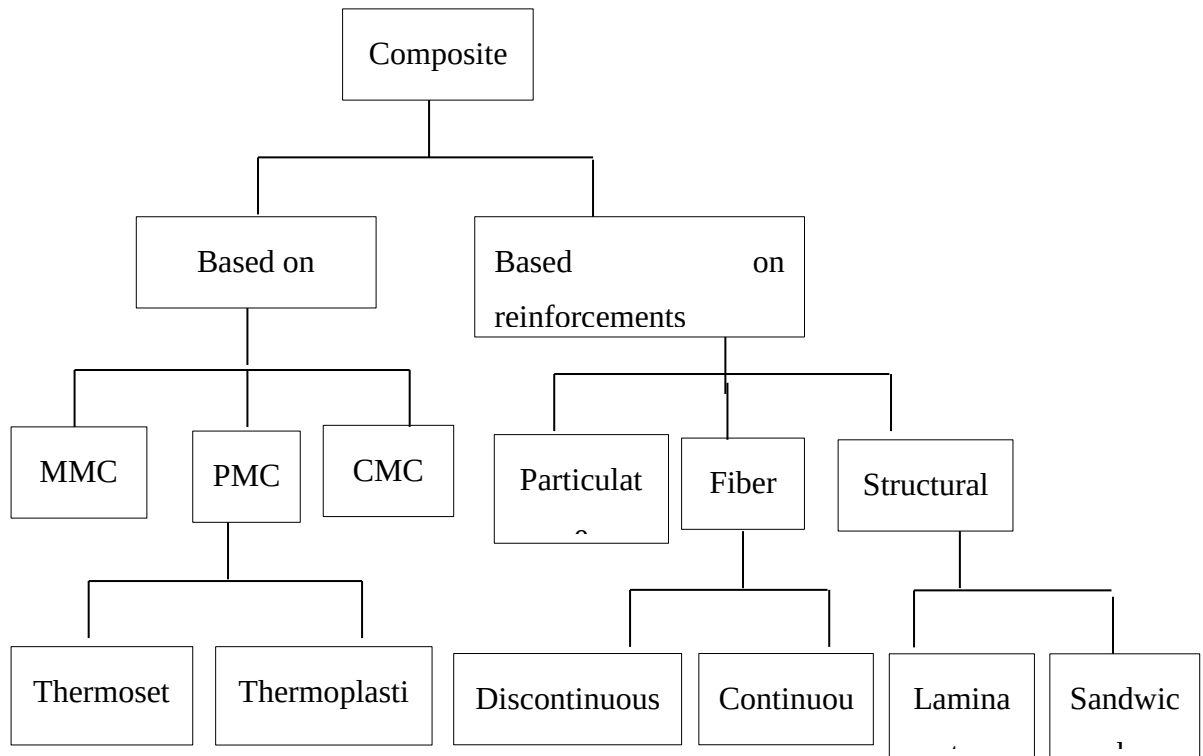


Figure 3.1 Classification of composites materials

2.3. Metal Matrix Composite

Metal matrix composites which combined matrix like: aluminum, copper, magnesium, titanium and reinforcements like: boron carbide, molybdenum dioxide, zirconium dioxide, Al_2O_3 , graphite, Silicon carbide. The metal matrix composites reinforced with various particles exhibit a blend of the properties of their constituents, proving greater qualities as compared to a pure (unreinforced) matrix (Salvo et al., 2019). The continuous phase, referred to as the matrix, which must be metallic, is surrounded by the reinforcement, which can take the shape of continuous fibers, whiskers, or particles (Martínez et al., 2018). The many forms of reinforcement can produce a variety of attributes depending on the goal and circumstances. By adding reinforcements to metal matrix composites (MMC), it is possible to improve the qualities of metals, including strength, hardness, and wear resistance. However, adding reinforcements will make the composite heavier than the underlying material (Rakesh, 2022). The size of reinforcements has a big impact on the mechanical properties of materials. Metal matrix composites are desirable building materials for a range of engineering uses (Vijayaram and Baskaralal, 2016). Composite initially begins as separate components, such that metal matrix and reinforcement. The matrix is nearly never a pure metal; instead, it is almost always an alloy. Taya and

Arsenault (2016) and Gautam et al. (2018) each type of metal matrix composite depend on strength is defined as:-

Dispersion-strengthened: This composite's unique characteristics are a result of its microstructure, which is made up of an elemental matrix with small particles equally distributed throughout. Particle volume fractions range from 1 to 15%, while particle diameters fall between 0.01 and 0.1 μm .

Particle-reinforced: This composite is distinguished by scattered particles with a volume fraction of 5 to 40% and a diameter greater than 1.0 μm .

Fiber (whisker)-reinforced: In fiber composite materials, the reinforcing phase covers the full size range (0.1 up to 250 μm in length to continuous fibers) and the entire volume concentration range (a few percent to more than 70%).

The matrix alloy's increased strength and stiffness is one of the main goals of MMC production (Taya and Arsenault, 2016). Due to their high strength-to-weight ratios, high temperature stabilities, and unique thermal, electrical, and chemical properties, they are well suited for usage in automotive, aerospace, electrical, electronic, energy, biomedical, and other applications (Kim and Choi, 2020).

2.4. Fabrication of metal composite

Metal matrix composites (MMCs) are recently created manufactured materials with properties including a decreased thermal expansion coefficient, increased specific strength, higher wear resistance, and low weight (Sharma et al., 2023). Fiber-reinforced metal matrix composites (MMCs), which are created from composite materials, offer mechanical and physical properties that make them a great choice for application in aerospace and different sectors. The goal of the matrix during MMC composition is to hold onto the reinforcement and organize the correct shape while the reinforcement concentrates on improving the necessary mechanical properties of the matrix (Gautam et al., 2018). There are three types of procedures for fabricating metal matrix composites: solid-state methods, liquid-state methods, and alternative preparation methods. These methods vary depending on the raw materials selected (Xiao-Su Yi, 2018). Liquid state processing procedures, including stir casting, compo casting, squeeze casting, ultrasonic-assisted casting, infiltration, thermal spray, laser melt-particle injection method (Sharma et al., 2020).

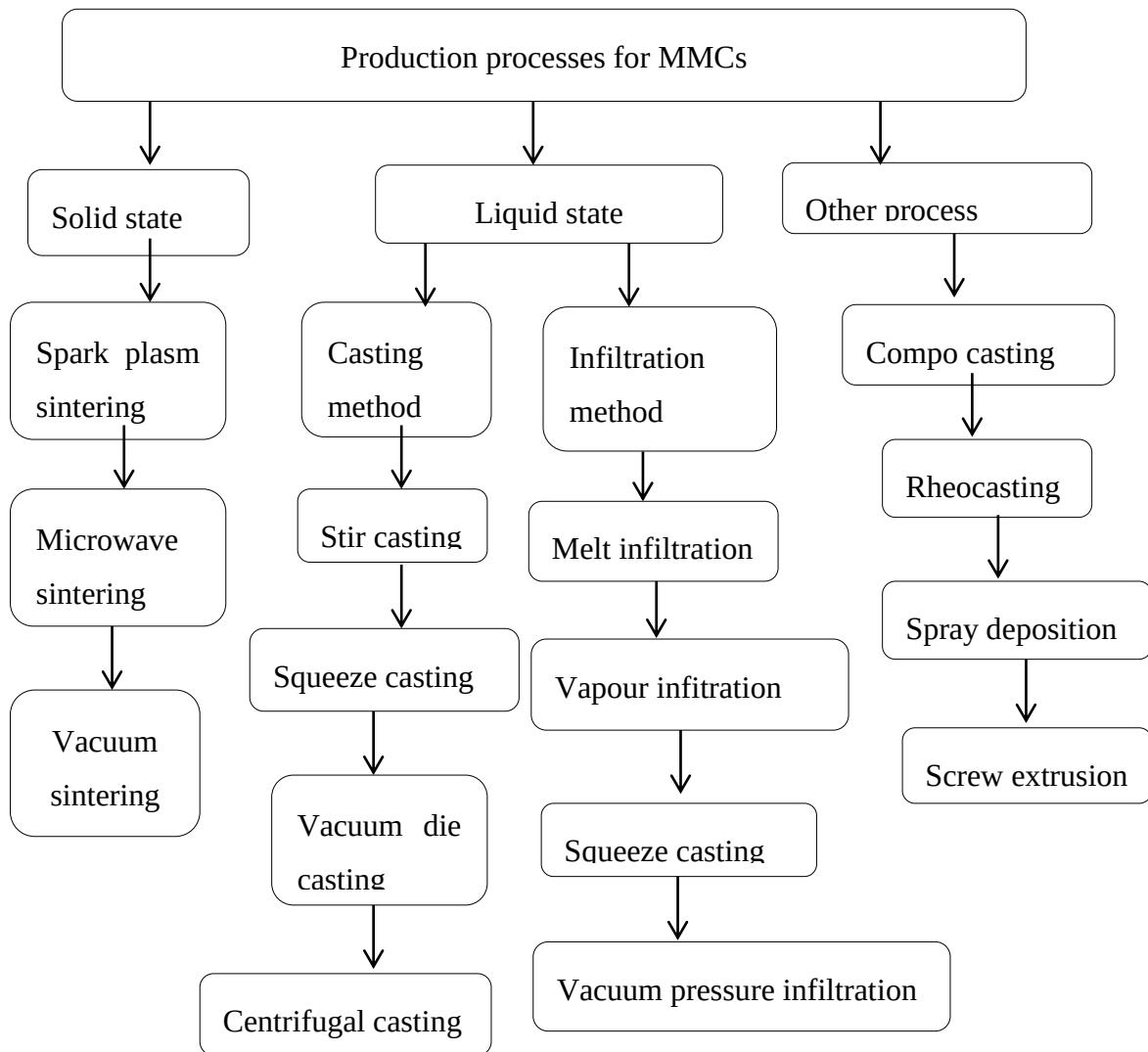


Figure 3.2 Classification the fabrication process of MMCs (Ramanathan et al., 2019)

2.4.1. Fabrication process of liquid state technique

Fabrication of Liquid state is the process of which liquid metal or molten metal matrix dispersed on reinforcement phase to follow its solidification good interfacial bonding (wetting) between the dispersed phase and the liquid matrix is essential to provide the composite a high level of mechanical characteristics. These procedures either include infusing molten metal into preforms or fiber packs, or they involve casting techniques such combining molten metal with reinforcing particles (Ramanathan et al., 2019). In this technique, liquid reinforcement from the discontinuous phase (reinforcement) is incorporated into the continuous metal matrix (Dhanesh et al., 2021). Stir casting, compo casting, squeeze casting, casting with ultrasonic help, infiltration, thermal spraying, and the laser melt-particle injection technique are all phases in the processing of liquid states

(Sharma et al., 2020). Coating the fibers during the dispersed phase may help with wetting (Kandpal et al., 2018).

2.4.2. Stir casting

Stir casting is one of the widely used economical processes that are used for the fabrication of the MMCs. Stir casting process is low cost process for manufacturing when compare with other process which fabricate metal matrix composite (Kumaraswamy et al., 2018). With reinforced discontinuous particles, MMCs have been made using this technique. Reinforcements (silicon carbide and graphite) added in to matrix within crucible furnace and mixed by mechanical stirrer at 300rpm for 40min (Jamwal, Seth, et al., 2020). So, stir casting process which simple process reinforcement added in to furnace mixing with molten metal material matrix was melted in furnace at high temperature continuously stirring by stirrer, continuously stirring used to reduce agglomeration or uniformly distribution. In the metal manufacturing industries, the use of the stir casting technique is increasing because it produces components with good quality and few casting defects (Vairamuthu et al., 2021). To improve interfacial bonding and uniform reinforcement distribution, stir-cast MMCs may be extruded (Sharma et al., 2020). To prevent particle settlement and achieve uniform distribution into metal matrix with perfect wettability, mechanical or electronic stirring can be used (Kadam and Shinde, 2020). The mechanical characteristics and microstructure of the composite are controlled by a large number of procedural affecting factors (Jamwal, Mittal, et al., 2020). In the stir casting method, the tensile strength of the composite was assessed using a variety of control parameters, including stirrer speed, duration, and furnace temperature (Reddy et al., 2022). The quality of the components can be improved by controlling several stir casting process parameters, such as temperature, speed, time, and preheating time, as well as the choice of matrix and reinforcements (Kandpal et al., 2018). Stir casting used for improving the mechanical properties of composites, excellent interfacial bonding between the dispersion face and melted matrix (Gautam et al., 2018).

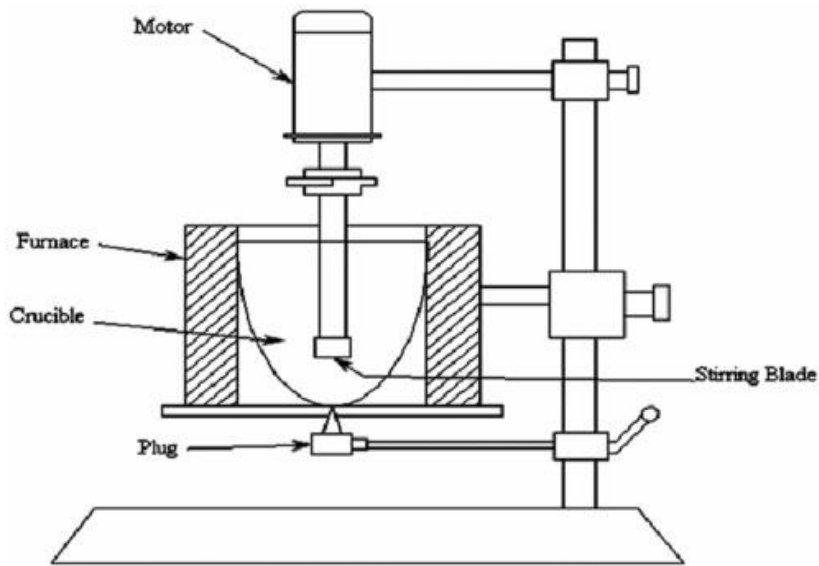


Figure 3.3 Stir casting setup (Jamwal, Mittal, et al., 2020).

2.5. Copper metal matrix composite

The use of ceramics as reinforcements is a key component of the sustainable development of copper metal matrix composites, and the choice of reinforcement material is greatly affected by the mechanical properties of the material, such as hardness, wear resistance, cost effectiveness, marketability, and refractory nature (Jamwal, Mittal, et al., 2020). The impact behavior and hardness of both the Cu-based MMCs were enhanced by the inclusion of the hard ceramic elements (Samal et al., 2023). Because of its excellent corrosion resistance, electrical conductivity, and workability, copper is widely used in electrical applications (Jahani et al., 2023) and (Kappagantula et al., 2022). To increase the thermal diffusivity at the friction surface, copper matrix is used. The effect of marble dust reinforcement on the mechanical and tribological characteristics of composites made from copper-based metal alloys (C93200 series) intended for bearing applications (Rajak et al., 2020). It was possible to find a material that would function effectively for bearing applications according to by using Copper-based alloy (C93200) composites reinforced type of strengthening (S. K. Rajak et al., 2019). Additionally, the functional characteristics of copper/graphene composites, such as their electrical and thermal characteristics, tribological characteristics, and corrosion behavior, are discussed (Hidalgo-Manrique et al., 2019). The added layer of graphene clearly improved the mechanical performance of copper (F. Chen et al., 2016). The network-interpenetrating structure formed by graphene and the copper matrix enhances the composite's mechanical and electrical capabilities in a way that is mutually beneficial (Pavan and Balamurugan, 2020). The performance of

graphene/copper composites as a whole is mostly determined by the dispersion of graphene in copper matrix and the strength of their interfacial connection (Li et al., 2021).

2.5.1. Fabrication of copper metal matrix composites

Recent developments in metal matrix composites have provided new means to produce high sustainable copper metal matrix composite materials with high wear resistance and high strength materials (Jamwal, Mittal, et al., 2020).

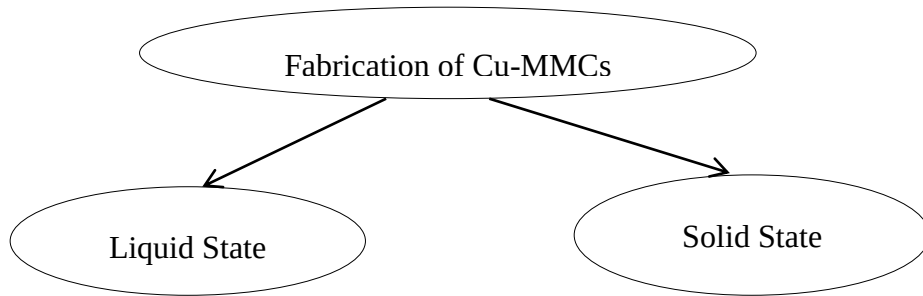


Figure 3.4 Fabrication techniques for Cu-MMCs (Jamwal, Mittal, et al., 2020).

2.6. Recent research developed on copper metal matrix and fabricated through stir casting process

Kandpal et al. (2018) Moulding, the friction stir process, mechanical alloys, and squeeze casting technologies were studied in relation to several MMC fabrication procedures and how MMCs are produced through stir casting. To study stir casting process variables such temperature, speed, time, preheating time of matrix and reinforcing options, this researcher employed stir casting. The stir casting technique and conditions can enhance the properties of metal matrix composites.

Singh and Gautam (2019) studied on the hybrid composites reinforced with ceramic copper metal matrix produced through stir casting techniques, the changes in structural, mechanical and electrical properties were studied in depth. The reinforcements were tungsten carbide, boron nitride, and chromium at fixed weight percentages combined with varying weight percentages of boron carbide (B_4C) in copper, which served as the matrix. The hybrid composites' hardness and ultimate tensile strength rise with increasing B_4C up to 1.0 wt%; after that, the characteristics decrease. The hybrid composites' compressive strength is greater than that of their matrix and exhibits an increasing trend when B_4C increases from 0 to 1.5 weight percent while keeping the weight percent of WC, BN, and Cr constant.

Rajak et al. (2020) investigated on copper matrix (C93200) and marble dust reinforcement (1.5 to 6.0 wt %) were studied among the ways of metals produced using liquid stir casting. To decreases, when the percentage of marble dust increases to void percentage i.e. from 0.785% to 0.497% and when marble dust is added up to 4.5 wt%, however, above 4.5 wt% the void fraction increases to 1.54%.researcher studied reinforcement of marble dust ceramic particulates enhances the mechanical properties (tensile strength, hardness, compressive, flexural strength) for the novel manufactured alloy composites when is added up to 4.5 wt%.

Jamwal, Seth, et al. (2020) Stir casting was used to produce Cu matrix composites with graphite-SiC ceramic reinforcements of matrix and reinforcements was used to increase the strength of the composites while it was investigated in this study that the density decreased when the reinforcement percentage increased. The Vickers hardness tester is used to test the composite's hardness, which decreases as the amount of reinforcement increases. Wear rates are lower with graphite and SiC particles acting as a lubricating coating on the counter part during wear testing than with pure copper. Maximum wear resistance, in the researcher's viewpoint, is found at 8% reinforcement content.

Kumar et al. (2021) Studied RG10 with different weight percentage of reinforcements of particle which SiC increased from 3% - 13% was fabricated by stir casting process with improved microstructure of developed samples and hardness property. As researcher examined result of relation between weight percentage of reinforcements and mechanical properties means when SiC weight percentage increase hardness increase but wear rate decrease. The wear rate mostly depends on the applied load and Sliding distance. As the result of this researcher discussed on SiC, when increase SiC, the theoretical and experimental density of composite decreases slightly because of the lower density of SiC in comparison to RG-10 (3.21 g/cm³ of SiC and 8.8 g/cm³).

Tarai et al. (2022) this study motivated on manufacture of CMMC with reinforced graphite and SiC ceramic reinforcements to create hybrid composites via stir casting process. Using XRD and FESEM, the microstructure of the composites was examined. Using the Vickers microhardness test, it was possible to determine how microhardness varied according to the amount of reinforcement. The results showed that the microhardness value increased as the amount of reinforcement increased. It was found that adding more reinforcement to the copper hybrid composites increased their tensile strength. When compared to the base

matrix alloy, the composites containing 1% Gr and 8% SiC showed a maximum improvement in ultimate tensile strength of 34%.

Samal et al. (2023) the study focused on the mechanical and microstructure characteristics of copper alloy (C87600), which was produced by stir casting and included reinforcements SiC-Gr and WC-Gr to create a hybrid composite. The percentage of graphite particles was kept constant, but the amounts of SiC and WC in the corresponding composites were changed in order to investigate the mechanical characteristics of the created composites. FESEM was used to observe the morphology of the composites, and the results showed that the reinforced particles were uniformly dispersed throughout the hybrid composites. Generally as the above researcher both of the Cu-based MMCs' tensile behavior and microhardness were enhanced. In comparison to the SiC-Gr reinforced hybrid composites, the WC-Gr reinforced composites showed higher mechanical properties.

2.7. Simulation and optimization process for selection sample.

Copper-based MMC to improved mechanical properties in hot extrusion process by supported simulation model can accurately predict the extrusion ration, extrusion load, effective stress (Gheysarian and Honarpisheh, 2021). Simulation process can obtained the values of response like damage, effective stress and extrusion load and graph analysis of strain effective, temperature. Before start simulation process combined parameters by BBD method in RSM which optimization used by Design- Expert software (Zahed et al., 2019).in optimization process select parameters effect on response were utilized to investigate the effects of process variables extrusion process with ANOVA table to analysis responses and interacted those parameters effect on response (Gugulothu et al., 2023).

2.8. Extrusion process

Extrusion is a manufacturing process that uses a material that is pushed through a die with the desired cross-section to generate items with a set cross-sectional profile (Bahl, 2021). Extrusion is a very versatile manufacturing method since it allows for the extrusion of a wide range of materials and profiles on a single press by simply altering the die and the equipment that goes with it (Chahare and Inamdar, 2016). Extrusion appears to be a method of disintegrating forged billet structures that are only subjected to compressive stresses in the extrusion process (V Sanabria et al., 2019). Metals like aluminium, brass, copper, lead and tin, magnesium, zinc, steel, and titanium are frequently extruded and the most popular extruded material is aluminium (Yadav et al., 2018). The hot extruded

nanocomposites demonstrated homogeneous graphene dispersion in matrix and were primarily distributed in grain boundaries (Mirjavadi et al., 2018). Tensile and hardness test findings demonstrate that the material's properties can be improved following extrusion of the raw material, and microstructure analysis of the extruded products of copper shows that the deformation (extrusion) has resulted in more uniform and homogenous grains (Kumari et al., 2015) and (Bahador et al., 2020). Cylindrical shape of ram, top and bottom die, container, and work piece cast or rolled are the four main parts of the extrusion process. To create the finished extrudes the work piece to move through open die by applied force. The basic material for the composite extrusion method is a billet, and the hybrid material is incorporated into the flow of molten metal inside a particular tool during the extrusion process. Then, inside the tool, the hybrid substance is created by introducing the additional material into the flow of molten metal extrusion (Bahl, 2021). Extrusion has several benefits over other metalworking processes, including the ability to produce a variety of sections (e.g., through hot extrusion), improvement of grain structure and desired strength (e.g., through cold extrusion), attainment of close tolerance (e.g., through cold extrusion), and typically little to no material waste (Ikumapayi et al., 2019).

According to Groover (2019) advantage of extrusion process:-

- to form various shape especially with hot extrusion
- to increase strength properties and better grain structure
- to reduce wastage material or little
- fairly close tolerances

2.9. Classification of extrusion process

Extrusion processes have various categories depend on different way like: classified based on working temperature (hot extrusion process and cold extrusion process), direction of work piece flow (direct extrusion process and indirection extrusion process) (Groover, 2019). So, extrusion process can classify as below:-

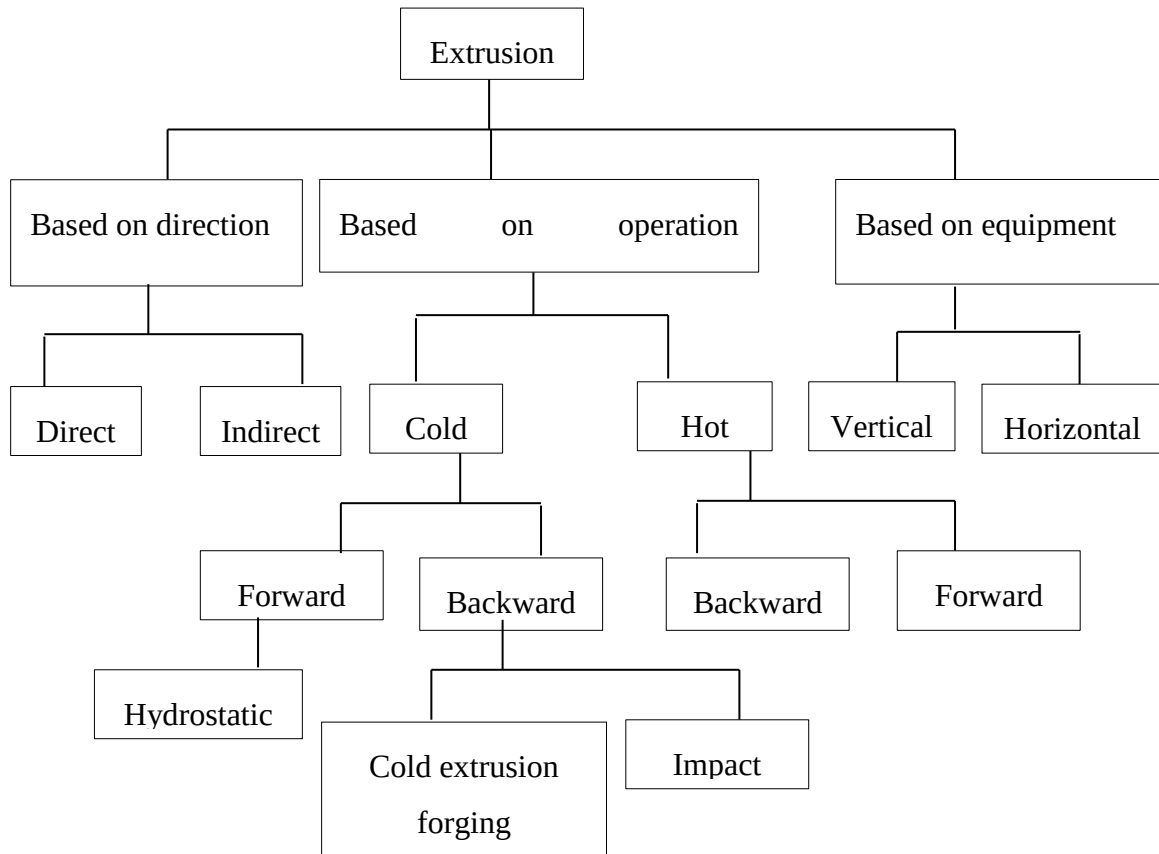


Figure 3.5 classification of extrusion process

2.9.1. Direct extrusion process

The direct extrusion press's press ram pushes the billet through a die and a fixed container. Depending on the form and shape of the die, direct extrusion is used to create solid rods, bars, hollow tubes, and hollow and solid sections. A type of extrusion known as direct extrusion, sometimes known as forward extrusion, happens when the direction of metal flow coincides with ram movement. Direct extrusion a cylindrical work piece is loaded in to a container and ram forced work piece pass through open die at opposite of the container (Groover, 2019). Molten glass is used as a lubricant in direct extrusion to alleviate the high friction brought on by steel at higher temperatures, while lubricants containing graphite powder are used for lubrication at lower temperatures.

2.9.2. Direct metal extrusion process advantages

- Both hot and cold extrusion can be done using it.
- Easy tooling in comparison to other extrusion techniques
- There is no need to modify the billet.

2.10. Hot extrusion process

MMC specimens with excellent electrical and mechanical properties are created during the manufacturing process of the MMC by combining powder compaction at room temperature with hot extrusion at a high temperature for a brief period of time (Tokutomi et al., 2015). Preheating, pressing, die interaction, cooling, and stretching are the steps in the extrusion process, which is followed by cutting and heating. Treatment (Thareja, 2015). Due to the severe plastic deformation that occurs during the hot extrusion process, a significant number of dislocations will spread throughout the alloy, and its density will rise quickly (Ni et al., 2023). Under hot-extrusion conditions, the hybrid composite's grain size decreased (Ramesh et al., 2020). Hot extrusion can manufacture complex parts and long products at a low cost of equipment and tooling, making it ideal for high volumes of manufacturing.

2.10.1. Study and analysis parameters of hot extrusion process

A, Extrusion speed

The ram speed was calculated from the pushing length and extrusion time (Tokutomi et al., 2015). Ram speed has a significant impact on the temperature distribution in the billet due to complex heat generation and loss processes, which affect both the heat produced and the heat lost to the extrusion equipment (Rahim et al., 2016).

B, Working Temperature

At increased temperatures between ambient temperature and 200 C, copper behavior is studied and novel micro extrusion die set assembly has been created for the experimental portion, together with temperature assistance, which allows the specimen to be heated inside the die assembly to analyses deformation behavior (Nanthakumar et al., 2020). The best processing temperature range is often determined by the melting point, thermal stability, and glass transition. Hot melt extrusion relies on temperature energy to melt the components in order to generate a solid dispersion system formula in a suitable form (Alshetaili et al., 2020).

C, Extrusion ratio

A larger extrusion ratio produced grains that were substantially coarser, had lower yield strengths, and had greater hardening capabilities (Sadeddin et al., 2021). The refining of the grain size is responsible for the increase in yield strength and ultimate tensile strength

of as-extruded specimens with increasing extrusion ratios (Zhang et al., 2011). The dimples on the tensile fracture surface grew and became more uniform when the extrusion ratio was raised from 7 to 30, resulting in an outstanding elongation of 32.4% (Ni et al., 2023).

D, Die profile

To increase the quality of the end product and production effectiveness, numerous research on the optimization of the die profile through the integration of the traditional mathematical methods of optimization with the finite element method (FEM) have been conducted (Lin et al., 2003). The primary factor in die failure is excessive wear in a die caused by an excessive extrusion load. Due to their high strength/stiffness in relation to weight, extruded profiles/sections are frequently utilized as fundamental building blocks to manufacture structural component (Zhou et al., 2020).

2.11. Recent research done on CMMC through hot extrusion process

Ren et al. (2015) by combining Cu-Al alloy sheets internal oxidation with hot extrusion by Cu-0.16 wt% by Al alloy, investigated Cu-Al₂O₃ bulk nanocomposites were created. Internal oxidation of 90 mm in diameter x 0.5 mm thick objects with Cu₂O + Cu + Al₂O₃ mixed powders was carried out at 900 °C for 8 hours. Cu-Al₂O₃ nano-composite rods with a 20 mm diameter were created by cleaning, stacking, vacuum-sealing, and hot extruding oxidized sheets at 800°C. Rods were then cold pulled to a wire with a 3 mm diameter. The smaller the Cu-Al₂O₃ sheets were after that, the better the interfacial bonding was, and the electrical conductivity of the rod was 96.3%. This study used a novel approach of bulk dispersion. Strengthened to increase tensile, yield, and electrical conductivity and reduction of the cross-sectional area of the drawn wire were 94.1% IACS (International Annealed Copper Standard), 417 MPa, 445 MPa and 80.2%, respectively.

Kumari et al. (2015) was examined the factor of extrusion process has improved mechanical properties compressive strength as well as hardness has improved. This researcher was studied on the circular rod of diameter CMMC 12.5mm and more studied the reinforcements of particles added on copper matrix more homogeneous to obtain good result from OM after samples extruded and enhanced mechanical properties after extruded was increased by 6.8% and the braking load was increased by 22.9%.

Lee et al. (2015) Fabricated Al with Copper composites was extruded through hydrostatic extrusion process with a repeated 3 times with described the directional microstructure by

a repeated three times when 200 °C temperature. To analyze the effect of the intermetallic compound at the interface and the asymmetric physical properties of the fabricated Al/Cu composite, to examined thermal property of extruded samples by coefficient of thermal expansion was obtained result 18.3 $\mu\text{m}/\text{m}^\circ\text{C}$ at difference between temperature 25°C and 450°C as well as improved ultimate tensile strength of the Al with Copper composite is 102.8 MPa were determined. Then lastly identify fracture started at aluminum near the interface between aluminum and copper through repeated extrusion process.

Leema et al. (2015) copper alloy with 5%-20% reinforced tungsten composite performed by milling mechanical to mixed, compacted also sintered within hot extrusion process. Through this process investigated the pore area and pore perimeter measurement by used SEM after sintering and after extrusion. After extrusion improved with mechanical property density of 87%to 94%, wear rate, friction coefficient hardness and tribological behavior of composites.

Shamsborhan and Ebrahimi (2016) carried out fabrication of nanostructure pure copper the capability, mechanical and microstructural characterization of commercial pure copper processed by planar twist channel angular extrusion. After extrusion deformed has 71%, 69%, 113% and 12% higher yield strength, ultimate tensile strength, hardness magnitude and reduction of strain hardening and grain refinement occurrences respectively in comparison with the initial condition. And also, elongation to failure is decreased from 71% to 37% after the one pass deformation as compared to the as-received condition.

W. Chen et al. (2016) carried out work on Ag-Cu-MoS₂ hybrid composites, was made by powder metallurgy. After fabricated by this method developed sample in powder metallurgy was extruded the parameter of extrusion ratio 10-70 and hot press at 680°C temperature was extruded. Ag-Cu-MoS₂ of hybrid composites by hot press were extruded under various extrusion ratio improved mechanical properties, microstructure, fracture and wear test investigated were the normal load at 5N and slide speed at 0.27m/s on a pin on disc equipment for improved wear rate was reduced.

Selvakumar and Christopher Ezhil Singh (2016) Investigated hybrid composite of Cu–4%Cr–ZrC were fabricated through powder metallurgy was added various weight percentage of ZrC from 0-10% and extruded. Developed samples deformed through extrusion process discussed on SEM, XRD, wear test and hardness improved mechanical

properties by added ZrC. As researcher conclude when ZrC weight percentage increase density was decrease when compared with Cu-Cr matrix with improved porosity.

Senthil Kumar et al. (2016) fabricated of copper alloy as metal matrix with reinforcement Tin at different percentage 0, 5%, and 10% were compacted through powder metallurgy with MoS₂ added for improved as lubricant. The microstructure analysis of the composites with both of reinforcement's particles mixed uniformly in the copper matrix alloy without agglomeration and orientated randomly in the matrix and the extruded composites. Conclusion that observed decreases in the average size and better distribution of the MoS₂ particles and wear properties of sintered Cu-Sn-MoS₂ composite before and after extrusion.

Singh and Selvakumar (2017) Investigated of copper-chromium reinforcement with milled B₄C nanoparticle strengthened with Cu–Cr matrix alloy provided properties superior to that of the base materials. Above researcher was concluded when fabricated through hot extruded at 600°C temperature and he obtained result from density measured was improved porosity by 92%. So summered by when porosity improved, hardness and wear resistance was improved.

Wang et al. (2017) successfully prepared by dispersing nano-sized SiCp/Al master alloy into the molten Al-Cu alloy with combined semi-solid stir casting and hot extrusion processes. With nano-sized SiCp adding to the Al-Cu matrix, the tensile strength of the described composites increased remarkably especially under the temperature of 453 K. After hot extrusion process, the tensile strength of the present composites was improved when the contents of nano-sized SiCp particles were arranged from 1 wt% to 3 wt %. From above research paper the Al-Cu composites having 2 wt% nano-sized SiCp displayed the best tensile properties and output $\sigma_s = 373$ MPa, $\sigma_b = 527$ MPa and $\epsilon = 11.5\%$, $\sigma_s = 325$ MPa, $\sigma_b = 360$ MPa and $\epsilon = 14.9\%$, $\sigma_s = 235$ MPa, $\sigma_b = 269$ MPa and $\epsilon = 17.1\%$ at 298 K, 453 K and 493 K, correspondingly.

Mirjavadi et al. (2018) developed sample of Al-10Zn-3.5Mg-2.5Cu-Gr via casted and after casted deformed through hot extrusion. With used heat treatment for enhanced properties of material also studied effect Gr at varying percentage start from 0.1, 0.3, and 0.7 added on Al alloy with discussed when Gr content increased to improved strength.

Volkov et al. (2018) fabricated two Cu/Mg-composite rods of different content of components were produced by hydrostatic extrusion. Produced composites revealed the possibility of forming eutectics of different composition at the Cu/Mg interface. Patterns of

the formation of strength and electrical properties of Cu/Mg composites became clear. Cu/Mg composite was extruded. Estimate calculations of the strength properties and electrical resistance of deformed composites were carried out and compared with the experimental results

Zhang et al. (2019) experimented on Cu 110 and Al 1100 alloy with varying initial grain sizes, achieved through ECAP and post annealing by axisymmetric reverse extrusion. At a characteristic extrusion dimension of 100–200 μm , the study was focused on the influence of the initial average grain size on the mechanical response and homogeneity of plastic deformation, the characteristic plastic strain for extrusion and the influence of initial grain size on the extrusion mechanical response and shape of extruded parts were examined in detail through SEM and EBSD, in combination with CPFEE simulations.

Li et al. (2019) fabricated Cu-4.5Al₂O₃ bulk composite first by separated nanoparticle of Cu-0.8 % and Al 1.5 % and O powder, heat treatment and lastly consolidation of the powders by powder compact extrusion. The result of this research paper investigated an excellent combination of a high tensile strength (522 MPa), good ductility (elongation to fracture: 8.2%) and a high electrical conductivity (90%IACS) after extruded.

Vidal Sanabria et al. (2019) focused on preparation of aluminum alloy EN AW-1080A and the copper alloy Cu-ETP by backward rod extrusion at room temperature. The copper cross section was equivalent to 30% of the billet was extruded applying a conic die angle of 90° and an extrusion ratio of 14:1. Billet was completely covered with the graphite paper Grafoil (250 μm thickness), the conic die face was with a high surface roughness by grinding with SiC-paper grid 80 and covered with a graphite-based lubricant, which was burned onto the surface at 400 °C. When increase of the flow stress ratio $\sigma_{\text{Cu}}/\sigma_{\text{Al}}$ at higher temperatures, as well as the progressive increase of the plastic strain seem to be the main cause for the fracture occurrence. So, cold aluminum core and hot copper sleeve would reduce the flow stress ratio between the two applied alloys and could contribute to a more uniform material flow.

Hu et al. (2021) Investigated 2%Cu–0.15%Ag–La alloy composed of large-size Ag-rich primary phases was processed into wire billets by successive drawing, annealing and continuous extrusion forming. Obtained from new fabricated sample the YS, UTS, and elongation of the annealed 2%Cu–0.15%Ag–La alloy increased from 65 MPa, 253 MPa, and 34% to 150 MPa, 300 MPa, and 49.5%, respectively. Friction heating promoted the

dynamic recrystallization of more than 80% of the alloy microstructure. From above research paper identified the microscopic mechanism behind the effect of precipitates on the microstructure in the CONFORM process and clarified the impact of the latter on the yield strength, tensile strength, elongation, and high-temperature softening resistance of Cu–Ag–La alloys.

Guo et al. (2021) used to mixed extrusion and powder metallurgy process made hybrid CMMC with CNTs reinforcement when weight percentage of CNTs was 4.2% and TiB₂ 1.6% enhanced resistance of arc erosion fabricated copper matrix composites. CNTs has role to improve the electro emission and decrease emission of current density.

Li et al. (2021) successfully fabricated and designed the copper/graphene composites with high strength and conductivity by using CVD in-situ growth of graphene, hot extrusion and cold drawing processes. Graphene distributed inside the copper matrix can effectively improve the strength copper composite by tensile strength (595 MPa) and conductivity (5.46×10^7 S/m) of the composite indicate an increase of 23.4% in tensile strength, and similar (98%) conductivity of the matrix, respectively as well as affected grain refinement on conductivity.

Zheng et al. (2021) fabricated copper alloy with reinforced MWCN were mixed by ultrasonic and nitrogen-assisted, electro less Ni-Cu in hot extrusion and cold drawing. 1%, 3%, 5%, 7% and 10% percentage MWCNTs reinforced copper matrix composites were arranged in the direction of hot extrusion and cold drawing to improved mechanical properties with tensile strength was 402.6 MPa means 52.15% higher than that of pure Cu, the yield strength was 378.6 MPa by 87.22% higher than that of pure Cu, and tribological property friction coefficient was 0.151 improved by 74.7% lower than that of pure Cu and the wear rate was $0.52 \times 10^{-5} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$ was improved by 70.3% lower than that of pure Cu.

Gao et al. (2021) worked on Al₂O₃/Al–Cu composite which has been in-situ synthesized by a liquid–solid reaction method, followed by hot extrusion process. After extrusion improved heterogeneous structure of the composite, mechanical properties of the as-extruded and T6 treated were fabricated samples of ultimate tensile strength improved in to 420MPa and elongation of the T6 treated improved 5.7%.

(Li et al., 2022) Focused on study about copper metal matrix composite with Gleeble-1500D simulator was used to conduct thermal compression experiments on Cu/CeO₂/Cr

and GO-Cu/CeO₂/Cr composites at 0.001-1s⁻¹ strain rate and 600-900 °C deformation temperature. Effects on the graphene oxide composite as studied by the researcher the composites' activation energy increased by 27.99%e210.130 kJ/mol with the addition of GO. When the deformation temperature decreases or the strain rate increases, the flow stress increases. The flow stress of GO-Cu/CeO₂/Cr composites is increased by the small-size nanoparticles CeO₂ at the metal matrix interface to pin dislocations, dynamic recrystallization, grain refinement, and twin formation.

2.12. Summary of literature review

Copper metal matrix composite are reinforced with a variety of particulate ceramic reinforcements, and research has been done on the effects of the reinforcement's weight content and particle size on the mechanical and tribological behavior of the resulting composite. Stir casting is a commonly employed CMMC manufacturing process; however they suffer from certain disadvantages which pose as challenges in production. CMMC fabricated to achieve homogeneous distribution of the reinforcement in most fabrication process adjustment of stir casting parameters like stirring speed, pouring temperature viscosity of the melt, stirring time speed and particle size with weight percentage was done. According to many researcher was studied on copper metal matrix fabricate through stir casting the reinforcements added on matrix better should be particles because if reinforcements is particles or small grain size to more homogenous or uniformly distribution of particles in matrix of copper for solve agglomeration problem. Also particle ceramics of reinforcement to enhanced mechanical properties and tribological properties of copper. During stir casting fabrication to concerned about the wettability between the molten copper alloy matrix and ceramic particle of reinforcement because the factor that affect the bonding between them and can also affect the mechanical properties. After sample was fabricated via stir casting developed samples was deformed through hot extrusion which an efficient metal forming method for reinforcing composite materials. Extrusion has the advantage of assisting in the alignment of the reinforcement within the composite material, increasing the overall strength of the finished product. To determine the impact of variable factors on the metal-forming process before the actual production process, finite element modeling and simulation approaches are needed.

2.13. Research Gap

It has been observed from the literature review that sufficient researches were carried out on fabrication, characterization and testing the properties of copper metal matrix composites applicable to various applications but very limited work have performed including the post deformation behavior of casted Copper metal matrix composites. Moreover, the deformation behavior of casted Copper metal matrix composites on optimum extrusion conditions has been rarely studied. Additionally, stir casting is very important for fabrication copper composite in mixing matrix and reinforcements in uniformly distribution of particle. In stir casting copper metal matrix composite contains pores and vacancies throughout the material. Therefore, it is challenging to prevent defects in stir casting. Few studies have been done on the relationship between defects that occur during stir casting and those that occur before to the extrusion process. Therefore, the proposed work aims at fabrication of reinforced Copper metal matrix composites and evaluation of its pre and post deformation behavior such as metallurgical, mechanical and tribological.

CHAPTER THREE

MATERIALS AND METHOD

Deformation behavior of hybrid copper composite with varying weight percentages of CeO₂, SiC, and Gr nanoparticles is investigated through fabrication, testing, numerical analysis, and experimental analysis. By using casting to create the composite material, optical microscopy will be employed to investigate how the reinforced particles are distributed throughout the matrix. The wear rate of the developed samples was calculated based on results of using the pin-on-disc equipment, as well as the Vickers hardness of the developed samples. In order to ascertain the mechanism underlying wear behavior, the wear morphology of the used samples was examined using SEM. The samples' tensile and compressive strengths were tested on UTM.

3.1.Flow Chart of the Research Idea Procedure

Here is a brief outline of the process by which the research begins and ends. This process is the main idea of the research in an organized manner and the sequence of work is written in a simple manner.

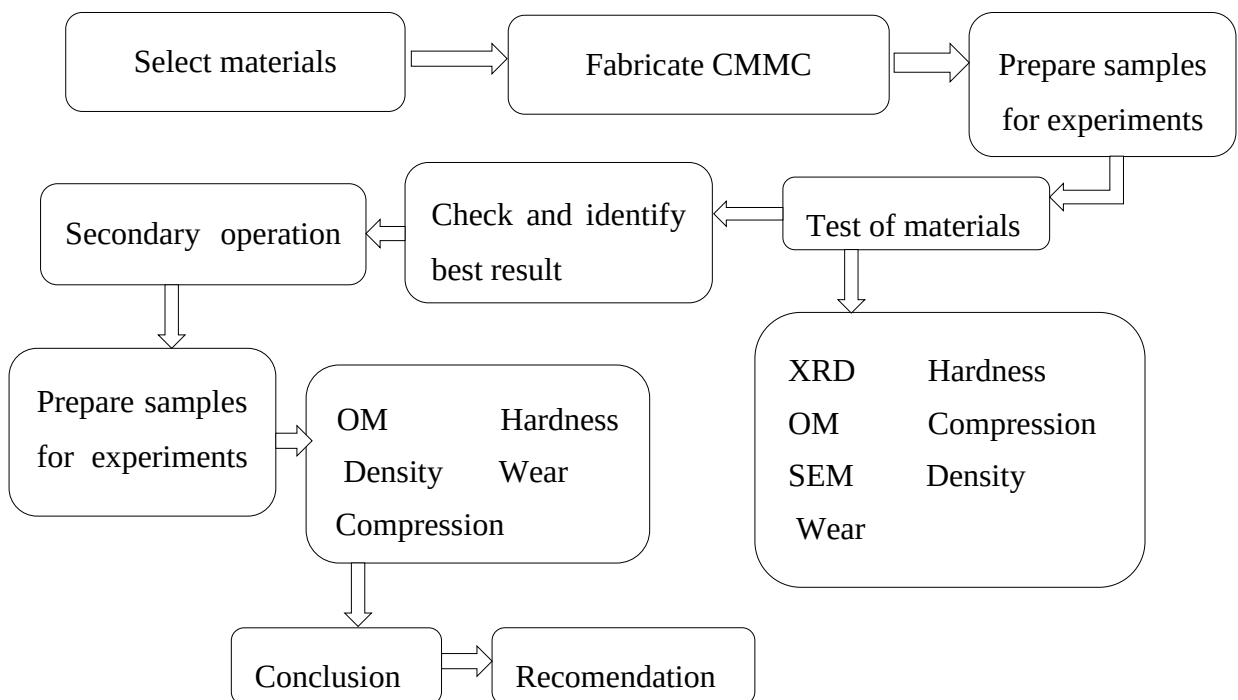


Figure 4.1 Flow chart of research methodology

3.2. Materials

For use in aerospace applications, composite materials must possess specific qualities. These characteristics need to cover every investigation of mechanical properties, microstructural properties, tribological properties, and fracture mechanics. This helps to easily select the materials used in both metal matrix and reinforcement matrix phase. Materials that are used on this research worked on copper alloy of matrix and reinforcement matrix (SiC, Gr, CeO₂) that yields the needed properties and small amount of graphene used for self-lubrication properties, silicon carbide improve strength of materials. This decision took into consideration cost, characteristics, and usability.

3.2.1. Matrix

The fiber system of a composite is embedded in the matrix, which is a homogeneous, monolithic material. The term "matrix phase" describes a continuous phase in which a composite or two-phase alloy is created in a metal microstructure and completely encloses the other (or dispersed) phase to maintain the stability of the dispersion phase.

3.2.2. Copper

Copper is ductile material, it has good conductivity and electrical transparenence. It is used in electric generators, as wire in appliances, lights, household and automotive electrical wiring, phone lines, motors, and kitchen sinks because it resists corrosion and has anti-microbial properties to prevent infection. The excellent ductility, thermal and electrical conductivity, malleability, and corrosion resistance of copper make it a vital component in many industrial processes. The thermal and electrical conductivity of OFC are good. There are more than 400 different types of these copper alloys, but OFC is commonly preferred in order to excellent strength and wear resistance at high temperatures.

Table 4.1 Content composition of OFC (Oxygen Free Copper)

Element	Content (%)
Copper	99.99%
Oxygen	0.0005%

3.2.3. Reinforcements

In the field of materials science, reinforcement is a component of a composite material that raises the stiffness and tensile strength of the composite. In order to the fabricate composites using particle reinforcements. Particle-reinforced composites are used in

applications that need high levels of wear resistance, such as road surfaces, because particle-based composites are less effective at strengthening than fiber-based composites (Ayyappadas et al., 2017). A Nano-particles composite exhibits improved wear behavior, increased hardness, and improved particle distribution within the matrix (Keneshloo et al., 2017). Because Nano-composite powders create reinforcement in the form of nanoparticles, it has been discovered that a very small grain size composite powder with a uniformly mixed particle size dispersed in matrix was obtained with an increasing volume percentage of Nano-SiC (Keneshloo et al., 2017).

3.2.4. Gr (graphene)

Applications for graphene reinforce add on other matrix materials and their composites are promising in several fields, including membranes, biomedical support, sensors, actuators, and electronics (Iqbal et al., 2020). Gr add on copper alloy to enhanced the wear properties, the most cost-effective reinforcements are silicon carbide and graphite (Jamwal, Seth, et al., 2020). The uniformly dispersed graphene in the Cu matrix provides a strengthening effect and protects the as-fabricated bulk Gr/Cu composites from corrosion by 50% in NaCl solution and at the same time, the yield strength of the composites increases by approximately 18% (2.5 vol% Gr) without compromising electrical conductivity (Jin et al., 2019).

3.2.5. Silicon Carbide (SiC)

SiC reinforcement which ceramic particle to add hardness for materials and improved the wear properties matrix materials as well as most cost-effective reinforcements are silicon carbide and graphite (Jamwal, Seth, et al., 2020). Increasing the quantity of SiC additives substantially enhances the hardness of Cu matrix composites, and as a result, the wear rate reduces.

3.2.6. CeO₂ (cerium dioxide)

The results of SEM and XRD uniform distribution of Cerium Oxide in copper matrix with diffraction pattern indicate good solubility of CeO₂ in copper matrix (Mishra et al., 2018) and (Pandey et al., 2022). To create a copper matrix with high strength that is supplemented with powdered Cerium oxide in order to improve the mechanical properties of the composites (Mayilsamy et al., 2020).

3.3. Method

3.3.1. Synthesis of copper metal matrix via stir casting process

Copper alloy (OFC) used as matrix with fine powder particles of reinforcements which are size nanoparticles (Gr, SiC, and CeO₂) are mixed and fabricated through stir casting process. The molten metal matrix which is melted in to furnace at above melting temperature during the stir casting process, and reinforcements powder particle are added to the molten matrix and mixed using a mechanical stirrer (Sharma et al., 2020). In stir casting process the weight percentage of reinforcements should be (≤ 30) as several researchers reported, when mixing with matrix because of large weight percentage of reinforcement (≥ 30) to increase agglomeration or not achieve uniformly distribution of reinforcement. So total weight percentage of reinforcements (19%) mixed with 81% of matrix in to furnace formed copper metal matrix composite. weight percentage/volume of reinforcements have factor on fabrication of metal composites, less weight percentage of reinforcements can reduce agglomeration of reinforced particles (Kumar et al., 2023). Copper alloy melted inside of crucible by using a furnace at 1200°C. After the copper is melted, add the existing reinforcements to the stirring time. However during stir time, control the speed, time, and temperature to avoid agglomeration. In stir casting process must be properly measured amount of reinforcements which add on matrix by analytical weight balance for reduce agglomeration, to achieve good mechanical properties, to identify specific object as amount of reinforcements. Before the reinforcements are added to the matrix the types of reinforcements should be premixed by ball milling and preheated in the required its weight percentages. Reinforcements of particles (SiC and Gr) were preheated at 800°C (Jamwal, Seth, et al., 2020). This allows them to mix into the matrix more easily and in a shorter time. Stirring times (30, 40, 50 min) as described in one study revealed that the copper and its reinforcements formed homogeneous distributions during these minute and studied reinforced preheated at 800°C (Vairamuthu et al., 2021). As this researcher depends on his result of SN ration was used to show the effect of factor with respect to the responses with got best result value of hardness test, tensile strength and impact at stirring time of 30 minute. Due to properly mixed matrix and reinforcement particles in furnace to continuous stirred for 30 minute. After finished mixing process the liquid of molten to pour in which prepared mold cavity and solidified in mold.

3.3.2. Design experiment of copper metal matrix composite fabrication

Based on the previous research, he now focused on taking the percentage of reinforcement added to the matrix which was done by different researchers, developed new samples and investigated the behavior of the samples was fabricated. The pre-produced composites were selected for the preferred parameters of this process, which are used as base (stirring time, stirring speed, pouring temperature) and the quality conditions of the composite depend on them. Many researchers was optimal value time of stirring 10min and speed of stirring at 300rpm (Sakthivelu et al., 2018), (Almadhoni and Khan, 2015). The new sample was developed this to selected input parameters, stirring time of 30 min and stirring speed of 300rpm based on the above literature review. Below weight percentage of reinforcement particles was selected on articles(Parikh et al., 2023), (Msomi et al., 2021), (KALISHWARAN et al., 2023),(Sharma et al., 2020)

Table 4.2 Design Experiment of the CMMC via stir casting

S. No	Reinforcement and their weight percentages			OFC
	CeO ₂	SiC	Gr	
1	5%	10%	4%	81%
2	7.5%	7.5%		
3	10 %	5 %		
4	-	-	-	100%

3.3.3. Mold preparation for stir casting

The size of the mold required to make the composite depends on the size of the specimen used for the various tests. Size of the sample to determine but depending on the number of features we test produced by the size of the entire sample needed. Since the number of samples to be tested is known it is now easy to know our mold content. But small numbers of samples required to be produced by identifying the types of tests (destructive and non-destructive), using one sample for different tests for non-destructive test and new samples for destructive test. After identified the number of samples and type of tests used test to understood size of mold which required making samples. For check amount of sample depends on the test standard of ASTU lab.in ASTU lab as standard. According to ASTU standard tests done in laboratory like XRD, SEM, OM, corrosion test, hardness test,

compression test, fractography and density measurement and other test done at AASTU is wear test. Therefore, the sample size and the mold size required to prepare the sample were made.

Calculate size of the mold required make sample depend on number of samples with the test standard. Total length = for (XRD, SEM, OM, hardness test of 8mm) + (corrosion test as ASTU standard of 4mm) + (wear test as AASTU standard of 12mm) + (machine allowance 15mm + extrusion process 45mm)

Total length = 84mm and diameter = 20mm

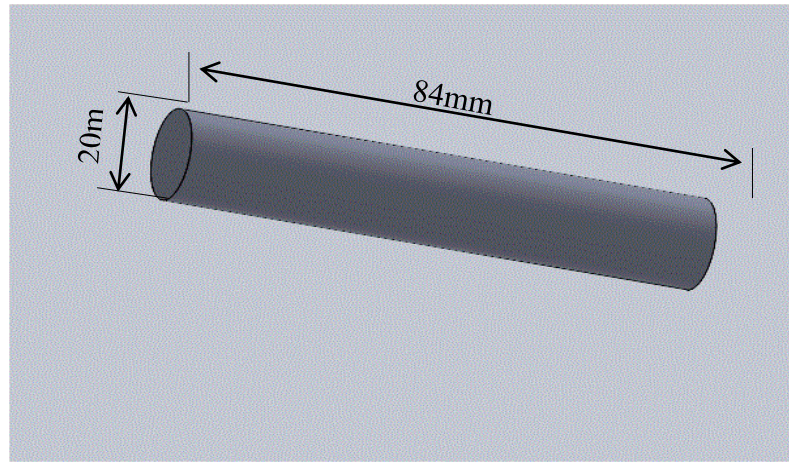


Figure 4.2 3D drawing of sample prepared.

The volume of required mold for the sample were calculated as follows,

$$V = \frac{\pi}{4} D^2 \times L \quad (3.1)$$

Where, V= volume of sample in mm³

D= diameter of sample in mm

L= length of sample in m

$$V = \frac{\pi}{4} (20\text{mm})^2 \times 84\text{mm} = 26.376\text{cm}^3$$

Volume of mold = 27cm³ (cylindrical shape of sample)

$$V_T = 27\text{cm}^3, \quad \text{total volume of each sample}$$

Total volume required for one sample = 27cm³ so, total volume of four samples = 4*27cm³

V_T = 108cm³, total volume of material fabricated through stir casting process (matrix and reinforcements particle).so depend on volume of material required for fabrication to made

mold cavity. Then depend on total volume samples fabricated to calculate total mass and for each sample.

$$m_y = V_T \times \rho_y \times \text{wt\%} \quad (3.2)$$

Where, m_y = mass of reinforcement and matrix

V_T = total volume

ρ_y = Density of reinforcement and matrix

wt% = weight percentage of reinforcement and matrix

Calculated mass in gm required for fabrication as follow:

$$(S1) = m_{cu} = V_T \times \rho_{cu} \times \text{wt\%cu}$$

$$= 260.6904 \text{ gm}$$

$$m_{SiC} = V_T \times \rho_{SiC} \times \text{wt\%SiC}$$

$$= 1.156 \text{ gm}$$

$$m_{CeO_2} = V_T \times \rho_{CeO_2} \times \text{wt\%CeO}_2$$

$$= 12.987 \text{ gm}$$

$$m_{Gr} = V_T \times \rho_{Gr} \times \text{wt\%Gr}$$

$$= 3.265 \text{ gm}$$

$$(S3) = m_{cu} = V_T \times \rho_{cu} \times \text{wt\%cu}$$

$$= 260.6904 \text{ gm}$$

$$m_{SiC} = V_T \times \rho_{SiC} \times \text{wt\%SiC}$$

$$= 5.778 \text{ gm}$$

$$m_{CeO_2} = V_T \times \rho_{CeO_2} \times \text{wt\%CeO}_2$$

$$= 2.597 \text{ gm}$$

$$(S2) = m_{cu} = V_T \times \rho_{cu} \times \text{wt\%cu}$$

$$= 260.6904 \text{ gm}$$

$$m_{SiC} = V_T \times \rho_{SiC} \times \text{wt\%SiC}$$

$$= 8.667 \text{ gm}$$

$$m_{CeO_2} = V_T \times \rho_{CeO_2} \times \text{wt\%CeO}_2$$

$$= 19.48 \text{ gm}$$

$$m_{Gr} = V_T \times \rho_{Gr} \times \text{wt\%Gr}$$

$$= 3.24 \text{ gm}$$

$$(S4) = m_{cu} = V_T \times \rho_{cu} \times \text{wt\%cu}$$

$$= 260.6904 \text{ gm}$$

$$m_{Gr} = V_T \times \rho_{Gr} \times \text{wt\%Gr}$$

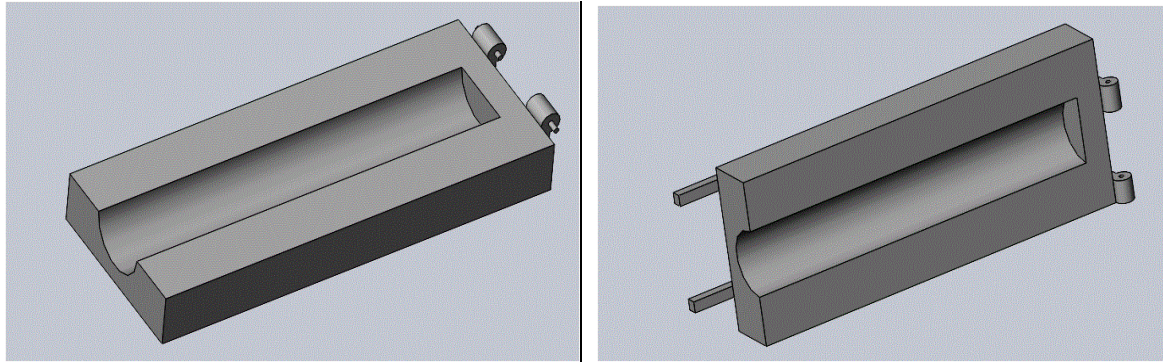


Figure 4.3 Mould (a) drag (bottom mold) (b) cope (upper mold)

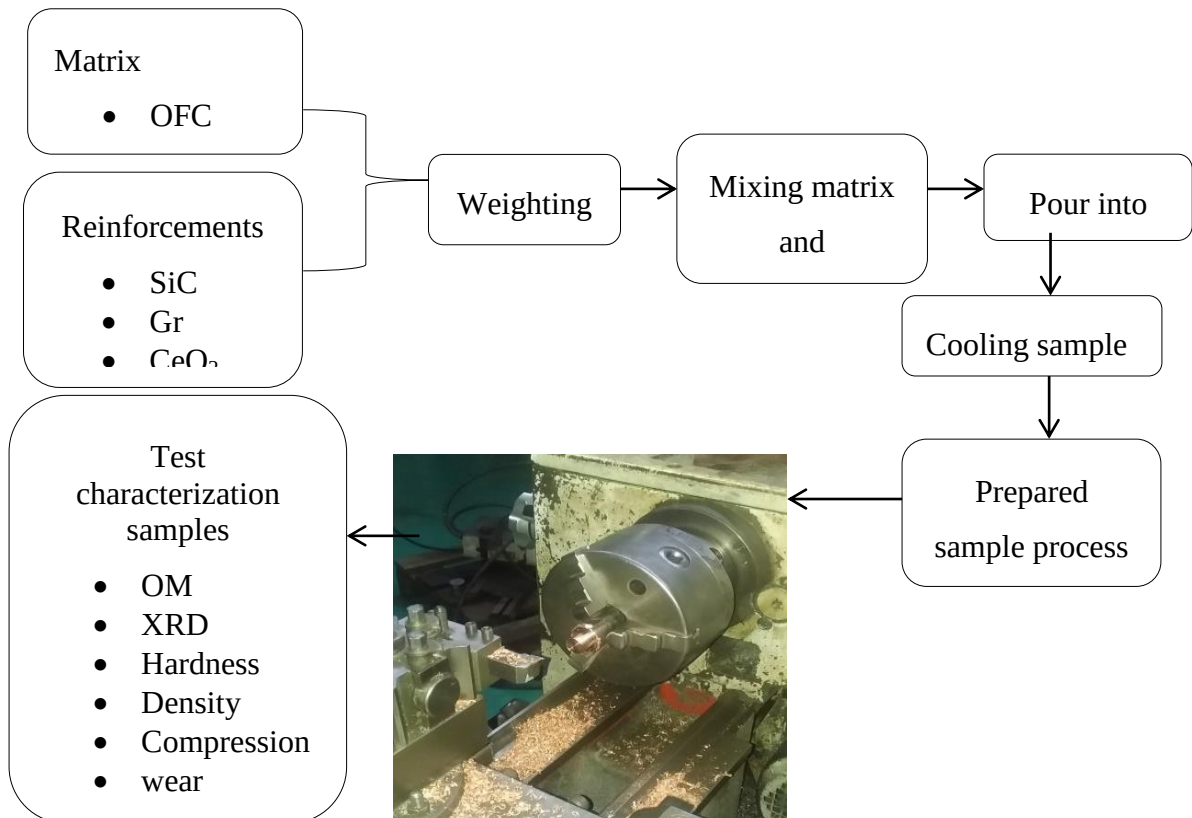


Figure 4.4 The fabrication process of CMMC through stir casting and testing samples.

3.3.4. Step Fabrication of copper composite by using stir casting

Stir casting technique is the easier process, the most cost effective and molten metal matrix with reinforcements mixed in to furnace to formed phase of MMCs development method in all liquid state fabrication processes. The furnace achieve sufficient temperature to copper alloy (OFC) mixing with reinforcement by using mechanical stirrers which helps to dispersed particles form uniformly distribution of composite. When composite fabricated by stir casting process: achieve uniform distribution reinforcement, achieve wettability between the two main substances and minimize for improve the MMC property.

1, **Melting Metal Matrix:** copper alloy melted into furnace as melting point of OFC and moulding in to suitable shape. Metal matrix material melted in crucible heated by high temperature in furnace (Kumar, 2017). Copper alloy (C87600) ingots were in high temperature to heated in an electric furnace at 1200°C temperature (Samal et al., 2023).

2, **Stirring molten metal and Added Reinforcements:** mechanical stirrer is very important in stir casting for stirring or mixing matrix with reinforcements to form uniform distribution. In stirring process should be consider time stirring, speed of stirring, angle and geometry of blade. Reinforcements whether, added to matrix composites to improve many mechanical properties, but when fabricate composite should be consider to operational parameters of stir casting process (Kumar et al., 2023). During reinforcement added on matrix material, mechanical stirring process was very important to obtained uniformly distributed of reinforcement in to matrix material composites (Abebe Emiru et al., 2023). After added reinforcements (SiC + Gr + CeO₂) on matrix material (OFC) properly mixed by using mechanical stirrer to fabricated copper metal matrix composite by the stir casting process.

3, **Pouring of Mixture in the Prepared Mold:** mold casting made from ceramic because mixtures are both of matrixes and reinforcements have high temperature. Ceramic mold is highly resisted to high temperature.

4, **Solidification:** when the latent heat of fusion is released, the liquid phase changes to a solid phase. The homogeneity of the reinforcing particles created when the composite solidifies depends on pouring temperature and solidification rate, stirring speed and time, percentage of reinforcements and its size and density of reinforcements.

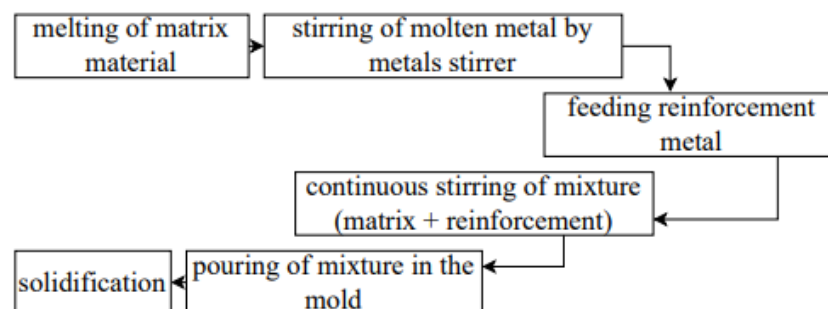


Figure 4.5 Stir casting flow process

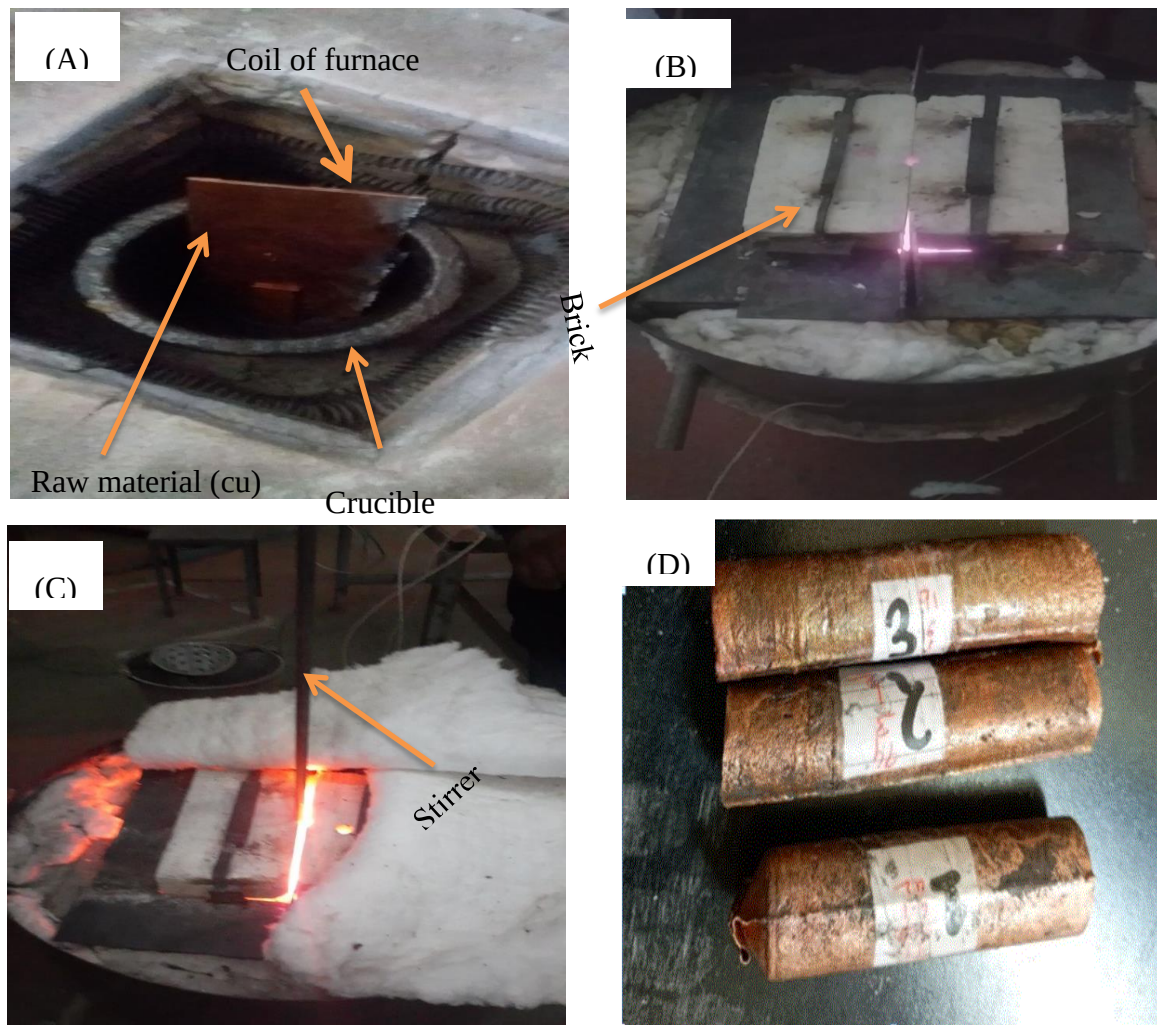


Figure 4.6 Fabrication process (A) raw material (B) melting of matrix (C) stirring process with add reinforcement (D) fabricated sample

3.4. Density and Porosity measurement

Theoretically density is the density of the material which can be defined as mass per unit volume. Density of the samples, were measured after fabricated via stir casting. When measured density of the samples was fabricated follow procedure and compared theoretical and actual density of samples. The main reasons about the diversity between the theoretical and experimental density values of the composites are the voids and porosity of the composite.

(1) Theoretical Density

The theoretical density can be measured after the sample is placed in water and is a required parameter to estimate the pore size of a material. Density of the samples in theoretical are determined by reacting, based on the mixed rules, determining the place of matrix composite and strengthening their liquid (Ayyappadas et al., 2017).

$$\rho_1 = (\rho_{mat} \times V_{mat}) + (\rho_{rei} \times V_{rei}) \quad (3.3)$$

Where, ρ_{mat} is density of matrix and V_{mat} is volume fraction of matrix

ρ_{rei} , is Density of reinforcement and V_{mat} is volume fraction of reinforcement

$$(S1) = (\rho_{cu} \times V_{cu}) + (\rho_{sic} \times V_{sic}) + (\rho_{ceO_2} \times V_{ceO_2}) + (\rho_{Gr} \times V_{Gr})$$

$\rho_1 = 7.725 \text{g/cm}^3$ follow the same direction calculated

$$S2) = 8.113 \text{g/cm}^3 \text{ and } (S3) = 7.5644 \text{g/cm}^3$$

Next was calculated by standing on the standard density of matrix and reinforcement, the actual density was calculated by measuring the samples in air and water.

(2) Actual density

Actual density is a measure of how tightly packed a substance. When the actual density of a sample is measured, the value obtained by first measuring the density in air was obtained by dividing the difference between the densities of air and water. The amount of decrease in the actual density of one sample when compared theoretically is used to determine the porosity of the sample. The actual density of a sample after measurement in air and distilled water (1g/cm^3) is calculated using formula of the Archimedes principles as follows.

$$\rho_{actual} = \frac{m_{air}}{m_{air} - m_{water}} \times \rho \quad (3.4)$$

Where, ρ_{actual} , actual density m_{air} is mass of sample measured in air.

m_{water} , Mass of sample measured in distilled water, ρ density of distilled water.

(4) Porosity of composite Materials

Porosity refers to the voids in a material or the open space between particles or the particle space surrounded by microstructures, and defines the presence of small openings or interstices in a material. Pore size and distribution in composites, plays an important role in controlling mechanical properties and therefore minimizing the porosity in composite in use is a necessity to achieve performance.

$$\% \text{porosity} = 1 - \frac{\rho_{act}}{\rho_{theo}} \quad (3.5)$$



Figure 4.7 Measurement of density (a) measured in water (b) measured in air

3.4. Study the Characterization of fabricated copper metal matrix composite and equipment

3.4.1. XRD (X-Ray Diffraction)

XRD study for crystallite size, phase composition of samples, quantitative phase of sample analysis with determine amount of phases in relative of peak intensities. In XRD to understand peak position, peak width (basically depend on main factor like crystallite size and lattice strain), and intensity produced by bombarding high energy electrons on metal. X-rays are used for over visible light of crystal diffraction because of its shorter wave length. Diffraction which method to identify crystal structure by treating inter atomic distances as slits. Peak width study the macro strain within angle (2θ) in which no strain peak at its natural position, compression strain its lattice size decrease with increase angle, tensile strain at its lattice size with increase angle and non-uniform strain peak gets broader.

1) Analysis of phase fabricated composite

Phase test conditions of the samples produced by the stir casting process, observed by X-ray diffraction (XRD, at ASTU) were subjected to $\text{Cu-K}\alpha$ radiation at 40 kV and 30 (mA) 2θ values between (10° - 80°) $3^\circ/\text{min}$ scan rates. Phase identification and quantitative phase analysis of the processed sample were studied through origin pro-2022 software

2) Calculation of lattice strain and crystalline size

The microstructural characteristics, namely crystalline diameters, mean squared lattice strain, root mean squared strain, and dislocation density, and are determined using X-RD peak profile analysis by Gauss method (Han et al., 2021) in equation 3.8. Many researchers have used various mathematical models to determine the crystallite size of the strengthening phase in various ways using XRD peak analysis, among which the Scherrer equation or the Williamson-Hall method are the main ones. These models used the widths of the XRD peaks to estimate that average crystallite size within the material. A popular technique for estimating crystallite size based on XRD peak broadening is the Scherrer equation. The related of Peak width (β) and crystallite size (D) can be calculated using the following formula.

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (3.6)$$

Where, D= crystallite size

λ = wavelength (μm) of x-ray

K =Shape factor can be 0.62 - 2.08. 45

β = line of broadening at FWHM in radians

θ =diffraction angle

The dislocation density is defined as the defects in the sample and is defined as the number of dislocations within a unit volume of the crystalline material (Rai et al., 2023). Dislocations, which are line defects that result from a mismatch or misalignment of the crystal lattice planes, are important for understanding the mechanical characteristics and behavior of materials. Diffraction peaks broaden as a result of local lattice strain caused on by dislocations within a crystal.

$$\delta = \frac{1}{D^2} \quad (3.7)$$

Where δ = dislocation density in nm^{-2}

D^2 = crystallite size of the particles

Using the data from the crystallite size, the possibility of determining the dislocation density of produced composites was tested (Rai et al., 2023). This researcher was studied the increase in the dislocation density of a crystalline material plays an important role in increasing the stress required to move the dislocation, so that strain hardening in the crystal takes place along the crystalline width.

The existence of defects (such as dislocations or vacancies), thermal expansion or contraction, mechanical stress or deformation, or the introduction of impurity atoms into the crystal lattice are some of the causes of lattice strain (Sahoo and Panigrahi, 2021).

$$\varepsilon = \frac{\beta}{4\tan\theta} \quad (3.8)$$

3.5. Scanning Electron Microscopy

A scanning electron microscope is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons. SEM studied microstructure and wear morphology of developed samples. SEM micrographs were image taken at 10 KX magnification size to get clear image of morphology (Jamwal, Seth, et al., 2020). However in process samples should be prepared during SEM testing for more study morphology of sample. common application of SEM in study the MMC microstructure analysis to examine the distribution of reinforcements within matrix, size, its shape, interfacial bonding, porosity of composite. The alignment of the reinforcing phase within the metal matrix directly affects the studied material strength, stiffness as well as wear and erosion done by SEM and to allow the surface damage studied. Study effect of corrosion on surface of samples, detail study wear morphology after which sample studied on wear test by pin on disc machine and check fractography after compression test.

3.6. Optical Microscopy

An optical microscope, like as a light microscope, used to a series of lenses to magnified images of very small size samples with visible light. The lenses are placed between the sample and the viewer's eye to magnify the image so that it can be examined in greater detail. Therefore optical microscopy will test grain size of copper metal matrix composite in thesis. Optical microscope is used to study the structural of materials. Preparation of samples for optical microscope used to metallographic specimens observations. The preparation of the specimen for microstructure examination begins with surface preparation with machining, followed by finishing procedures. In metallographic observation determine the accurate microstructure of sample to carefully require process: grinding sample by sand paper, mounting, polishing and etching.

1) Machining of samples on lath machine

The samples were machined on the lathe according to the required shapes and sizes depending on the experimental and standard machine types of test like: XRD, SEM, OM, Vickers hardness test, compressive test, wear resistance test and corrosion test machines. The samples were cut in Addis Ababa (MDI) with standard lath machine and best surface finishing.

2) Grinding of samples by sand paper

A sand paper is used to remove the cutting edges and scratches that remain on the material after machining. Adjusted to spacemen for microstructure examination depend on sand paper grade 220, 800, 1200 and 200. After the cutting edges of the samples were grinded with sandpaper and polished with a polishing machine, the structure of the samples was inspected by optical microscope before being etched.

3) Mounting process

The hot mounting press is used to mount the sample and the specimen is thoroughly dry and clear before being placed in the loading cylinder and the appropriate loading resin is added. The hot mount pressing method provides the best edge protection and for polishing to maintain a good edge and when study surface of sable by optical microscope the sample to consider suitable surfaces.

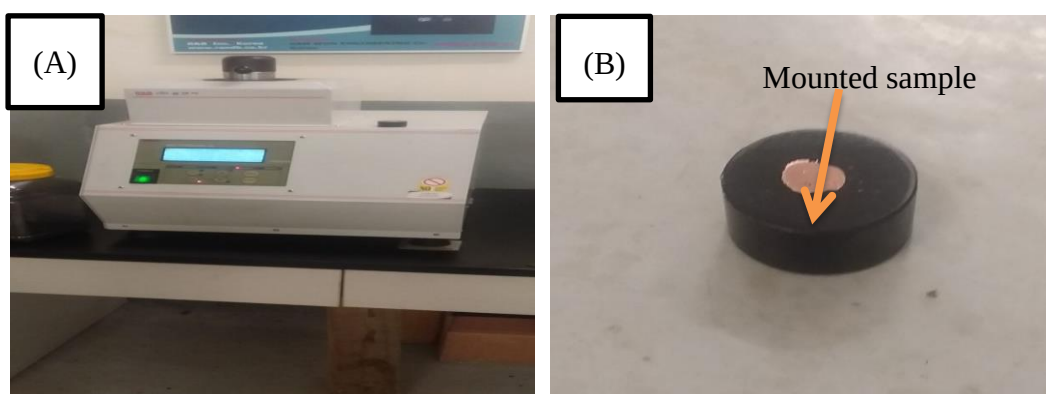


Figure 4.8 Mounting (A) hot mount press (B) mounted sample

4) Polishing

Polishing is the process the surface that is level, smooth, and devoid of scratches is necessary to produce detailed and crisp optical microscopy images. Polisher machine very important for avoid unnecessary material from material surface, clearer surface of material to form shine face by using diamond suspension.



Figure 4.9 Polisher machine

Before it is etched, the sample is observed but its structure and grain boundaries were not clearly visible and must be etched. FeCl_3 , an etchant, is used to etch the prepared specimen's surface of copper metal matrix composite (Kumar et al., 2018). Copper with Gr and Copper flakes' etching rates were studied in an aqueous solution containing 0.5 weight percent FeCl_3 (Jin et al., 2019). After polishing the specimens, FeCl_3 (30 gm.), HCL (15 ml), and H_2O (100 ml) were used to etch them (Mishra et al., 2018). So, the specimen after polished by polishing machine etched by solution of FeCl_3 (25gm), HCL (25ml) and distilled water (100ml).

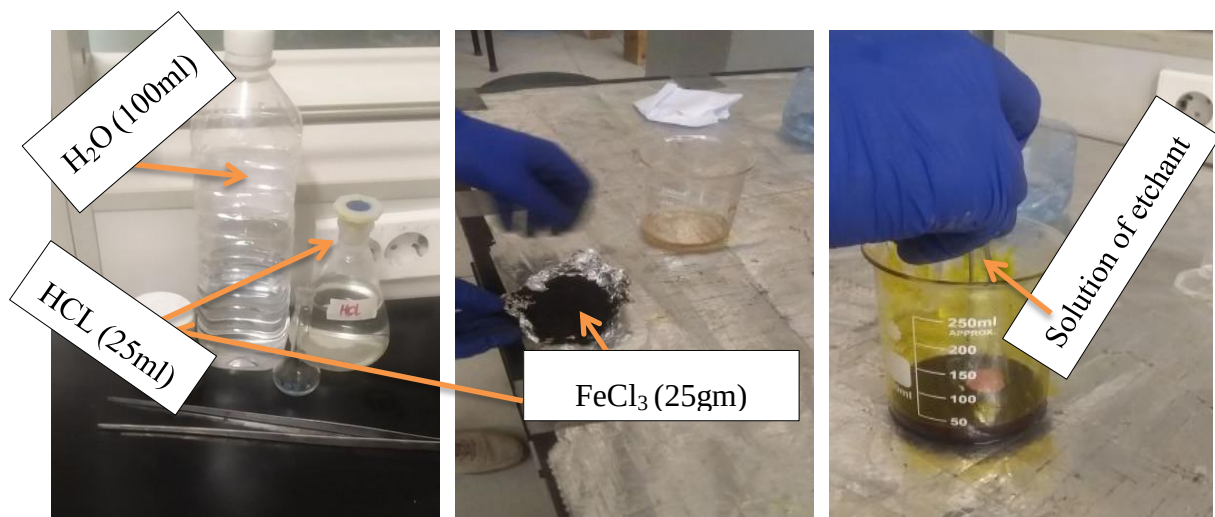


Figure 4.10 Etching process

Generally samples prepared for OM to studied surface of structure as follow process in the below figure.

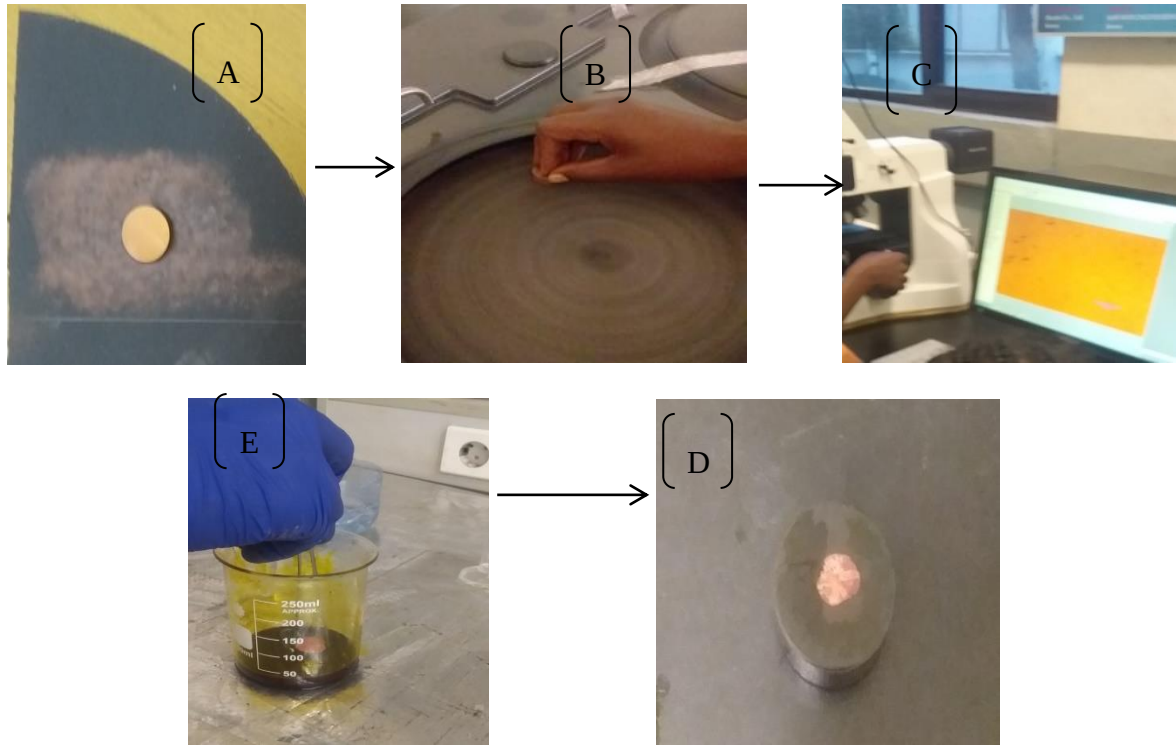


Figure 4.11 process of sample preparation (A) grinding (B) polishing (C) test OM before etching (D) sample after etchant (E) etching process.

3.7. Hardness test

Hardness is property of materials to resistance indentation or scratching process in permanent deformation. Hardness testing which the type of non-destructive test method when a constant load applied small area of specimen and form a pointed on the specimen and under controlled conditions, to create an indentation in a metal surface. Hardness test can categorized into macro and micro-hardness depend load to applied on indenter and diameter of sample (indentation) formed by indenter. Micro hardness which the load applied on indenter less than 1kg, like: knop hardness test as well as macro hardness test which the load applied on indenter greater than 1 kg, like: Brinell hardness test, Rockwell hardness test and Vickers hardness test. Micro indentation tests made with Vickers indenter (a square-based pyramidal-shaped diamond indenter with face angles of 136°) under test forces with standard ASTM E384-22 (Fenghong et al., 2019). Vickers hardness test which consists of Right Square of pyramid diamond indenter with base angle 136° to measure the distance between opposite corners of the pyramid after removed applied load.

$$HV = \frac{2F \sin \frac{136^\circ}{2}}{d} \quad (3.9)$$

Where F= load in kgf

d=mean of two diagonals, d1 and d2 in mm

HV= Vickers hardness

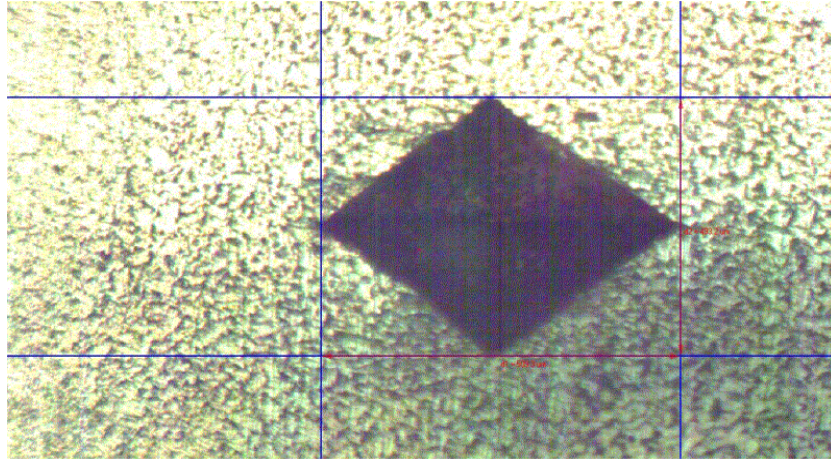


Figure 3: 1 Hardness test and measurement

3.8. Corrosion test

Corrosion tests were carried out by immersing the specimen (copper material) in a bath of 3.5% NaCl for 30 minute. The specimens for the tests were, cut to size 15mm diameter and 4mm length as an electrochemical analyzer (Biologic Science Instruments, ASTU, Material science laboratory. Composite specimens were grinded with sand paper 800 and after grinded to polished with 12µm diamond paste on a double disc polishing machine to remove any sort of stray marks from the surface of the specimen and to make it mirror polished. CMMC specimens with an exposed were, used as working electrodes, graphite electrode was, used as an auxiliary (counter) electrode, and standard calomel electrode (SCE) or mercury was used as reference electrode. Experiments were, done to observe the effect of reinforcement content on the corrosion of fabricated composite through stir casting process. Sometimes interphases formed between the matrix and reinforcements in the production and manufacturing process of MMC can effect on corrosion. Extrapolation at anodic and cathodic regions was useful to obtained corrosion current (i_{corr}) and corrosion potential (E_{corr}) while reinforcement fill is done around the defect and a specimen was attacked by electrolytic solution on a neutral surface of evaluated phase metal (Jamwal et al., 2019).

$$\mu_p (\%) = \left(\frac{I^{o_{corr}} - I^{i_{corr}}}{I_{corr}} \right) \times 100 \quad (3.10)$$

Where, μ_p (%) corrosion protection efficiency, I^{corr} and I_{corr} are values of corrosion current density for copper and for samples with reinforcements SiC/Gr/CeO₂ respectively.

Corrosion rate calculated by using formula:

$$\text{Corrosion rate in (mpy)} = \frac{CEwI_{corr}}{d} \quad (3.11)$$

Where C = conversion constant (0.13),

Ew = equivalent weight of the specimen (g),

I_{corr} = corrosion current and d is the density of the specimen (g/cm³).

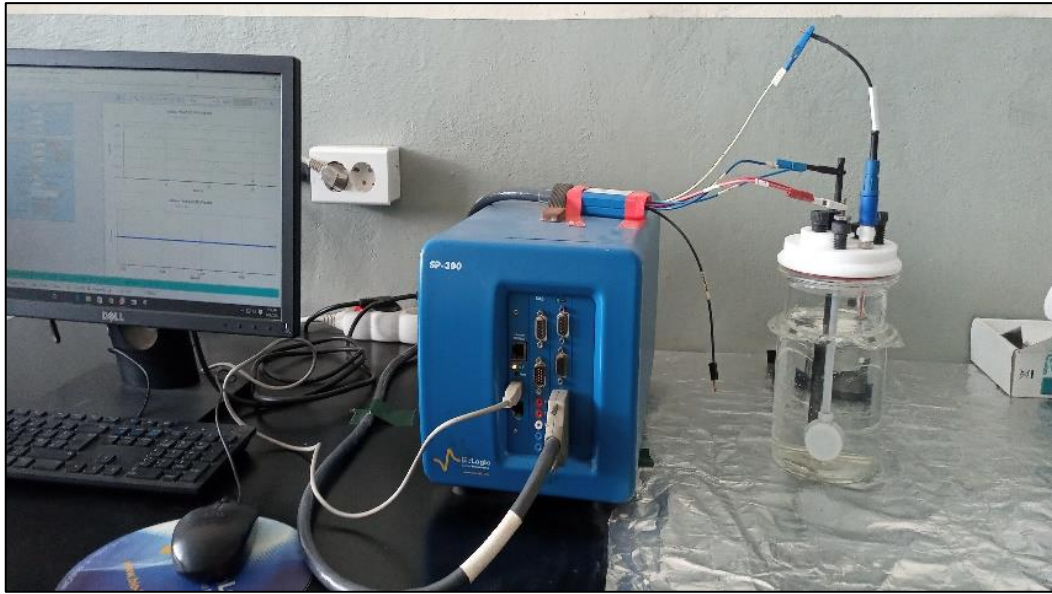


Figure 4.12 Electrochemical Analyzer machine

3.9. Compression test

Compressive testing is used to recognize the compressive strength of a material, determine the elastic behavior of the material, and determine stress after compression (fractography) under applied fracture loads. Compression testing was done on CMMC samples based on ASTM E9-09 standards with a diameter of 10 mm and a length of 10mm. The compressive strength was determined by applying a compressive load to a copper matrix composite specimen and was calculated by dividing the cross-sectional area force of the specimen by the gradual loading applied to the specimen.

$$UCS = \frac{F}{A} \quad (3.12)$$

Where, UCS = Ultimate compressive strength

F = Ultimate compressive load

A = Cross-sectional area of samples.

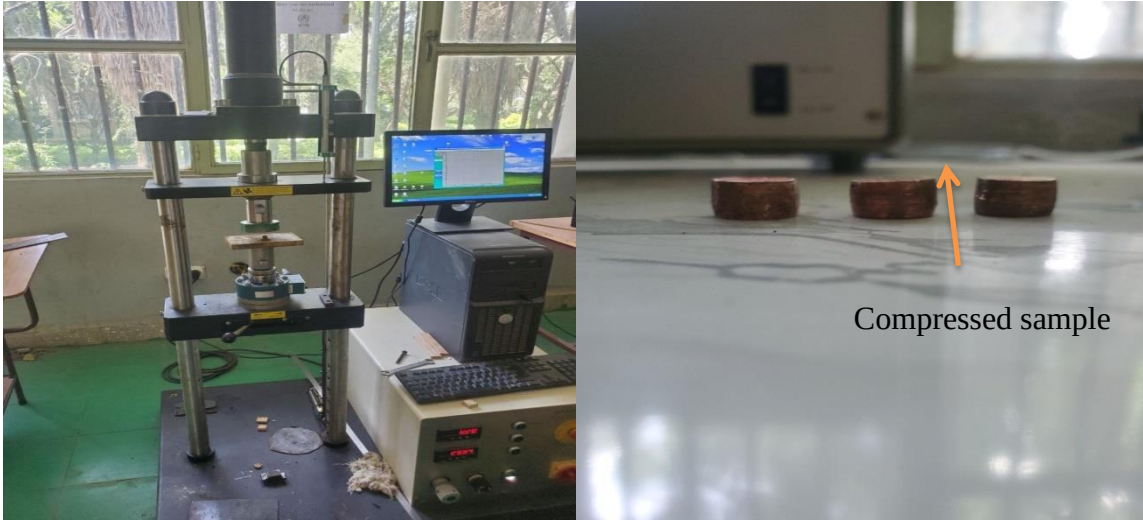


Figure 4.13 (a) compression tester (b) after compressed samples

3.10. Wear resistance

The specimens were cut to a cylindrical shape ($\varnothing 6\text{mm}$ and length 12mm) as required for the AASTU standard by pin-on-disc machine can form copper composite through stir casting. The wear resistance of developed samples were tested at room temperature in accordance with ASTM standard of G99 Pin-on-Disc tester (Amirthagadeswaran, 2019). There are many factors that can affect the rate of wear, some of which are material type, surface finish, load, speed and environment. One of the key elements influencing the various wear rates is the different types of materials. For example metals are more resistant to wear to plastics and Compared to smooth surface material, rough surface material will cause wear and friction. Wear and friction can be increased by both high speed and load.

Considering wear rate has an impact on component endurance, it is an essential issue in mechanical component design. wear rate is decreasing as reinforcement content increases (Jamwal, Seth, et al., 2020). The weight loss was significantly reduced when nano-scaled particles were utilized, with the SiC particle size decreasing from 15 nm to 30 nm (Keneshloo et al., 2017).

$$\text{Volume loss (mm}^3\text{)} = \frac{(\text{Initial mass in gram}) - (\text{final mass in gram})}{\text{density (g/mm}^3\text{)}} \times 100 \quad (3.13)$$

$$\text{Wear rate (mm}^3\text{/m)} = \left(\frac{\text{Volume loss}}{\text{Sliding distance}} \right) \times 100 \quad (3.14)$$

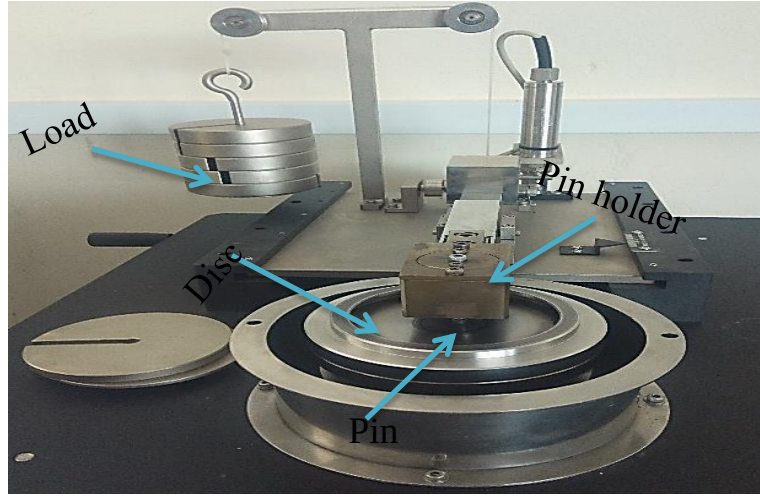


Figure 4.14 Pin on disc machine

3.11. Sample selection process for second operation

The procedure to selected sample fabricated through stir casting based on property of material obtained results from experiment with compared S/N ratio as following below step.

Step 1: Based on the type of quality characteristic, transform original response data to S/N ratio using related formula stated

Step 2: Normalize the data to minimize variability and the effect of using numerous units to make comparability easy. Normalize Y_{ij} as $Z_{ij}(0_Z_{ij_1})$, where, Z_{ij} represents the normalized for i th experimental/trial and the j th dependent variable/response. For normalizing S/N ratio with larger-the-better performance characteristic and small-the-better the following equation is used respectively.

$$Z_{ij} = \frac{Y_{ij}-Y_{min}}{Y_{max}-y_{min}}$$

$$Z_{ij} = \frac{Y_{max}-Y_{ij}}{Y_{max}-y_{min}}$$

Step 3: Determine grey relational coefficient (GRC) for normalized S/N ratio values.

$$GRC_{ij} = \frac{\Delta_{min} + \delta \Delta_{max}}{\Delta_{ij} + \delta \Delta_{max}}$$

Where $i=1,2,3,\dots,n$ is number of experiments and $j=1,2,3,\dots,m$ is the responses.

GRC_{ij} =GRC for i th trial, Δ =absolute difference between Y_{oj} and Y_{ij} , Δ_{max} =maximum value of Δ , Δ_{min} =minimum value of Δ , δ =is the distinguishing coefficient ($0 \leq \delta \leq 1$)

Y_{oj} =optimum performance value or the ideal normalized value of j th response

Y_{ij} =the i th normalized value of j th response/dependent variable Δ

Step 4: calculate the grey analysis grade ($GRGi$)

$$RGi = \frac{1}{m} \sum GRGi$$

Where m is the number of responses

Step 5: select optimal levels for the factors based on the maximum average $GRGi$ value using ANOVA.

3.12. Secondary operation

3.12.1. Extrusion process

The deform process which reduced the diameter and extend the length, the feedstock material is squeezed to flow through a small hole with a constant cross-section or one with a variable cross-section. When a piston or plunger is forced into a die, either hot or cold, metal is formed. The extruded metal is given a die shape as it passes through a hollow. In order to lower diameter and increase length, a circular billet must be pressed through the die hole. Hot extrusion process the force applied through die which takes place above crystallization temperature about 50-60% of its melting temperature. Hot extrusion and effects of different fabrication parameters on microstructural, physical, mechanical and thermal properties as well as low required compare to cold working, easy to work in hot form and product is free from strain hardening. When the fabricated metal composite pass through under secondary operation (metal forming) like extrusion, forging, rolling can obtained better grain size structure and good mechanical strength (Kumar et al., 2023).

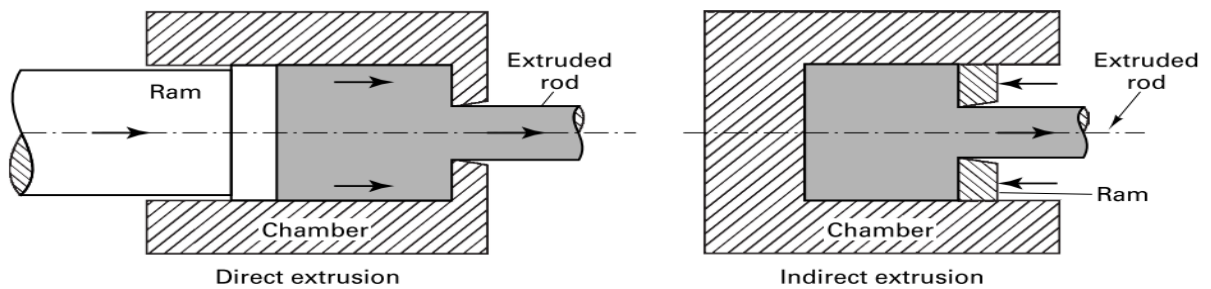


Figure 4.15 Direct and indirect extrusion process (Black & Kohser, 2017)

3.12.2. Selection of extrusion process parameter

In hot extrusion process of composite is oxidation cause by increase temperature that occurs over the surface of the hot work, oxide scale build up on external surface work piece scale can affect surface finish and accuracy, increase friction and wear at die

interface. Robust finite element model to determine the most influential parameters and was studied influence of them on extrusion force, stress and damage combined by Box Benhken Method. In this research, the effect of parameters on deforming process (extrusion speed, extrusion ration and temperature) three different levels and decided optimum value of applied load, stress and damage to analyzed and identified.

Table 4.3 Levels of parameters and their range used for an experiment

S. No	Process parameters	Levels		Units
		Low	High	
1	Extrusion ratio (A)	2	4	mm/min
2	Extrusion speed (B)	1	3	
3	Temperature (°C)	850	950	°C

3.12.3. Design of Experiment for analysis parameters

DOE (design of experiment) is the tool used to analysis parameters and influences of process on the response variables. The response variable is an unknown function of the process variables, which are known as design factors or independent variables. Selected parameters depend on literature review and response variables (damage, load and stress) combined Design-Expert 13 software. In order to used Design-Expert 13 software to selected RS (response surface) method was used to combine by BBD method and performed regression models. BBD method used to combine selected parameters of and response variables extrusion process for simulation process. Before simulation parameters of extrusion process was selected and combined by BBD method and after combined parameters used 3D-deform software used to obtained value of response variables. Based on obtained value of response variable to analysis of variance (ANOVA) was done and adequacy checking of regression models.

3.12.4. Finite element analysis through hot extrusion

Finite element analysis is a method of tool used for simulating and analyzing different manufacturing processes, like hot extrusion, rolling, and forging processes. It is used to define the state of a material during hot extrusion of the effects that occur and the factors of those effects, which include temperature distribution, stress and strain distribution, deformation, and material flow. The numerical modeling and study of the deformation behavior of three-dimensional objects or structures is required for the simulation of 3D

deformations. Simulating 3D deformations provides insights into the effects of process parameters and material properties on the extrusion process like: billet temperature, lubrication condition, material properties to optimize the process and achieve desired outcomes. Main step of DEFORM-3D seen as follow:

Pre-processor: A preprocessor in the context of 3D deformation simulation is an object of software or a series of steps used to set up the simulation's parameters and prepare the input data before it is performed. In pre-processor before run software at begin to completed geometry import, material define, meshing, boundary condition. Geometry import used to allow importing geometry of object to be simulated. Imported top die, work piece, bottom die and holder for simulation process in geometry import of pre-processor. The geometry was separated into a finite number of elements by the preprocessor, which creates a computational mesh. After mesh to defined material for top die, work piece, bottom die and holder depend on mechanical properties. For define material used to basic equation of rigid-plastic element.

$$\text{Equilibrium equation} \quad \sigma_{ij,j} = 0 \quad (3.15)$$

Where, $\sigma_{ij,j}$ = the stress at the boundary surface

$$\text{Velocity strain-rate relations and incompressibility equations:} \quad \dot{\epsilon} = \frac{1}{2}(u_{i,j} + u_{j,i}) \text{ and } u_{i,j} = 0 \quad (3.16)$$

$$\text{Yield criterion, } f(\sigma_{i,j}) = 0 \quad \bar{\sigma} = \sqrt{\frac{3}{2}(\sigma'_{ij} \sigma'_{ij})}^{1/2} = \bar{\sigma}(\bar{\epsilon}, \bar{\epsilon},) \quad (3.17)$$

$$\text{Constitutive equation} \quad \sigma'_{i,j} = \frac{2\bar{\sigma}}{3\bar{\epsilon}} \dot{\epsilon}_{i,j} \quad \bar{\sigma} = \sqrt{\frac{3}{2}(\sigma_{i,j} \sigma_{j,i})} \quad \bar{\epsilon} = \sqrt{\frac{3}{2}(\dot{\epsilon}_{i,j} \dot{\epsilon}_{j,i})} \quad (3.18)$$

Where, $\bar{\sigma}$ is effective stress, $\bar{\epsilon}$ is effective strain, $\dot{\bar{\epsilon}}$ is the effective strain rate $\dot{\epsilon}_{i,j}$ is strain rate.

$$\int_V \epsilon \delta \bar{\epsilon} dV + K \int_V \epsilon_v \delta \epsilon_v dV - \int_{S_F} F_i \delta u_i dS = 0 \quad (3.19)$$

Where, S is surface area of sample V is surface volume of the sample being examined, K is penalty constant.

Defined material of copper composite depends on result obtained from compression test for simulation. Only the work piece's flow stress should be specified when using DEFORM 3D software to solve an isothermal forming problem involving rigid dies and rigid-plastic work piece. Flow stress data selected from compression test of sample. Flow stress expressed by Hollomon's equation (Hao et al., 2017).

$$\bar{\sigma} = K\dot{\epsilon}^n \quad (3.20)$$

Where, $\bar{\sigma}$ is flow stress, $\dot{\epsilon}$ is true strain; K is material constant and n is strain hardening constant. At temperatures above the recrystallization temperature, the strain rate mostly determines the flow stress.

$$\bar{\sigma} = C\dot{\epsilon}^n \quad (3.21)$$

Power law used as constitutive equation to determine flow stress for simulation of copper composite developed during hot deformation. Defined property of copper composite depended on power law in 3D deform software for simulation process.

$$\bar{\sigma} = C\dot{\epsilon}^n \dot{\epsilon}^m + y \quad (3.22)$$

Where, n is strain exponent, m is strain rate exponent, C is material constant and y represented initial yield value. During a compression test, the initial yield stress is determined using the stress-strain curve. The boundary between elastic and plastic behaviour is known as the yield stress.

$$\epsilon = \log(1 + e) \quad (3.23)$$

Where, ϵ is true strain

$$\bar{\sigma} = S(1 + e) \quad (3.24)$$

Where, σ is true stress, e is engineering strain and S is engineering stress.

The metal's behavior throughout the forming process is determined by the strain hardening exponent (n). Strain hardening exponent and strain rate exponent is calculated by below equation.

$$n = \frac{d\log(\sigma)}{d\log(\epsilon)} \quad (3.25)$$

$$m = \frac{d\log(\sigma)}{d\log(\dot{\epsilon})} \quad (3.26)$$

Strength coefficient K is calculated by below equation.

$$\log(\sigma) = \log(K) + n \log(\epsilon) \quad (3.27)$$

Depend on above all equation defined material. Meshed geometry objects of top die, bottom die and work piece in pre-processor.

Input data for simulation in extrusion process for new material

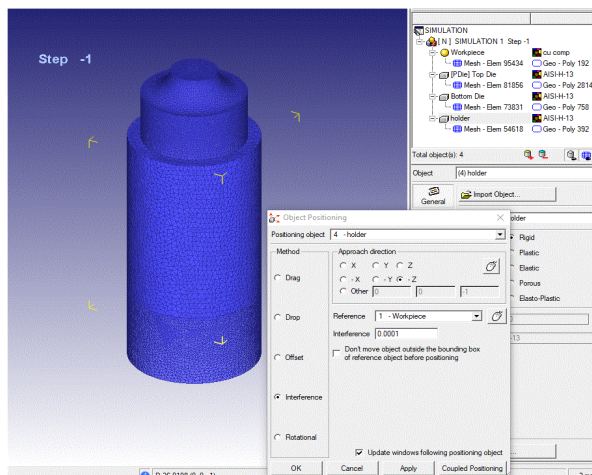
1, Density = 8.0932g/cm³ density of selected material

2, Compression strength = 393.758MPa

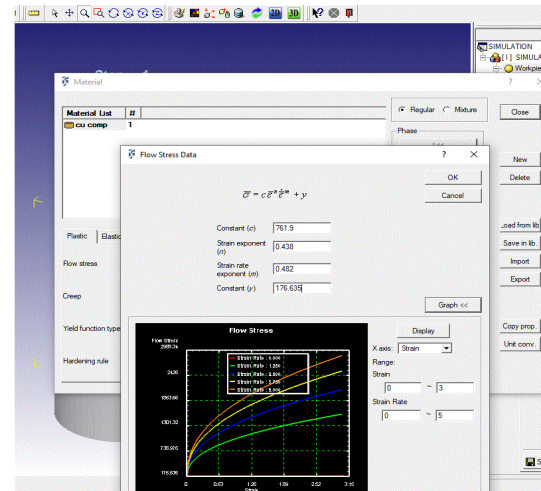
3, Flow stress constitutive equation formula $\bar{\sigma} = C\dot{\epsilon}^n \dot{\epsilon}^m + y$

C=material constant =761.9, n= strain exponent=0.438, m=strain rate exponent =0.482 and σ_y =initial yield value=176.635MPa

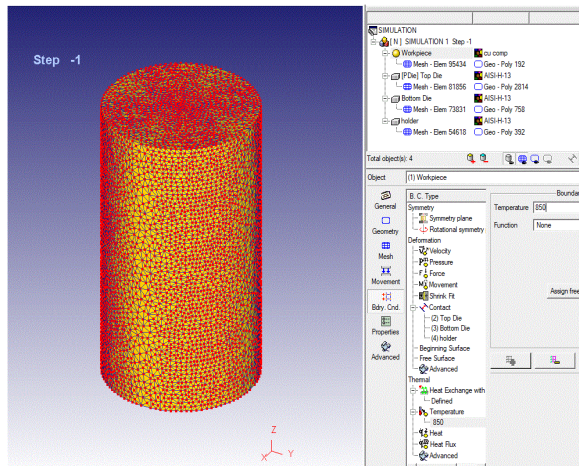
4, Constants define in power law and properties of material thermal conductivity=30.5W/mk, specific heat=380J/kgk, emissivity =0.15, Coefficient of friction =0.3 used as lubricate and mesh number =80,000.



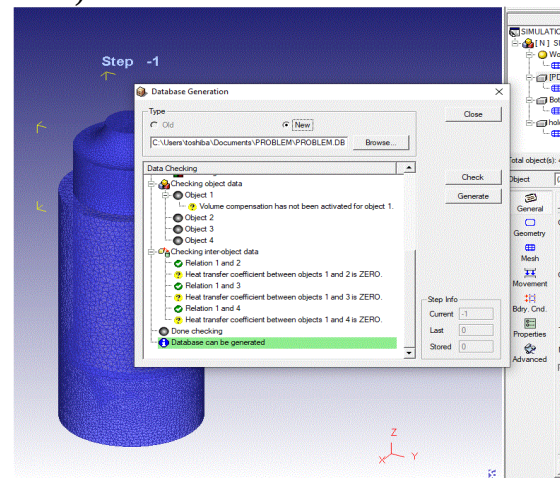
a) Meshed and object position



b) defined new material



c) Boundary condition



d) checked and generated database

Figure 4.16 pre-processor of 3D deform

Simulation Engine: used to carry out the mathematical calculations required to examine the procedure, storing the results in a database file. To control run process with to create a FEM mesh on the billet, die, the simulation engine and the Automatic Mesh Generation (AMG) system communicated.

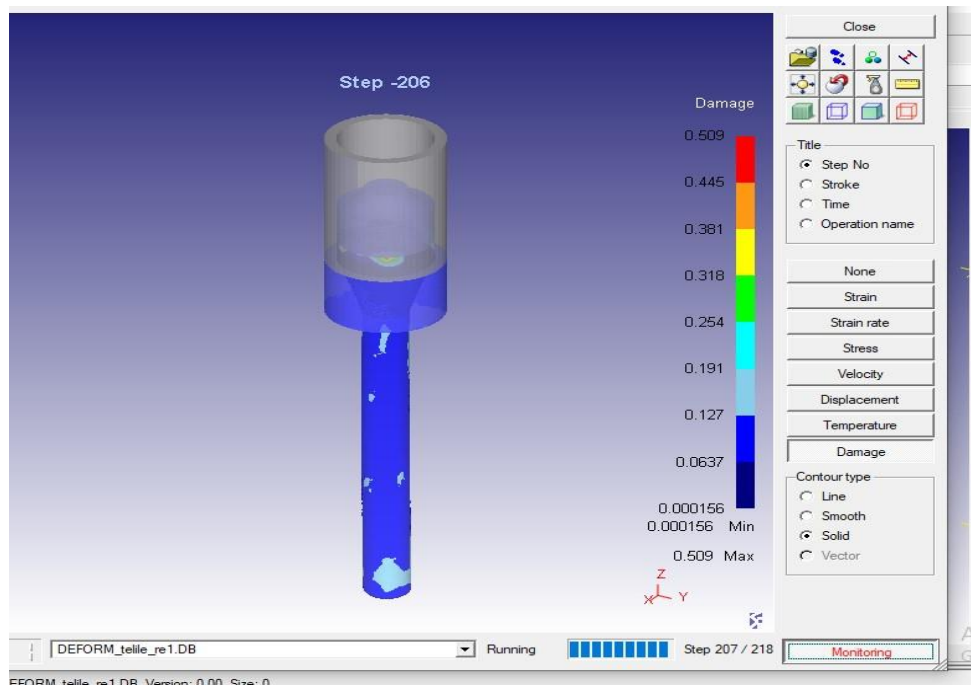


Figure 4.17 Simulation engine when software start running database

Post Processor: It is possible to create the geometry following deformation with tool movement at each stage from different viewpoints. A graphical user interface is provided by the postprocessor for viewing geometry, field data such as strain, temperature and stress, and other simulation data such as die loads. It is possible to plot and extract the graphs between any two state variables about any two abscissas.

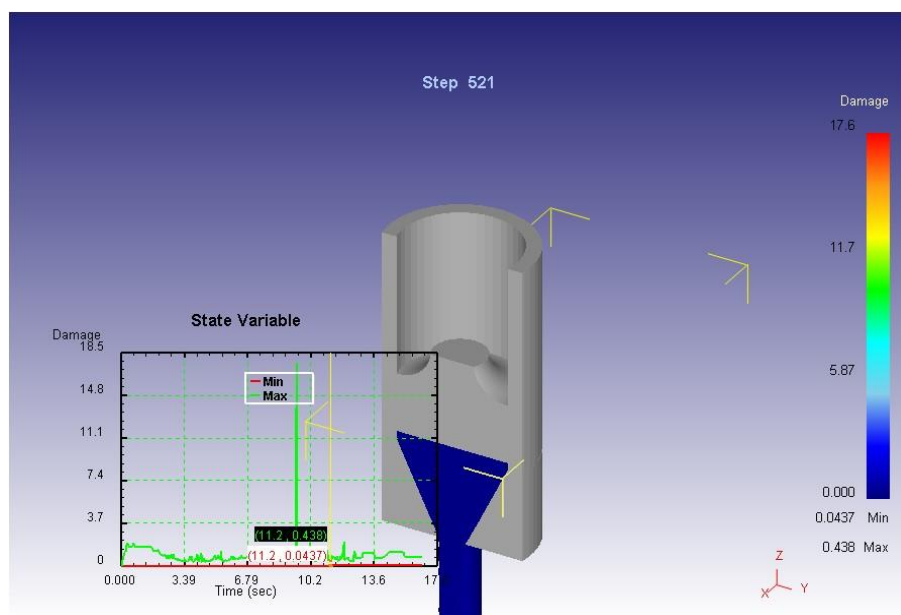


Figure 4.18 Post-processor extracted data of 3D deform

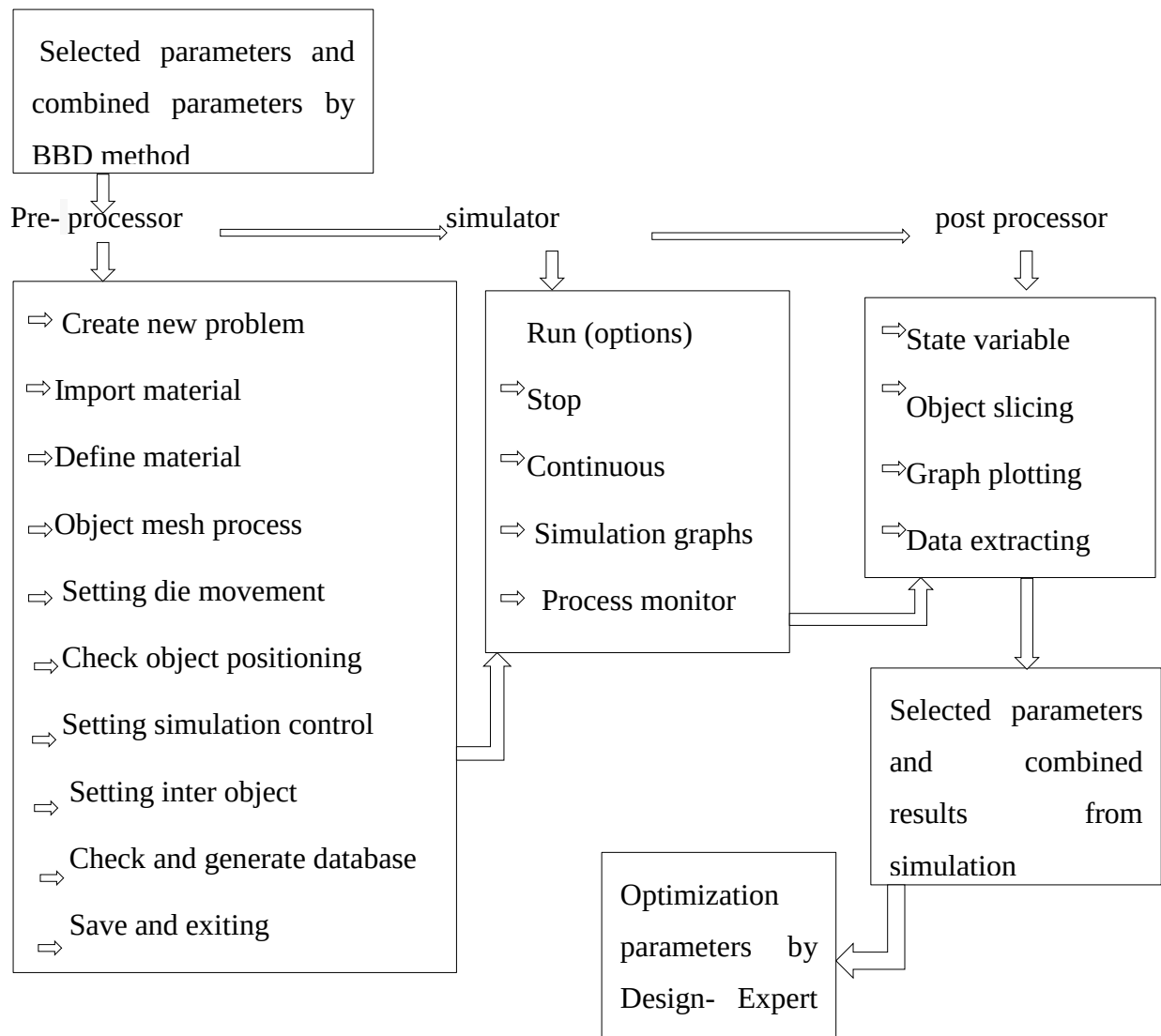


Figure 4.19 Simulation procedures

3.13. Multi-objective Optimization of Process Parameters Using GA

Genetic algorithms which generate solutions to multi-objective optimization problems with equation formed in MATLAB used different techniques involved by natural evolution, such as inheritance, mutation, selection, and crossover. In a genetic algorithm, a population of candidate solutions in formed equation like linear, quadratic equal for regression model to an optimization problem was evolved toward which is the better solutions. Commonly, the algorithm terminates when either a maximum number of generations has been produced, or a satisfactory fitness level has been reached for the population. A typical genetic algorithm requires:

1. A genetic representation of the solution domain.
2. A fitness function to evaluate the solution domain.

The main important of developed a Genetic Algorithm (GA) which can be applied to optimized the extrusion process parameters to obtained the better value of damage, load and effective stress for surface finish in extrusion process. Even though it is initially developed for extrusion process parameter optimization, it can be easily applied to any optimization problems.

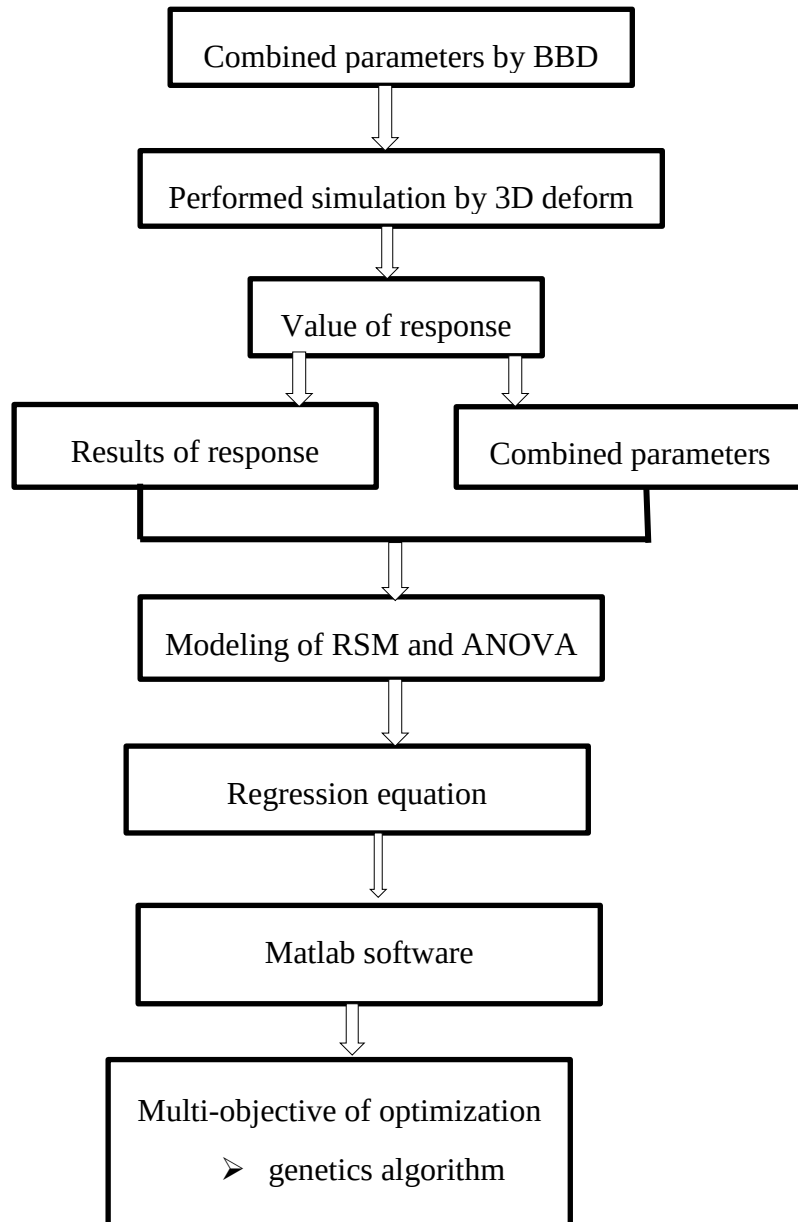
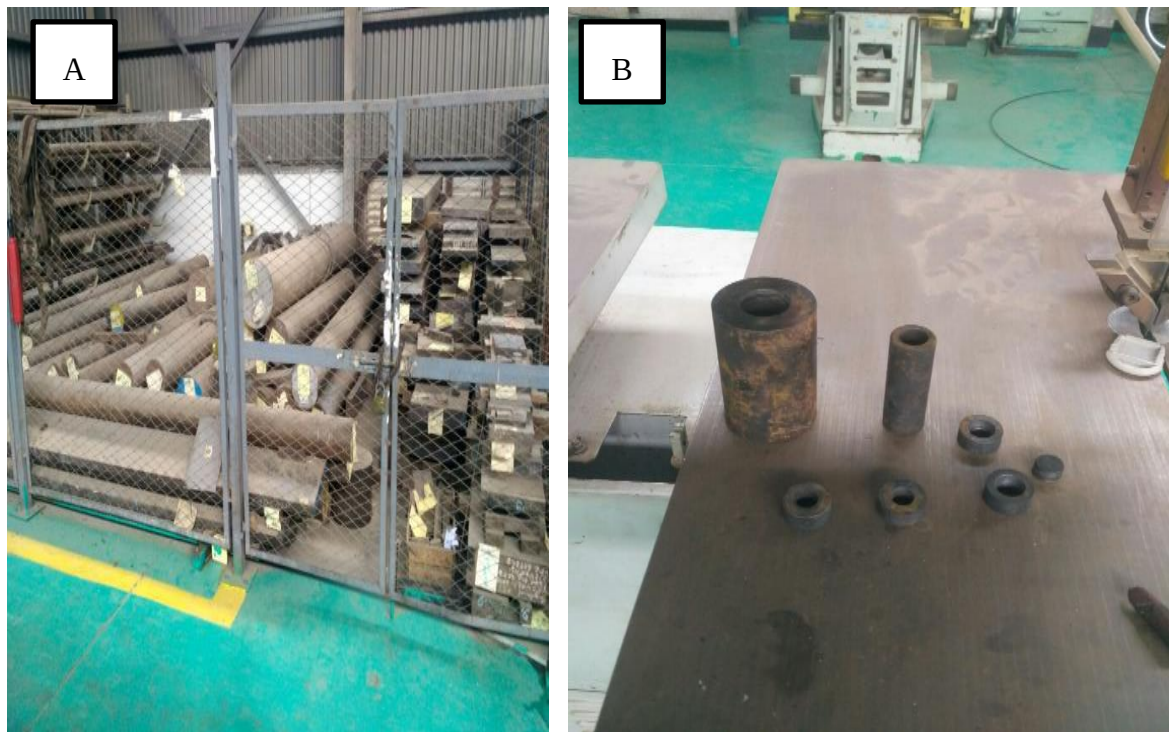


Figure 4.20 summarized the Schematic diagrams of experimental procedures

3.14. Die profile and die fabrication

Die profile an important to guiding the flow of material by applied force/load through ram in hot extrusion process. Flow of material in extrusion process is directly influenced by die profile and friction condition between ram and work piece or billet, profile angle like:

stream line die, cosine die. Cosine die selected because of enables greater flexible of design and improved material flow with reduced defects, reduced distortion of the final extruded shape(Huang et al., 2019). Cosines die to help minimize the residual stress within the extruded material which improves quality and performance the final product. The load requirement for the deformation process was determined by using a cosine die with the punch, billet, and container, and the geometry was designed by solid work software. After designed cosine die selected material AH13 die steel stock material was found in Addis Ababa from MIDI and made through machining process. The hydraulic press was pressed by the applied load, resulting in an optimal value and die fabrication based on the extrusion ratio.



a) steel type of material found in A.A (MIDI) b) cosine die

Figure 4.21 Fabrication of die

Table 4.4 The type of equipment's and its purpose in laboratory

No	Equipment	Purpose
1	Furnace	Used to melt copper alloy changes to liquid state
2	High energy ball milling	More mixing reinforcements (Gr, SiC and CeO ₂) suitable for uniformly distributed and reduce agglomeration
3	Stirrer	Mixing matrix and reinforcements in furnace.
4	Lath machine	Used for prepare sample as standard of equipment's for test by turning and facing.
5	Sand paper	More finish process and fine process of sample suited for polisher machine.
6	Polisher	Used to form flat mirror-like face of ample and free from debris.
7	Killer's etchant	(FeCl ₃ , HCL and distilled water) Avoid dust, debris and obtain more clear image for OM.
8	XRD	Phase analysis, strain crystallize, dislocation of sample by using relative peak intensity.
9	SEM	Study of morphology of sample, wear porosity after wear test and fractography after compression test.
10	OM	Test to structural characteristics of sample.
11	hardness test	Study hardness of sample.
12	Compression test	Measure to amount compressive strength of materials.
13	Pin on disc	Used to test wear resistance
14	Extrusion machine	Used to deform material applied load by ram and form desired shape of materials.

CHAPTER FOUR

RESULTS AND DISCUSSIONS

4.1.Introduction

In this analysis, the results obtained and done in this research are discussed in depth. In this section of the research, the results of the research work are divided into different sections and the results of this research are focused on providing great benefits in the future. The results of experimental work done have been discussed on the properties of copper metal matrix composite by tested in XRD, SEM, optical microscopy, hardness test, corrosion test, compression test, determination of density, wear test of samples fabricated via stir casting process and compared results got from secondary operation (through hot extrusion).

4.2.Microstructural characterization and analysis

4.2.1. XRD analysis

Phase formation between reinforcements SiC, CeO₂, Gr, and copper was investigated using X-ray diffraction of Cu-10SiC-5CeO₂-4Gr, Cu-7.5SiC-7.5CeO₂-4Gr, Cu-5SiC-10CeO₂-4Gr samples were phased at different angles (10° - 2θ – 80°) at scan speed $3^{\circ}/\text{min}$. As illustrated in the figure below for ceramic materials, the diffraction peaks of the samples were compared to XRD-JCPDS files of different materials. As figure 4.1 show that the 2θ value for copper at 43.3° given the maximum peak value in plane (1 1 1). SiC peak point value was generated at angle 50.4° and CeO₂ was recoded at angle 74.2° .

The peak intensity of the reinforcements is lower than that of the matrix because of the low weight percentage; it may even be below the minimum value for XRD method identification (Olani et al., 2023).

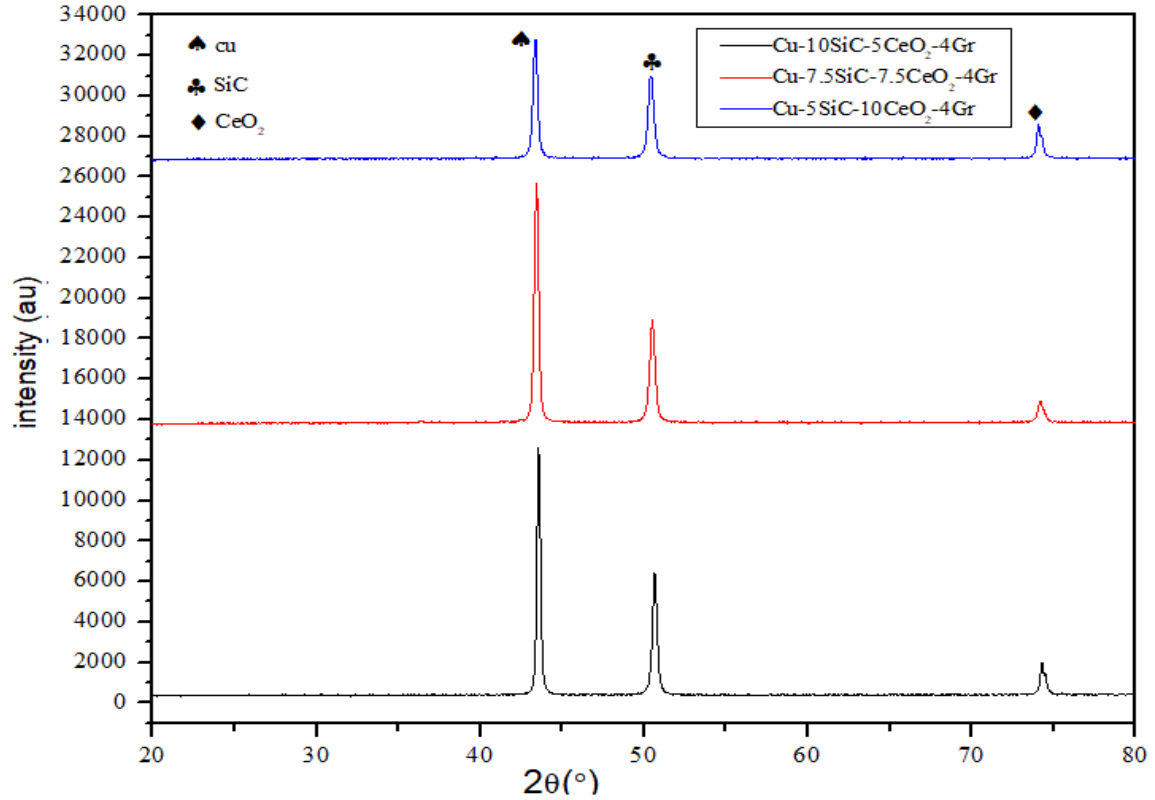


Figure 5.1 XRD analysis of copper composite

4.2.2. Crystallize size of fabricated samples

The crystalline size of the samples was obtained using data from XRD analysis. Since the K value of 0.9 was taken as Scherrer's most extreme acceptable condition and the studied on CMMC used the average calculation of crystallize size, the diffraction peak point was calculated from the XRD data by us $\lambda = 0.15406(\text{\AA})$ (Sun et al., 2020). According to the XRD data and the results obtained, since crystallite size was calculated, the smallest crystallite size was selected depending on the application area. Small crystallite size is often indicative of fine microstructure and can be associated with improved mechanical properties such as strength or increased hardness. Depend on result of below table average crystallize size smallest and best for application is sample 2 or $D = 28.99\text{nm}$.

Table 4.1 shows that results of crystalline size was calculated based on data which obtained from XRD analysis as the crystalline size decreases, the material's strength and hardness generally increase. So, when compared sample 2 with each other it is has lower crystalline size and improved mechanical property.

Table 5.1 The Crystallize size of samples

Sample Code	Parameters Obtained from XRD Data		Calculation of data from XRD peak analysis	
	$2\theta(^{\circ})$	$\beta(^{\circ})$ FWHM	D (nm) crystallize size	D(nm) average crystallize size
S1	43.3541	0.3142	28.4176	29.96091
	50.4031	0.3732	24.5719	
	74.1035	0.2818	36.89323	
S2	43.4268	0.3008	29.69103	28.99275
	50.4913	0.3518	26.07606	
	74.1934	0.3333	31.21115	
S3	43.5654	0.2129	41.96981	39.85736
	50.6387	0.2717	33.78407	
	74.3174	0.2376	43.81819	

4.2.3. Lattice strain and dislocation density of fabricated sample

Dislocations density in the copper matrix composite can also be caused by the mechanical stirring of the molten metal and reinforcements during the stir casting process. Dislocations may arise and multiply as a result of the turbulent flow and shear pressures experienced during the stirring process. The parameters of micro structure have estimated using the XRD peak analysis Gauss method of the Scherrer formula. The numerical dislocation density of sintered composite samples was estimated by equation (3.8).when decrease in crystalline size, an increase in lattice strain, and a rise in dislocation (Kundie et al., 2018). The development of residual stresses and a rise in lattice strain can be attributed to the integration of reinforcing phases and the mismatch in thermal expansion coefficients between the copper matrix and the reinforcements. When samples made by stir casting or other comparable processing methods, decrease in crystalline size, increase in lattice strain, and increase in dislocation density are frequently observed. As obtained analysis results from table 4.2 started crystalline size to increased sample 2 (28.99nm), sample 1 (29.96nm) and sample 3 (39.857nm) but, the lattice strain reduced from sample 2(2.87), sample 1 (2.84), sample 3 (2.06) and the dislocation density reduced from sample 2(1.21 nm⁻²), sample 1(1.209 nm⁻²) and sample 3(0.654 nm⁻²). According to the Hall-Petch connection, the yield strength and hardness of the material rise as the grain size (crystalline

size) decreases. So, more grain boundaries from smaller grains prevent dislocation movement, strengthening and resisting plastic deformation of the material.

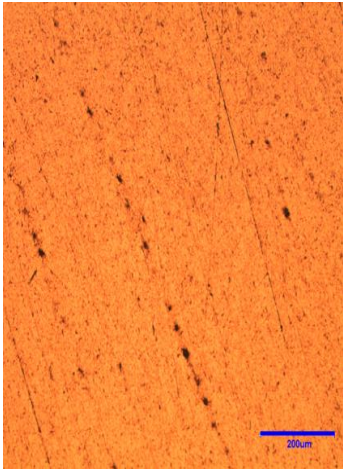
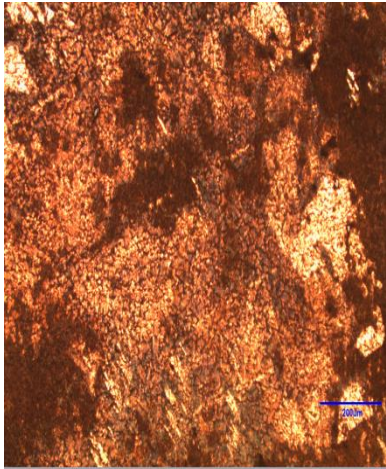
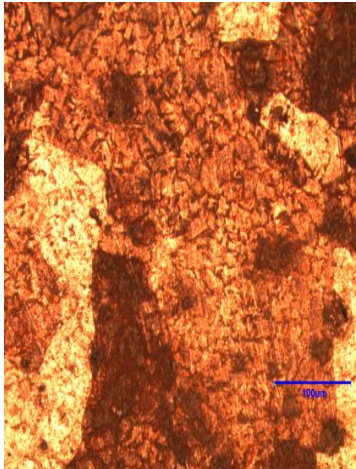
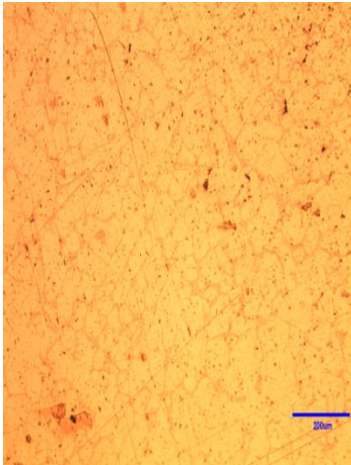
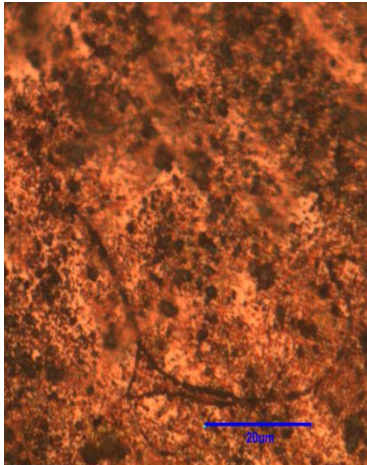
Table 5.2 Crystalline Sizes, Dislocation Density and Lattice Strain Experimental Calculation

Sample Code	Parameters Obtained from XRD Data		Calculation of data from XRD peak analysis		
	$2\theta(^{\circ})$	$\beta(^{\circ})$ FWHM	D (nm)	$\delta \cdot 10^{-3}(\text{nm}^{-2})$	$\epsilon \cdot 10^{-3}$
S1	43.3541	0.3142	28.4176	1.23829	3.44908
	50.4031	0.3732	24.5719	1.65623	3.46026
	74.1035	0.2818	36.89323	0.73469	1.62865
Average	-	-	29.96091	1.20974	2.846
S2	43.4268	0.3008	29.69103	1.13456	3.29599
	50.4913	0.3518	26.07606	1.47067	3.25534
	74.1934	0.3333	31.21115	1.02655	1.92315
Average	-	-	28.99275	1.21052	2.87479
S3	43.5654	0.2129	41.96981	0.567709	2.32458
	50.6387	0.2717	33.78407	0.87614	2.50578
	74.3174	0.2376	43.81819	0.52082	1.36788
Average	-	-	39.85736	0.6548	2.06608

4.3.Optical microscopy

The reinforcements particles distributed in matrix material (Gr, SiC and CeO₂ uniformly distributed in copper alloy) via stir casting was discussed and observed by using optical microscopy. The current investigation employed magnifications of 20x, 50x, and 100x times to determine the morphology and distribution of reinforcement particles within the copper matrix. In this section briefly discussed the grain boundary of samples before etchant and after etching as below image. Before etchant the grain boundary samples visualization image could not be obtained with qualitative accuracy. After etchant, however, the grain boundary image of the sample could be clearly observed. Therefore, immersion in the solutions (25ml of HCL, 25gm of FeCl₃ and 100ml of distilled water) for 30 second showed the sample image below. Figure 4.3 S1, S2 and S3 shows the images of copper reinforced with SiC, Gr and CeO₂ was observed by optical microscope at different magnifications. As the samples of copper metal matrix composites observed the nano-

crystalline structure was the larger grains were broken down and prevented from expanding by the strong mixing by mechanical stirring, which results in the performed of a finer and more uniform grain structure. The grain size was determined using the line intercept depending on the OM image. The porosity level and grain size of the fabricated composite with different weight percentages through stir casting were analyzed using ImageJ. Calculated the size by using ImageJ was obtained result $S1=47.992\mu m$, $S2=42.0802\mu m$ and $S3=67.044$ with selected sample 2 was improve mechanical properties with small gain size.

S1  Before etchant (200µm)	S1  After etchant (100 µm)	S1  After etchant (200 µm)
S2  Before etchant (200µm)	S2  After etchant (100 µm)	S2  After etchant (200 µm)

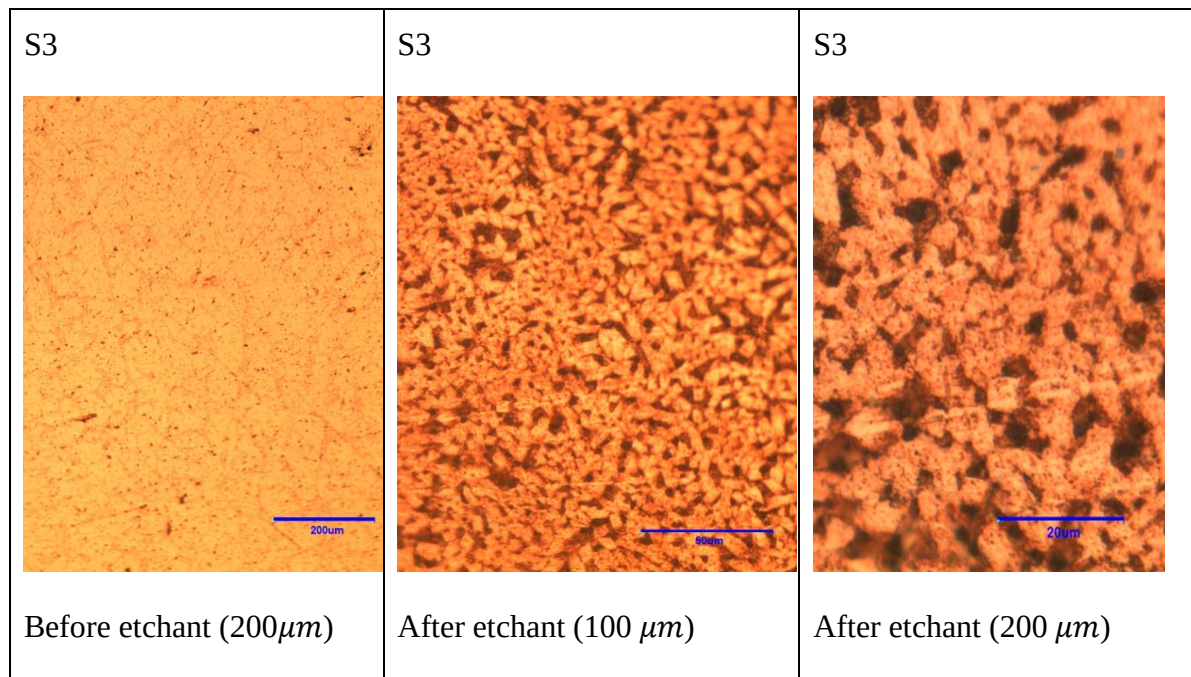


Figure 5.2 Optical microscope image before etchant and after etchant of sample.

4.4.SEM analysis of copper composite fabricated through stir casting

The distribution of reinforcements in copper matrix composite since it has a significant effect on the final mechanical properties of composites. The dispersion and distribution of the reinforcements, SiC, CeO₂ and Gr phases inside the copper matrix composite can be investigated using SEM. Through the use of SEM, the grain or crystallite size of the copper matrix can be seen, making it possible to measure and observe directly how the stir casting process and the presence of reinforcements has caused the crystalline size to decrease. SEM observed uniformly distribution of reinforcement particles in matrix to shows presence of porosity in sample fabricated (Ali et al., 2020). The results obtained from Figure 4.3 show the distribution of reinforcements in copper matrix material and the presence of porosity when performing agglomeration due to the fabricated process. Sample 2 good uniformly distributed the reinforcements in copper matrix when compared with other samples. As below figure when SEM analyzed based on the information provided, was visible on SEM image like porosity, distribution of reinforcements, void, crack and inclusions.

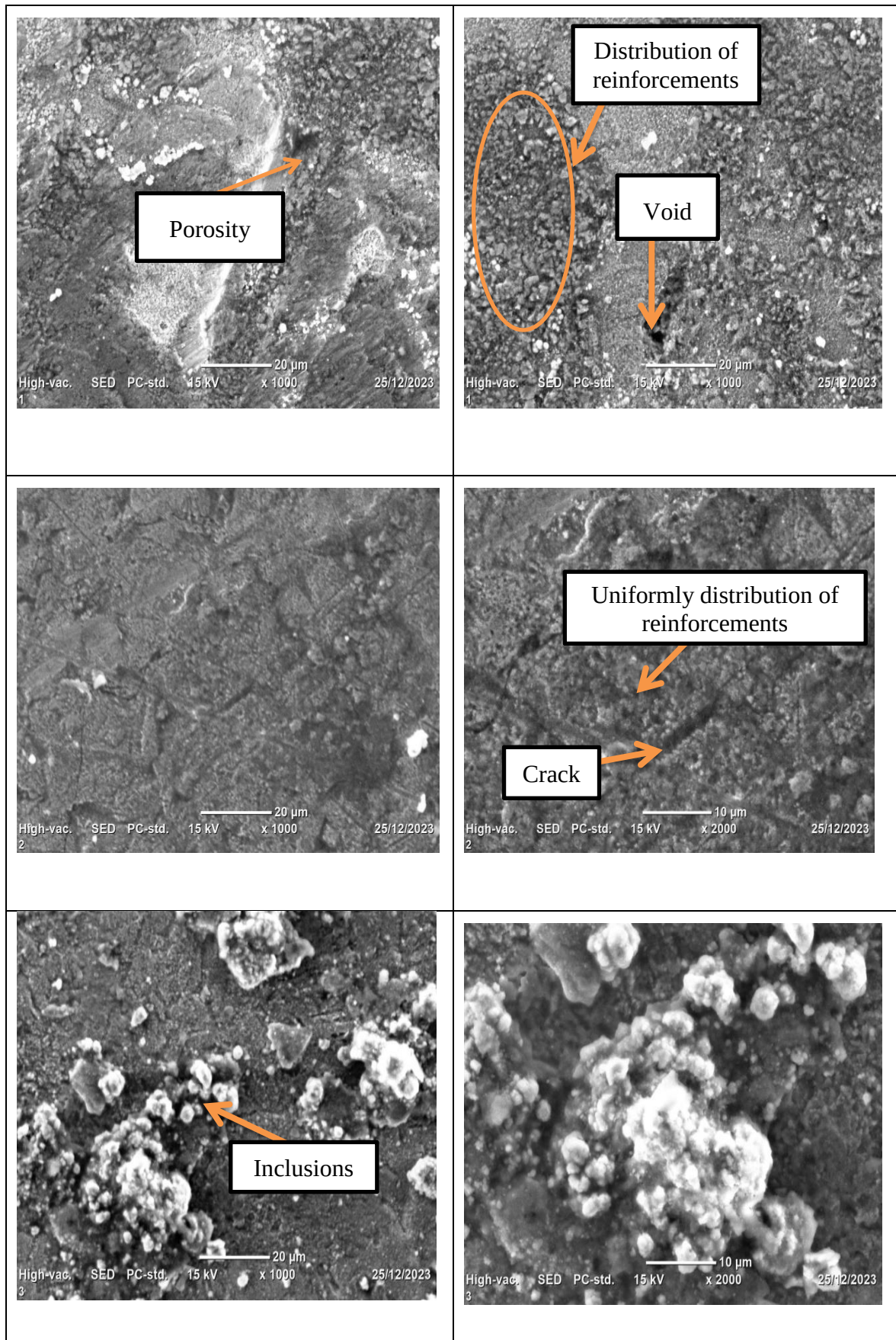


Figure 5.3 The SEM image micrograph of samples

4.5. Analysis of Density and Porosity of fabricated Samples via stir casting process

Analysis density and calculated porosity of copper metal composite to improved mechanical properties with related like hardness, compressive and wear rate. In density analysis actual density was decreased when compared with theoretical density of composite materials. When actual density of samples reduced the porosity of sample was increased. Reducing the porosity could improve mechanical properties with compressive, hardness (Meng et al., 2020).

Table 5.3 Analysis of density with porosity level of samples

samples code	Theoretical density(g/cm3)	Actual density(g/cm3)	Porosity (%)
S1	7.725	7.5234	2.6
S2	8.115	8.0451	0.86
S3	7.5644	7.4253	1.83
Pure	8.94	8.612	3.66

The theoretical density of samples was calculated by adding the mixed matrix components and reinforcements inside the sample volume using the mixing rule for matrix and composite using equation (3.3). The difference value between the theoretical density and actual which measured density to explain amount of porosity to perform voids and present pores in fabricated samples. When the weight percentage of reinforcement particles added to the matrix material was increased, the volume available for the matrix composite materials decreased. The reduction in matrix material volume leads to reduce in the overall porosity of the composite. Reduced the porosity result performed in samples to show small porous, small porous indicated uniformly distributed reinforcement particles in matrix material and can reduced the measure density of composite with improved mechanical properties. So, on table 4.3 to show the sample 2 (81%Cu-7.5%CeO₂ -4%Gr) the difference value between actual density and theoretical density is smallest to other samples, its show small porosity and improved mechanical properties.

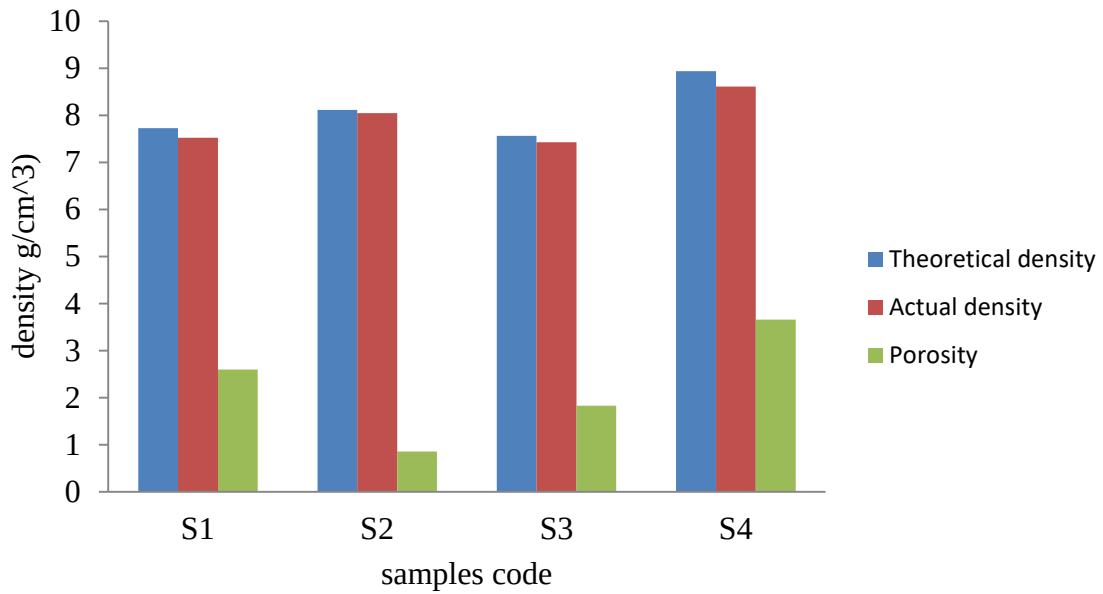


Figure 5.4 Theoretical density and actual density of copper composite

4.6.Hardness test

The results from the hardness test now defined the hardness of the sample by more resistant of penetration or scratching that composite. When Vickers test machine used to careful without shocking load, checked good image of specimen by optical camera with applied force on diamond indenter to obtained indentation of hardness samples. Work in the hardness test which defined the difference between the maximum and minimum values of samples to describing their hardness amount and from pure copper to which reinforcement has been added. The derived hardness values depend on the differences in the types of reinforcements and reinforcement content added to the matrix composite. So, the hardness values of the samples change when the percent of reinforcements change. As the percentage of SiC added to the matrix increases, the hardness of the composite increases (Somani et al., 2018) . The hardness value of the copper becomes much better with the addition of SiC reinforcement at the expense of its ductility (Kumar et al., 2020) and (Kumar et al., 2021). Because SiC is a ceramic particle which helps to improve the hardness of pure copper. When added increasing weight percentage of the reinforcements to the matrix, the grain boundary strength increases while the displacement density of atoms decreases, which provides strength to the matrix and consequently increases the strength of the composite (Radhika et al., 2021). Overall the hardness values of copper metal matrix (S1, S2 and S3) were improved by 47.94 %, 51.73 % and 32.21 %

respectively when compared with non-reinforced copper. Sample 2 selected out samples for best hardness property.

Table 5.4 Results of hardness test

Sample's code	Experimental Results Hardness values (HV) of the samples.				
	Trail 1	Trail 2	Trail 3	Average values	Percent of improve (%)
S1	110.7	85.2	96.7	97.533	47.94
S2	103.9	99.8	96.4	100.033	51.73
S3	86.5	86.4	88.6	87.166	32.21

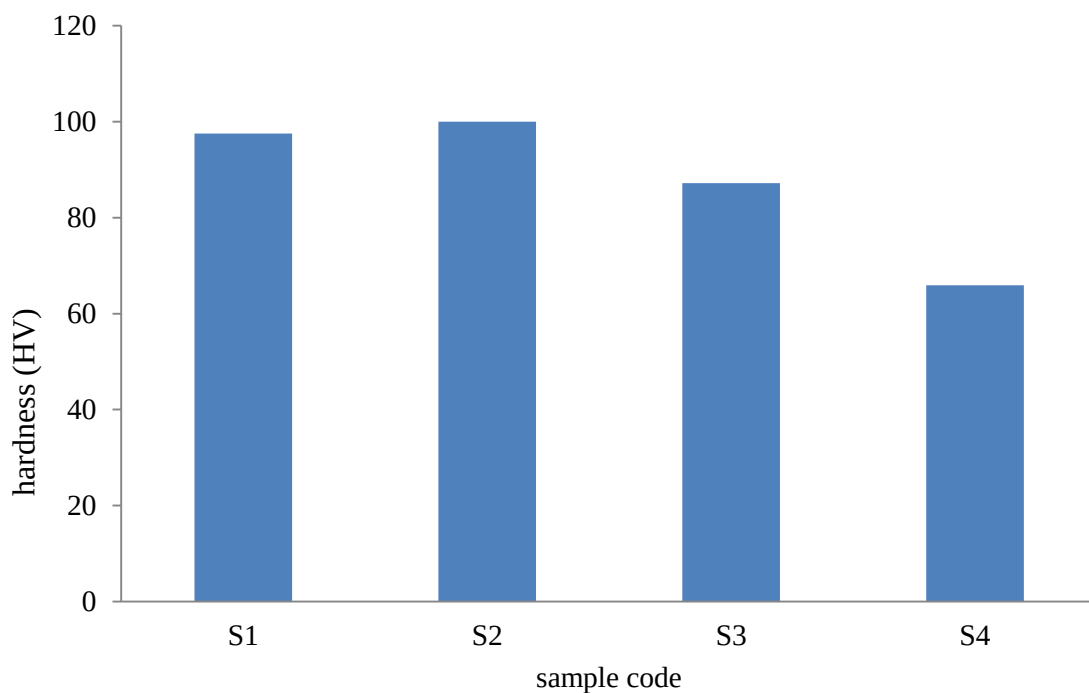


Figure 5.5 The Average Vickers Hardness

4.7.Wear test analysis

Copper is the material of choice for the current study because of its strong load-bearing and wear-resistant qualities (Radhika et al., 2018). Reduced wear rate is recognized as an advantage that denotes improved wear resistance and a longer lifespan for the material or component. The strong ceramic character of CeO₂ can increase the composites based on copper's composite resistance to wear. Wear property of copper metal composite was examined under load 20N and sliding speed 400rpm with 35mm wear truck of diameter for

20min. Volume loss was calculated as equ (3.15) by difference between mass loss before and after wear test. Wear rate reduced when CeO_2 .

Table 5.5 wear test value of copper metal matrix composite

Samples code	Mass in(gm) before wear test	Mass in(gm)after wear test	Volume loss %	Wear rate
S1	2.8932	2.7276	2.23	0.446
S2	2.8689	2.772	1.28	0.256
S3	2.985	2.7331	3.13	0.626

As above table the results of wear rate to show that the reinforced 7.5%SiC-7.5% CeO_2 -4%Gr (S2) composite exhibit lower wear rate than pure and other samples, it has lower wear rate and volume loss. So, when lower wear rate high wear resistance with improved mechanical property of CMMC by added reinforcements.

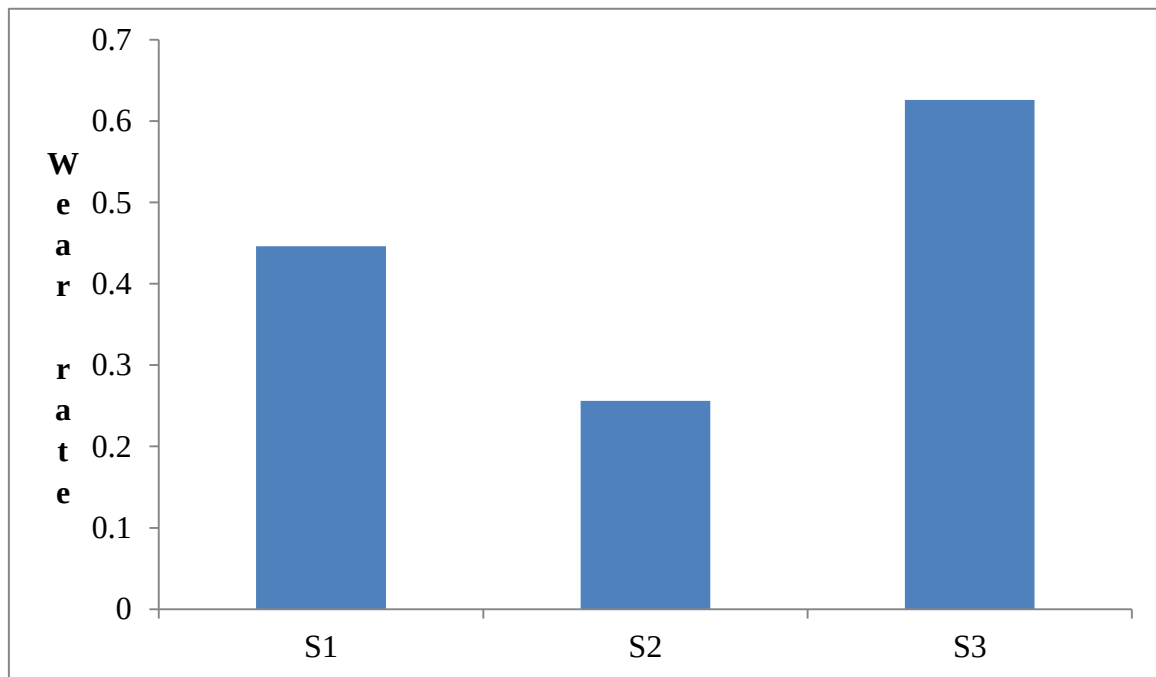
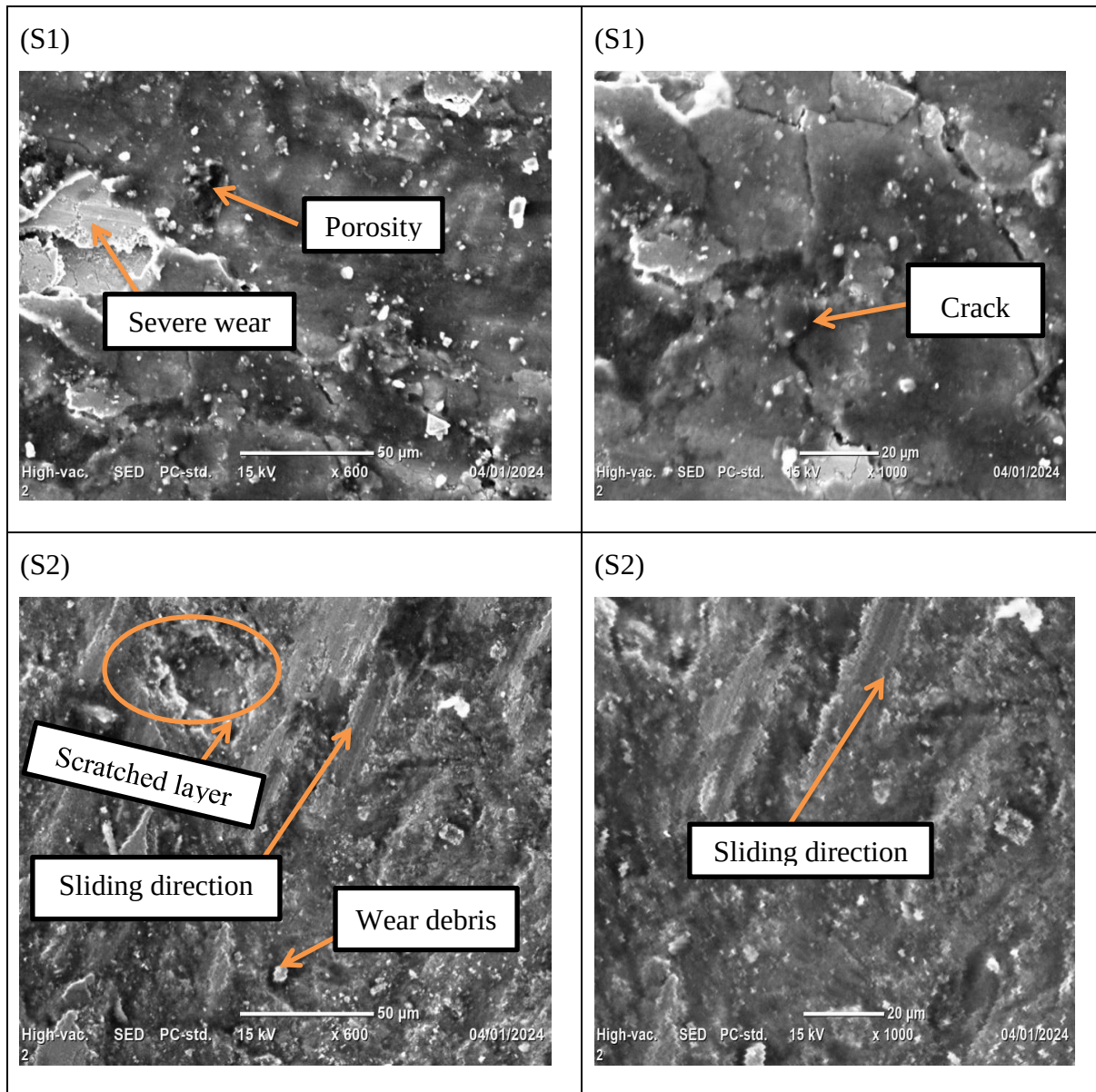


Figure 5.6 Graph of wear analysis

Graph taken during wear test revealed that sample indicated sample two shows decrease in wear rate, the material exposed to the wear environment increases when compared with the other samples as shown on figure 4.6.

4.7.1. Wear morphology analysis

SEM is an effective instrument for examining the topography and surface characteristics of a surface or worn component. Wear morphology analysis was used to SEM studied surface topography features like grooves, cracks, delamination clearly observed and wear debris analysis within wear mechanism.



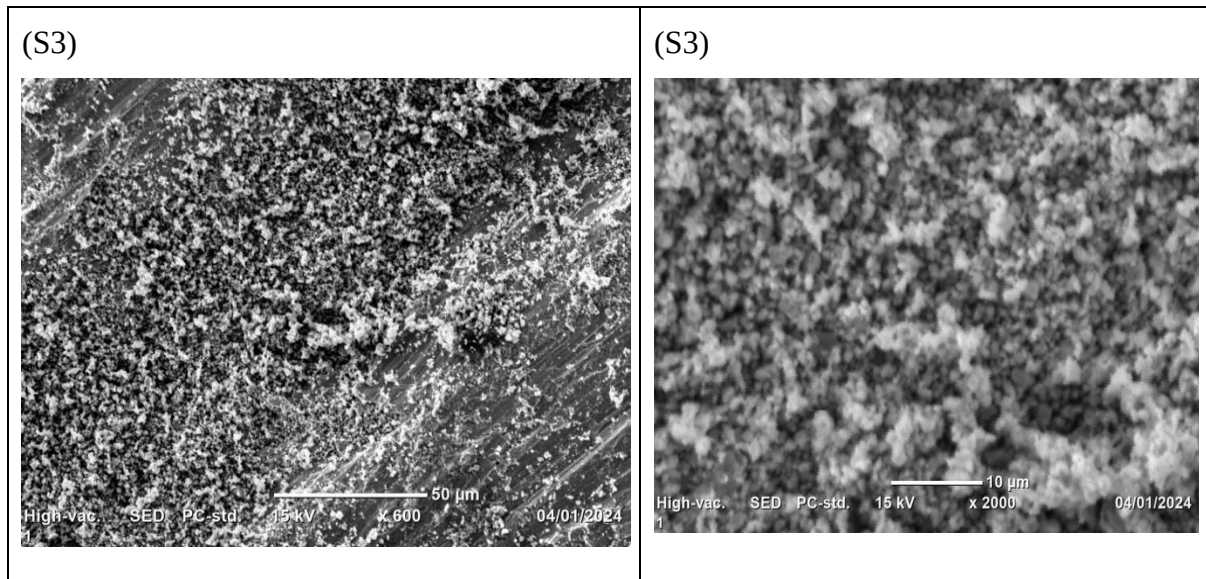


Figure 5.7 SEM image of wear morphology (after wear test)

4.8.Compression test

Specimens were compressed by using UMT it can be used evaluated the deformation of the compressive strength and failure modes of copper composites. Figure 4.4 shows the results of compressive strength to obtained from compression test data of the usual ultimate stresses 81%Cu-10%SiC-5%CeO₂-4%Gr, 81%Cu-7.5%SiC-7.5%CeO₂-4%Gr, and 81%Cu-5SiC-10%CeO₂-4%Gr were 379.366,393.758 and 367515 MPa, respectively. The results below table show that when adding total reinforcement weight percentage of (SiC-CeO₂-Gr) 19% to copper matrix samples better order yield strength than pure copper. When hard ceramic reinforcements of SiC and CeO₂ are added, the compressive strength of the copper composite was increased dramatically in comparison to unreinforced copper.CeO₂ is the one of ceramic reinforcement material can improve the compressive strength of copper matrix composites. Its decrease the copper matrix's ability to deform and shift when under compressive load, increasing the compressive strength overall.CeO₂ and SiC both are ceramics reinforcement effectively to improve compressive strength of copper composite. As well as Gr can improve compressive strength of matrix however, the achievement of a consistent dispersion and robust interfacial bonding inside the copper matrix could be a determining factor in its efficacy.

Table 5.6 Results of compression test

Sample code	Compressive strength (MPa)
S1	379.363
S2	393.758
S3	367.515

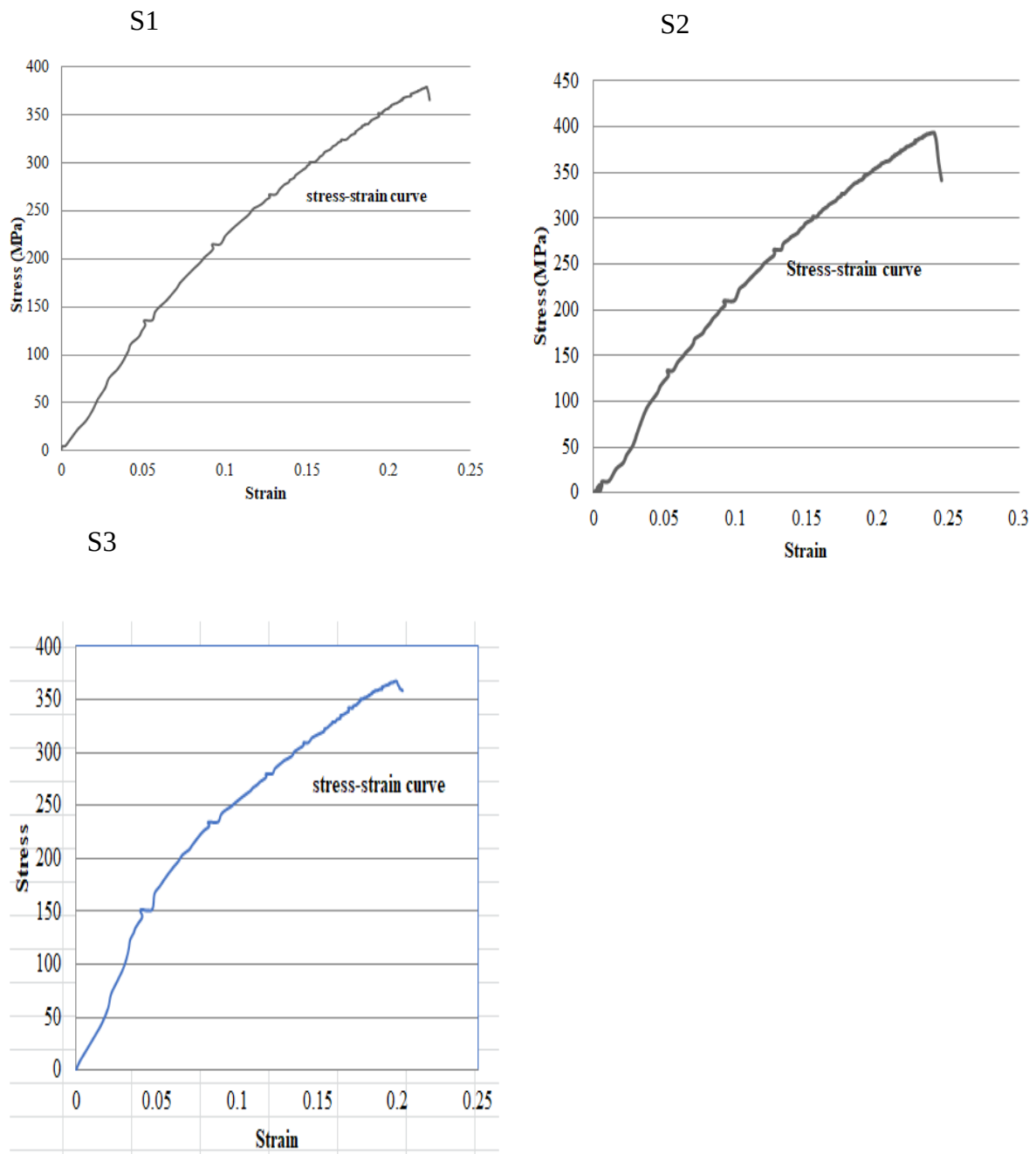
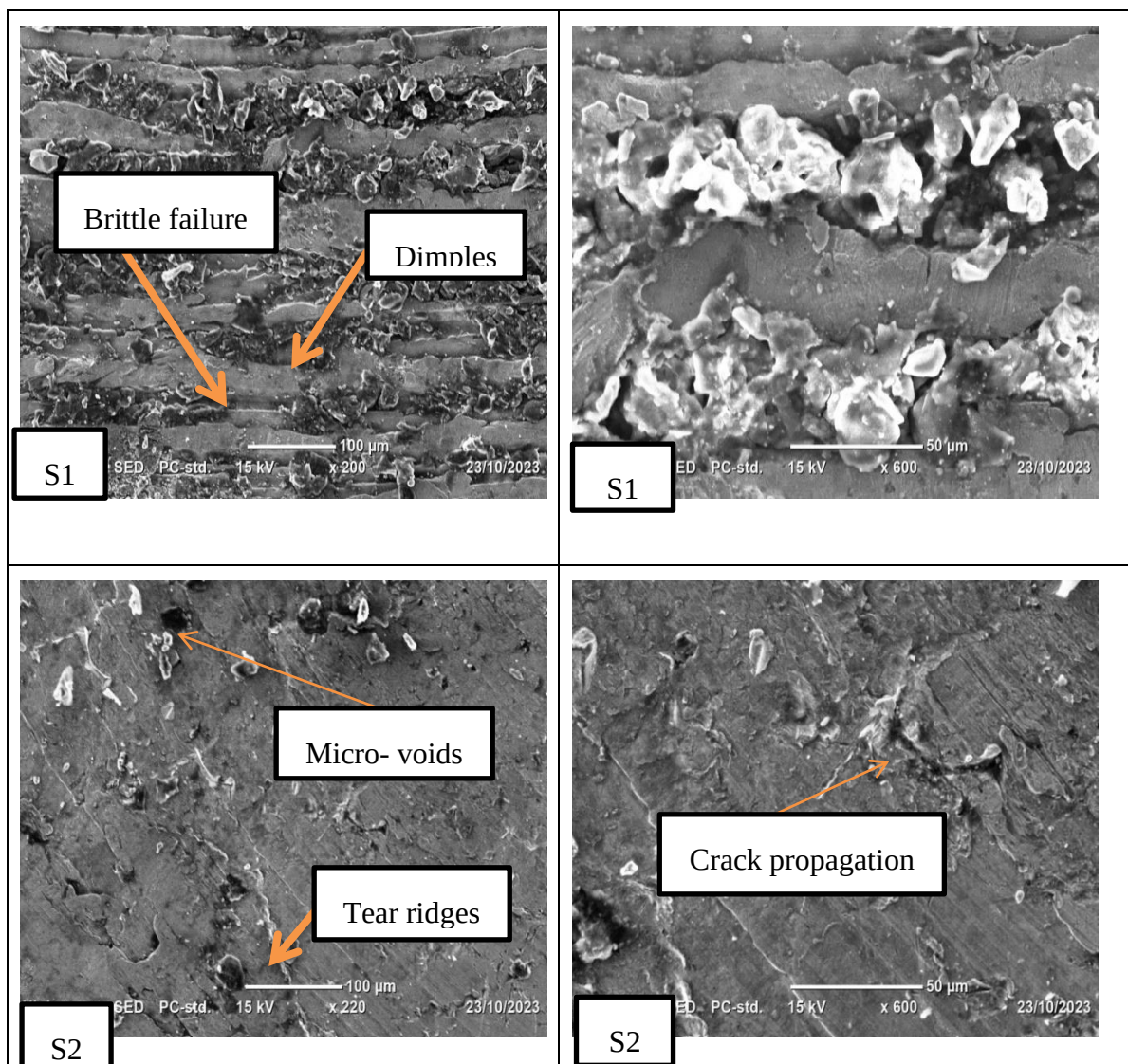


Figure 5.8 Compression result of sample

4.8.1. Fractography analysis after compression test

Fractography analysis by using SEM to provided fracture surface morphology, deformation mechanisms, and damage and reinforcement-matrix interaction of fabricated sample after compression test. In fracture surface morphology to observed and analyzed the fracture surface of the compressed samples in deeply studied fracture like ductile and brittle fracture. Micro cracks, voids, and other damage features that could have appeared during the compression test can be found and examined using SEM. A figure 4.9 was observed under SEM analysis after compression test performed micro-void, crack propagation, dimples and brittle failures.



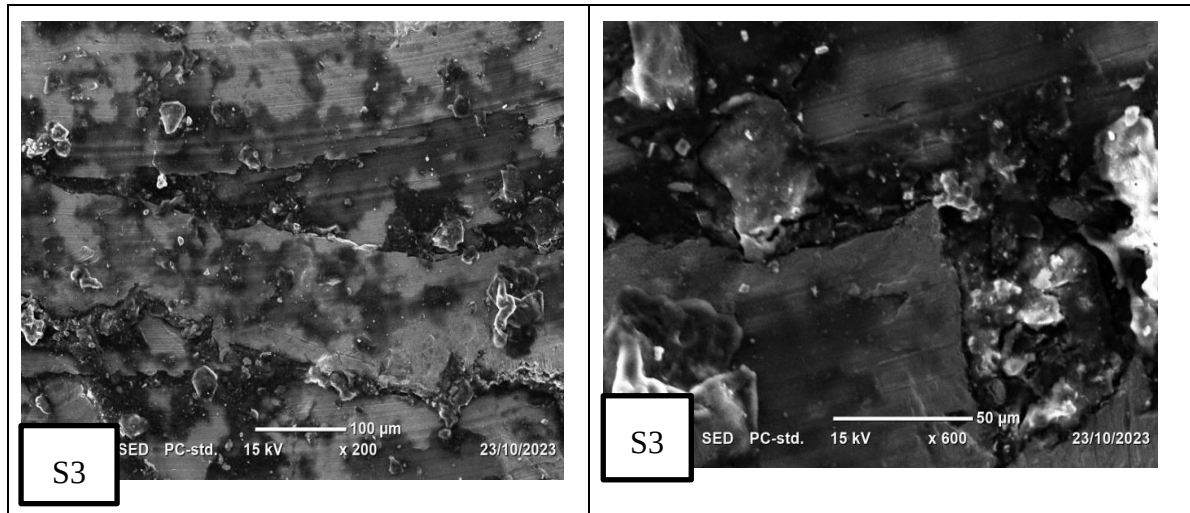
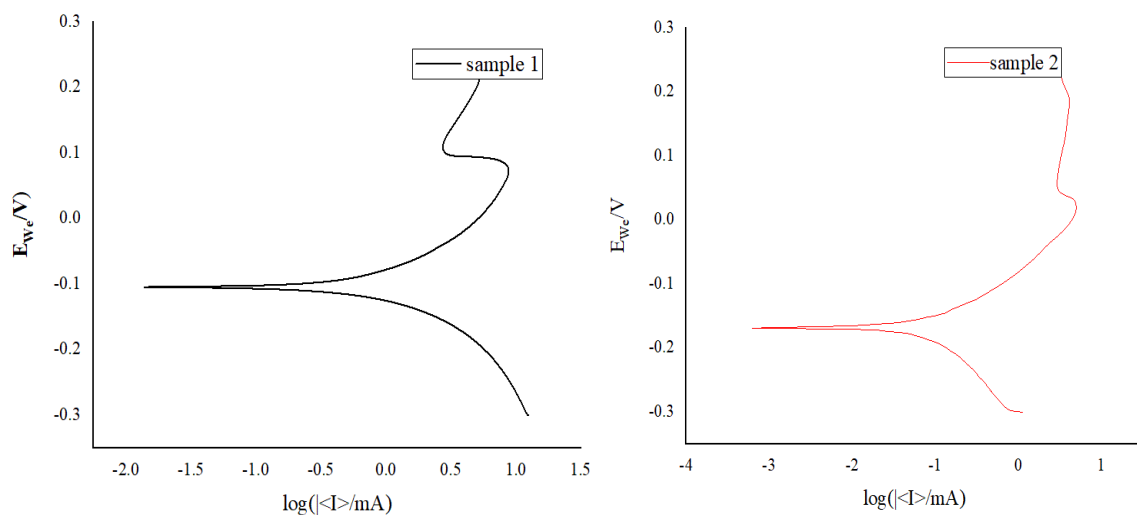


Figure 5.9 SEM image of fractography (after compression test).

4.9. Analysis of corrosion test

Potentiodynamic polarization curves demonstrating the identical polarization and passivity properties of unreinforced copper and copper matrix composites produced in 3.5 w.t % NaCl solution. One electrochemical method for examining how materials corrode is potentiodynamic polarization. The corrosion behaviour is significantly different, but according to the corrosion current densities (I_{corr}) and corrosion potentials (E_{corr}) groove for reinforced particles and the unreinforced composite series and the ceramic and metal reinforced composite series are separated by a void.



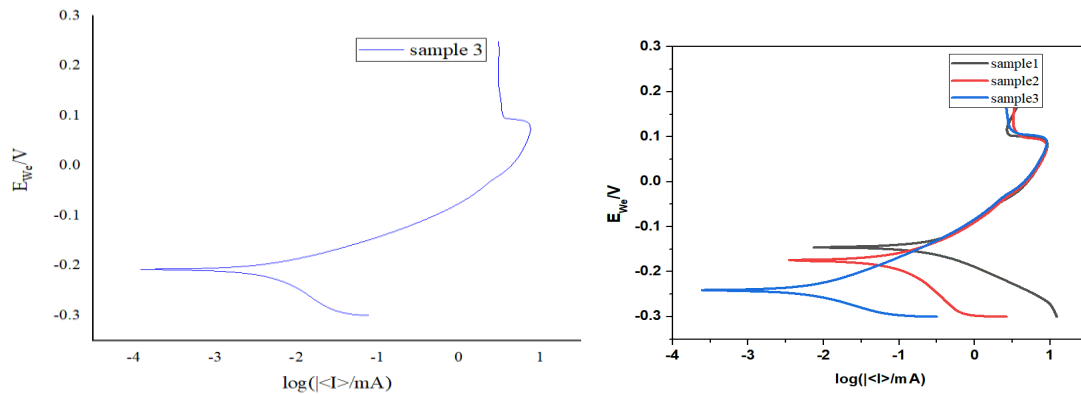


Figure 5.10 Tafel plot of copper matrix composite in corrosion test

The formation of micro-galvanic cells between the two materials can result in galvanic corrosion when silicon carbide is introduced to a copper matrix. as well as a lot of research has been done on cerium oxide (CeO_2), and how it might be used to increase the corrosion resistance of different materials, such as metals and alloys. When compared sample 2 with other sample, sample 2 have 7.5% CeO_2 and 7.5%SiC more added both ratio of percentage of reinforcements in matrix material. as result corrosion rate obtained from table 4.7 81%Cu-7.5%SiC-7.5% CeO_2 -4%Gr was calculated as equation (3.12) has smallest value of corrosion rate and improved corrosion resistance.

Table 5.7 value of electrochemical corrosion in corrosion test

Samp No	$E_{\text{corr}}(\text{V})$	$\beta a(\text{V})$	$\beta c(\text{V})$	$I_{\text{corr}}(\text{mA}/\text{cm}^2)$	CR (mm/year)
S1	-0.104	0.067	-0.054	-0.383	0.0088
S2	-0.207	0.038	-0.068	-0.072	0.0014
S3	-0.168	0.056	-0.072	-1.32	0.0366

4.10. Process of sample selection for hot extrusion process

Sample was selected used by grey relational analysis for select best sample was fabricated via stir casting. The Interrelationship between the values of responses to solved with grey analysis. Used to improve experimental input by signal- to- noise that is significant for the experimental data and to show the desired signal as being greater than the background noise. Selected material for hot extrusion process selected the results of hardness, corrosion rate, wear rate and compressive strength are used as responses to analyzed with experimental results converted to S/N ratio. S/N ratio used for select material based on

property smaller- the- better, higher-the-better and nominal-the-better (Xu et al., 2023). Depend on the ultimate goal for application area to increased hardness and compressive strength by used S/N ratio smaller-the-better and decreasing wear rate and corrosion rate by used higher-the-better. At first experimental data converted in to S/N ratio.

Table 5.8 Input of experimental data and S/N ratio for response

Experimental results					S/N ratio			
Sample	Hardness	Compressive strength	Corrosion rate	Wear rate	Hardness	Compressive strength	Corrosion rate	Wear rate
S1	97.533	379.36	0.0088	0.446	19.02	16.64	21.64	10.51
S2	100.03	393.758	0.0014	0.256	19.28	17.04	28.10	15.09
S3	87.166	367.515	0.0366	0.626	17.80	16.22	4.75	2.15

Second to normalized data on table 4.9

Table 5.9 Normalized data of multi-responses

Sample	Hardness	Compressive strength	Corrosion rate	Wear rate
S1	0.667	0.444	0.588	0.588
S2	0.778	0.556	1	1
S3	0	0.33	0	0

At third step done sequence of deviation the normalized data

Table 5.10 Sequence of deviation the normalized data

Sample	Hardness	Compressive strength	Corrosion rate	Wear rate
S1	0.333	0.556	0.412	0.412
S2	0.222	0.444	0	0
S3	1	0.667	1	1

Fourth calculated grey relational coefficient (GRC) and grey relational grade (GRG) and ordered sample by rank.

Table 5.11 Grey relational coefficient and grey relational grade

Sample	Hardness	Compressive strength	Corrosion rate	Wear rate	Grey Relational Grade	Rank
S1	0.	0.472	0.548	0.548	0.542	2
S2	0.692	0.53	1	1	0.806	1
S3	0.333	0.429	0.333	0.333	0.357	3

4.11. Numerical simulation of developed composite for deformation behaviors analysis through hot extrusion process

Copper matrix composite deformed through hot extrusion process to improved mechanical properties for widely application area. For the hot extrusion process the parameters which have significant effect on responses of extrusion process was selected to identified optimum value by 3D deform software for simulation process. To analyzed and optimized with selected parameters of extrusion process (extrusion ration, ram speed and temperature) combined by BBD method and the responses result (damage, load and effective stress) obtained from 3D deform of simulation by Design-Expert software. The values of responses are combined by BBD based response surface methodology was analyzed using ANOVA. Design Expert 13 was used for data analyzed and obtained regression model equations from Design Expert development in Analysis of Variance, and MATLAB software was used for multi-objective optimization based on regression mode equations. Data collected from 3D deform and those data (combined parameters of extrusion process and responses values) helped for significant of analysis in the statistical analysis decided to the best value and optimum .Data collected from simulation process as table 4:10.

Table 5.12 Parameters of extrusion process and responses values obtained from simulation

Std	Run	Extrusion ratio	Ram speed(mm/s)	Temperature(°C)	Damage	Load (KN)	Stress (MPa)
5	1	2	2	850	0.312	295	1302.59
8	2	4	2	950	1.05	2365	3693.41
1	3	2	1	900	0.487	992	1145
7	4	2	2	950	0.488	1147	1476
15	5	3	2	900	0.375	768	1014
12	6	3	3	950	0.52	1401.78	1304.8
13	7	3	2	900	0.374	845	1052
2	8	4	1	900	1.27	2452	3684
3	9	2	3	900	0.301	215	985
4	10	4	3	900	0.95	1894	2854
11	11	3	1	950	0.92	1897	2355
14	12	3	2	900	0.489	768	1014
10	13	3	3	850	0.375	491	1616
6	14	4	2	850	1.07	2345	3450
9	15	3	1	850	0.518	1354.4	1451.98

4.12. Optimization of effective stress, load and damage through RSM

4.12.1. Load analysis and discussion

Fit summary is the program to helped start regression and calculated to fit all the polynomial models with selected response like p-values for checked model significant or not significant, lack of fit, R-squared values for check and compared the models. Model to suggest linear, cubic, or quadratic, which is best for model fit. Table 4.13 the model term was quadratic suggested and the highest order model that significant in the response (p-value < 0.05), insignificant lack of fit (p-value > 0.10) and reasonable agreement between Adjusted R-squared and Predicted R-squared (within 0.2 of each other). Lack of fit or p-value > 0.05 to indicated the model good fit to the data. R-squared measured to the variation in the response, adjusted R-squared is preferred for model comparison and the value of R-squared and adjusted R-squared was indicated the model to better fit when closer to 1.

Table 5.13 fit summary of load analysis

Source	S-p-value	LF p-value	R-Sq(adj)	R-Sq(pred)	
Linear	0.0001	0.0137	0.7949	0.7441	
2FI	0.6651	0.0110	0.7658	0.6628	
Quadratic	0.0009	0.1201	0.9831	0.9101	Suggested
Cubic	0.1201		0.9965		Aliased

4.12.2. Analysis of Variance for Load

Table 4.14 it was observed that Models F-value of 91.41 suggests that the model is significant. Such a type of large F-value has 0.10% chances of being caused to noise. The model has P-value less than 0.05 which indicates that the model terms are significant. Given the pure error, the F-value for the lack of fit, which is 7.49, suggests that the lack of fit is not significant. A significant Lack of Fit F-value has a 40.85% probability of being caused by noise. As result of table 4:15 there is less than 0.2 variations between the R-Sq(adj) of 0.9101 and the R-Sq(pred) of 0.9831, indicating that there is good agreement. The ratio of 28.335 indicates an adequate signal.

Table 5.14 ANOVA of load response

Source	SS	Df	M S	F-value	p-value		% C
Model	7.956E+06	9	8.840E+05	91.41	< 0.0001	Significant	99.4
A	5.131E+06	1	5.131E+06	530.60	< 0.0001		64.1
B	9.069E+05	1	9.069E+05	93.78	0.0002		11.3
C	6.759E+05	1	6.759E+05	69.89	0.0004		8.44
AB	11990.25	1	11990.25	1.24	0.3161		0.14
AC	1.731E+05	1	1.731E+05	17.90	0.0082		2.16
BC	33889.13	1	33889.13	3.50	0.1201		0.42
A ²	6.615E+05	1	6.615E+05	68.40	0.0004		8.26
B ²	1.084E+05	1	1.084E+05	11.21	0.0204		1.35
C ²	3.806E+05	1	3.806E+05	39.36	0.0015		4.75
Residual	48352.87	5	9670.57				0.6
LF	44400.20	3	14800.07	7.49	0.1201	NS	
P E	3952.67	2	1976.33				
CT	8.004E+06	14					

Where A=extrusion ratio, B=ram speed, C= temperature, LF=lack of fit, NS=not significant, PE=pure error and CT=corrected total.

Table 5.15 Fit Statistics of load

Std. Dev.	Mean	C.V. %	R-Sq	R-Sq(adj)	R-Sq(pred)	Adeq Precision
98.34	1282.01	7.67	0.9940	0.9831	0.9831	28.3355

$$Z \text{ load} = 94,761.9 + 1,895.76X_1 - 2,843.02X_2 - 216.555X_3 + 54.75X_1X_2 + -4.16X_1X_3 + 1.8409X_2X_3 + 423.269X_1^2 + 171.314X_2^2 + 0.128426X_3^2$$

Where, X_1 = Extrusion ratio, X_2 = Ram speed and X_3 = temperature

4.12.3. Parametric Influence on load

Figure 4:11 a and b showed that the regression analysis of normal % probability versus external studentized and a predicted versus actual graph respectively.

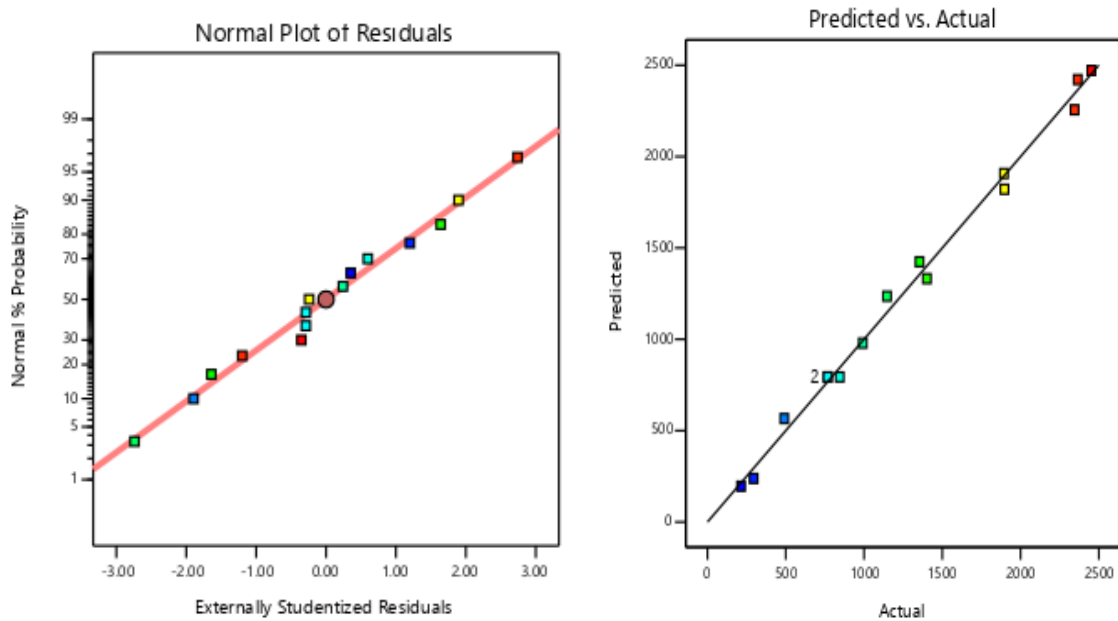


Figure 5.11 a) graph of Normal probability plot versus external studentized residuals, b) graph of predicted versus actual of load analysis

4.12.4. Effect of extrusion ratio on load

Based on the value of percentage contribution was distributed, the factors were large value of percentage contribution (> 20%) to considered the most important in the optimization. Largest value of extrusion ratio 64.105% as calculated from ANOVA table 4.14 and the result percentage contribution of temperature was calculated 11.33%, with ram speed had

the lowest percentage contribution 8.44%. Closely relate the value of ram speed and temperature percentage contribution and small value and did not effect on load. But extrusions ration effect on load response. The ANOVA with p values of extrusion ratio ($P < 0.001$) and R-square value is 99.4% as result of table 4.15 with extrusion ratio is significant factors effect on load. Percentage contribution of residual or error is ($< 20\%$) to indicate there are missing factors but, reducing residual percentage contribution improving experimental methods. Under this condition the percentage contribution is 0.6% as calculated on table 4.15, this result indicated improved experimental result and model more fit. Extrusion ratio has effected on load, when extrusion increased load applied on billet to increase. Below figure 4:12 and 4:13 show those the interaction effects of extrusion ratio and temperature on load and extrusion ratio effect on load at different extrusion ratio respectively.

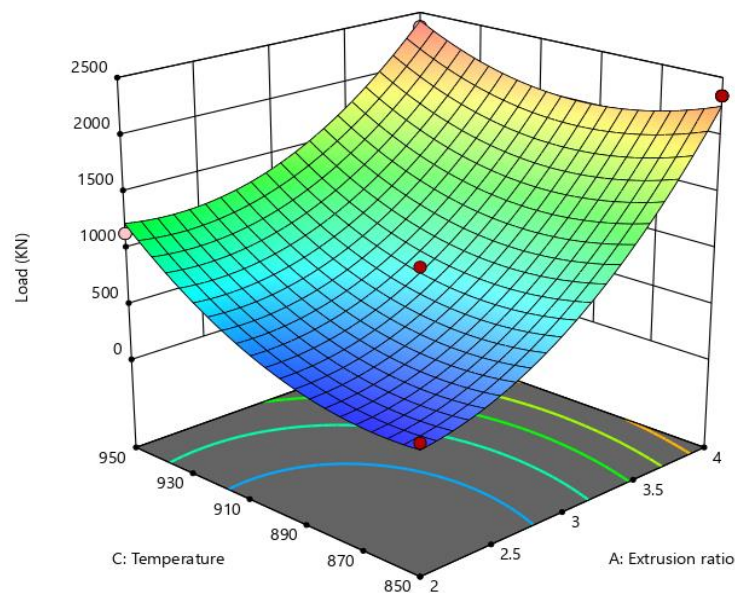


Figure 5.12 interaction effects of extrusion ratio and temperature on load

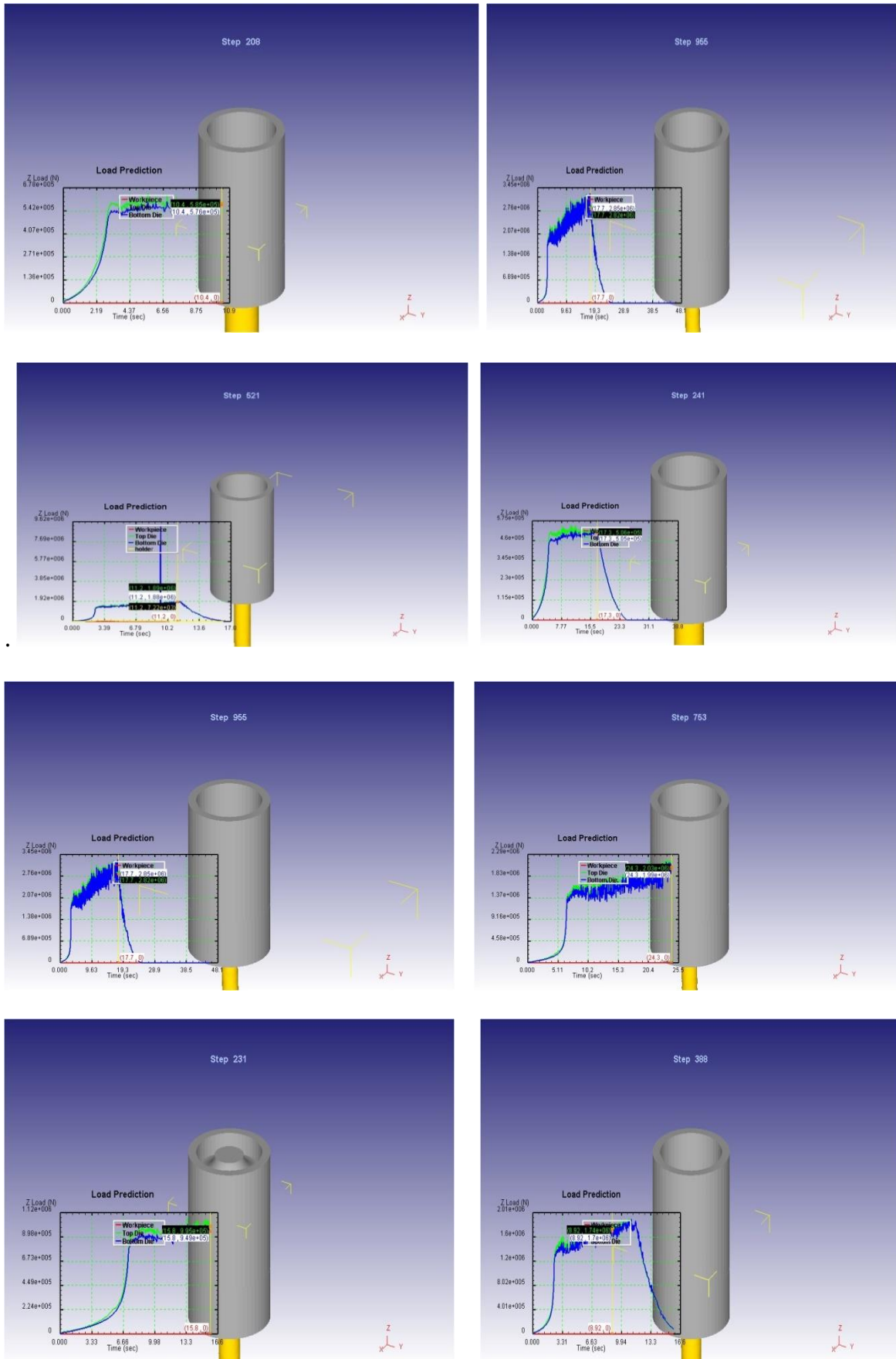


Figure 5.13 Distribution of Load

4.13. Effective stress analysis and discussion

Table 4.16 the fit summary indicated model term was quadratic suggested and the highest order model that significant in the response ($p\text{-value} < 0.05$), insignificant lack of fit ($p\text{-value} > 0.10$) and reasonable agreement between Adjusted R-squared and Predicted R-squared (within 0.2 of each other).

Table 5.16 fit summary of stress analysis

Source	S- p-value	LF p-value	R-Sq (adj)	R-Sq (pred)	
Linear	0.0039	0.0009	0.6041	0.4853	
2FI	0.8163	0.0007	0.5127	0.1539	
Quadratic	< 0.0001	0.2179	0.9988	0.9940	Suggested
Cubic	0.2179		0.9995		Aliased

4.13.1. Analysis of Variance for effective stress

ANOVA results were used to evaluate which important parameters affect effective stress in a statistically significant way, as well as the way they interact. Table 4.17 it was observed that Models F-value of 1288.81 suggested that the model is significant. F-value for the lack of fit, which is 3.75, suggests that the lack of fit is not significant. A not significant Lack of Fit F-value has a 21.79% probability of being caused by noise. As result of table 4.18 there (< 0.2) variations between the R-sq (adj) of 0.9940 and the R-sq(pred) of 0.9988, indicating that there is good agreement. The ratio of 93.231 was indicated an adequate signal. The total of squares for each individual source was divided by the sum of squares for the corrected total to determine the percentage of contribution in this ANOVA

Table 5.17 ANOVA of effective stress response

Source	SS	Df	M S	F-value	p-value		%C
Model	1.478E+07	9	1.642E+06	1288.81	< 0.0001	Significant	99.93
A	9.620E+06	1	9.620E+06	7549.85	< 0.0001		65.04
B	4.400E+05	1	4.400E+05	345.31	< 0.0001		2.97
C	1.272E+05	1	1.272E+05	99.80	0.0002		0.86
AB	1.122E+05	1	1.122E+05	88.07	0.0002		0.75
AC	1225.00	1	1225.00	0.9614	0.3719		0.008
BC	3.686E+05	1	3.686E+05	289.26	< 0.0001		24.92
A ²	3.470E+06	1	3.470E+06	2723.29	< 0.0001		23.46
B ²	1.078E+05	1	1.078E+05	84.62	0.0003		0.72
C ²	8.663E+05	1	8.663E+05	679.89	< 0.0001		5.85
Residual	6371.18	5	1274.24				0.04
LF	5408.51	3	1802.84	3.75	0.2179	NT	
PE	962.67	2	481.33				
CT	1.479E+07	14					

Table 5.18 Fit Statistics of effective stress

Std. Dev.	Mean	C.V. %	R-Sq	R-Sq(adj)	R-Sq(pred)	Adeq Precision
35.70	1893.19	1.89	0.9996	0.9988	0.9940	93.2308

effective Stress = 151,299 + -4,700.06X₁ + 5,048.41X₂ - 335.146X₃ - 167.5X₁X₂ + 0.35X₁X₃ - 6.0711X₂X₃ + 969.444X₁² + 170.889X₂² + 0.193756X₃²

4.13.2. Effect of extrusion ratio on effective stress

Determine the percentage that every important parameter and relationship contributes to the overall variation in the effective stress. Largest value of extrusion ratio 65.04% as calculated from ANOVA table 4.17 and the of result percentage contribution ram speed of was calculated 2.97%, with temperature had the lowest percentage contribution 0.86%. Closely relate the value of ram speed and temperature percentage contribution and small value and did not effect on stress. But extrusions ration effect on stress response. The ANOVA with p values of extrusion ratio (P< 0.001) and R-square value is 99.96% as result of table 4.18 with extrusion ratio is significant factors effect on stress. . Under this

condition the percentage contribution residual is 0.04% as calculated on table 4.17, this result indicated improved experimental result and model more fit. Below figure 4:14 and 4:15 show those the interaction effects of extrusion ratio and ram speed on effective stress and extrusion ratio effect on effective stress at different extrusion ratio respectively

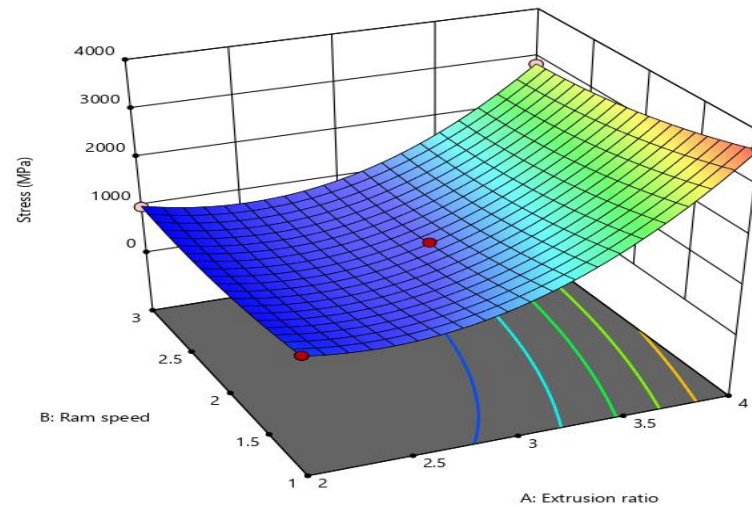


Figure 5.14 interaction effects of extrusion ratio and ram speed on effective stress

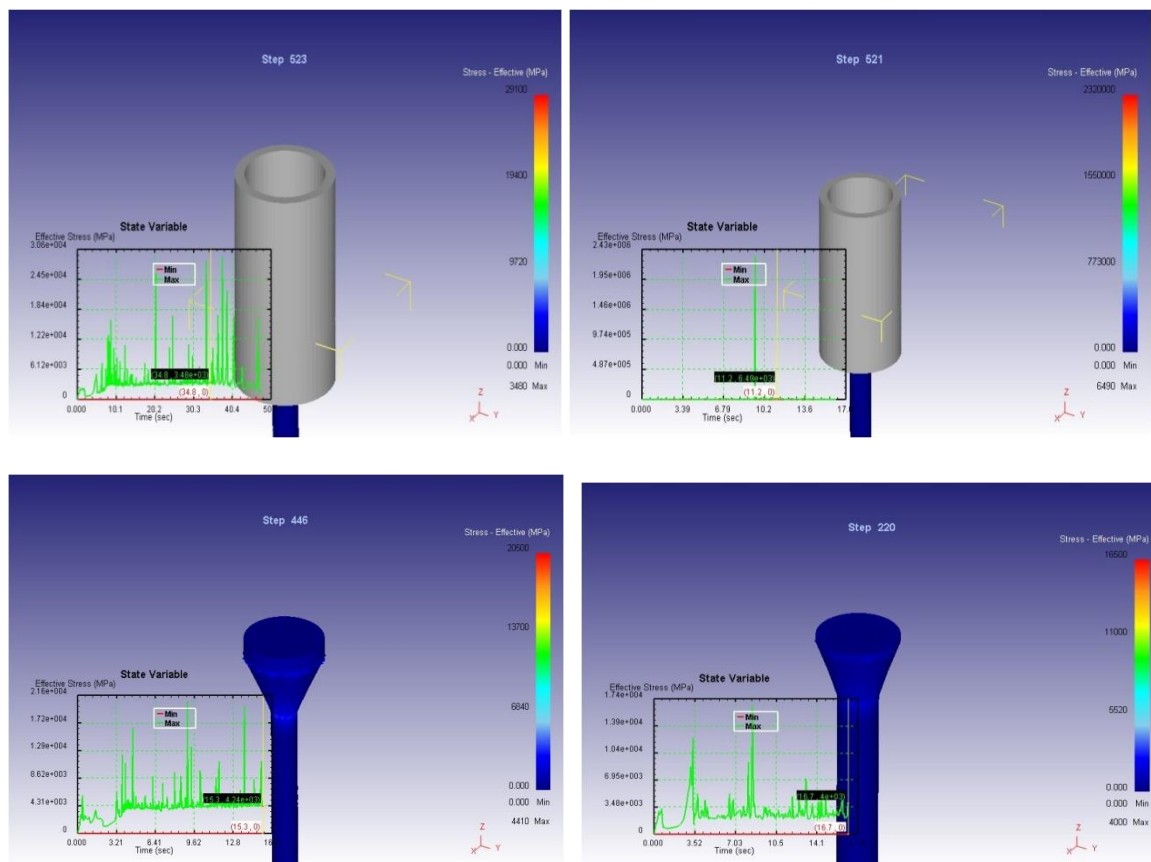


Figure 5.15 Distribution of Effective Stresses

4.13.3. Parametric Influence on effective stress

Figure 4.16 a and b showed that the regression analysis of normal % probability versus external studentized and a predicted versus actual graph of stress respectively.

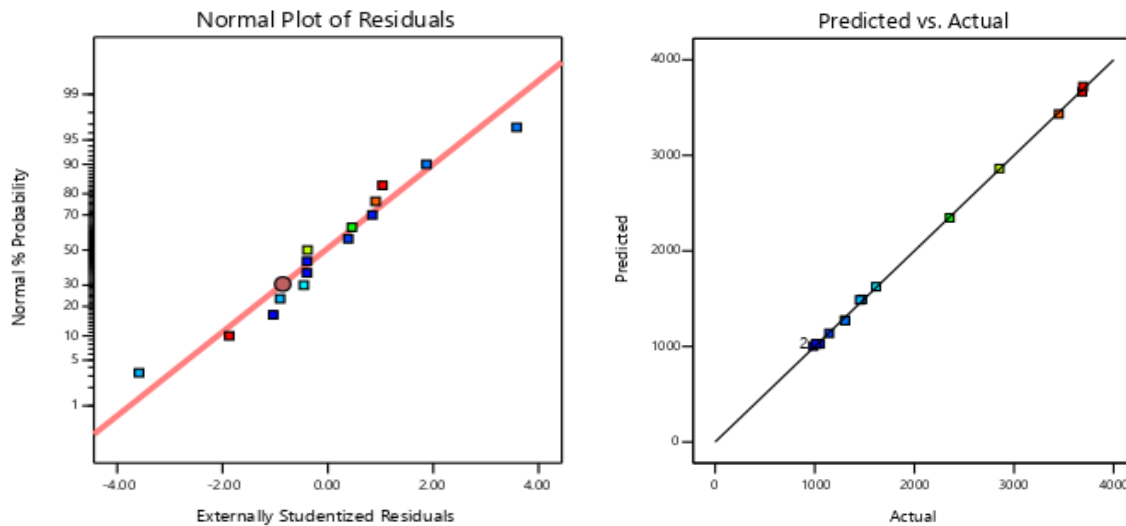


Figure 5.16 a) graph of Normal probability plot versus external standardized residuals, b) graph of predicted versus actual of stress analysis

4.14. Damage analysis and discussion

Program calculated effects for model terms and produced statistics value of p-value, lack of fit and R-squared. Table 4.19 suggested to the model term was quadratic model and the highest order model that explains significantly more of the variation in the response (p-value small), insignificant lack of fit (p-value > 0.10) and reasonable agreement between Adjusted R-squared and Predicted R-squared (within 0.2 of each other).

Table 5.19 fit summary of damage analysis

Source	S- p-value	LF p-value	R-Sq (adj)	R-Sq (pred)	
Linear	0.0005	0.1208	0.7290	0.6496	
2FI	0.8301	0.0909	0.6642	0.4220	
Quadratic	0.0071	0.4085	0.9431	0.7574	Suggested
Cubic	0.4085		0.9580		Aliased

4.14.1. Analysis of Variance for Damage

From ANOVA the damage in Table 4.20 it was observed that Models F-value of 26.78 suggests that the model is significant. Such a type of large F-value has 0.10% chances of being caused to noise. The model has P-value less than 0.05 which indicates that the model

terms are significant. In this case A, B, C, A² are significant model terms. Given the pure error, the F-value for the lack of fit, which is 1.59, suggests that the lack of fit is not significant. A significant Lack of Fit F-value has a 40.85% probability of being caused by noise. Non-significant mismatch is good. As result of table 4.21 was less than 0.2 variations between the Adjusted R² of 0.9431 and the Predicted R² of 0.7574, indicating that there is good agreement. Adeq Precision calculates the ratio of signal to noise. A ratio greater than 4 is desirable. This ratio of 16.104 indicates an adequate signal.

Table 5.20 ANOVA of Damage response

Source	SS	Df	M S	F-value	p-value		%C
Model	1.43	9	0.1585	26.78	0.0010	Significant	97.94
A	0.9467	1	0.9467	159.97	< 0.0001		64.84
B	0.1376	1	0.1376	23.24	0.0048		9.42
C	0.0618	1	0.0618	10.44	0.0232		4.23
AB	0.0045	1	0.0045	0.7585	0.4236		0.308
AC	0.0096	1	0.0096	1.62	0.2587		0.657
BC	0.0165	1	0.0165	2.79	0.1557		1.13
A ²	0.2181	1	0.2181	36.85	0.0018		14.93
B ²	0.0342	1	0.0342	5.78	0.0612		2.34
C ²	0.0204	1	0.0204	3.44	0.1227		1.39
Residual	0.0296	5	0.0059				2.02
LF	0.0208	3	0.0069	1.59	0.4085	NT	
PE	0.0087	2	0.0044				
CT	1.46	14					

The total of squares for each individual source was divided by the sum of squares for the corrected total to determine the percentage of contribution in this ANOVA.

Table 5.21 Fit Statistics of damage

Std. Dev.	Mean	C.V. %	R-Sq	R-Sq(adj)	R-Sq(pred)	Adeq Precision
0.0769	0.6333	12.15	0.9797	0.9431	0.7574	16.1037

Modeling and examining the relationship between a dependent variable and one or more independent variables is commonly done through the statistical method is known as regression analysis.

$$\text{Damage} = 19.5442 - 0.16525X_1 + 0.740708X_2 - 0.046222X_3 - 0.0335X_1X_2 - 0.00098X_1X_3 - 0.001285X_2X_3 + 0.243042X_1^2 + 0.0962917X_2^2 + 2.97167e-05X_3^2$$

Where, X_1 = Extrusion ratio, X_2 = Ram speed and X_3 = temperature

4.14.2. Parametric Influence on Damage

Normal probability plot versus external studentized residuals graph on figure 4:7a indicated that the follow of residuals were normal distribute, thus follow straight line. Figure 4.17a and b showed that the regression analysis also employs a predicted versus actual graph as a critical diagnostic plot to evaluate how well the regression model fits the data.

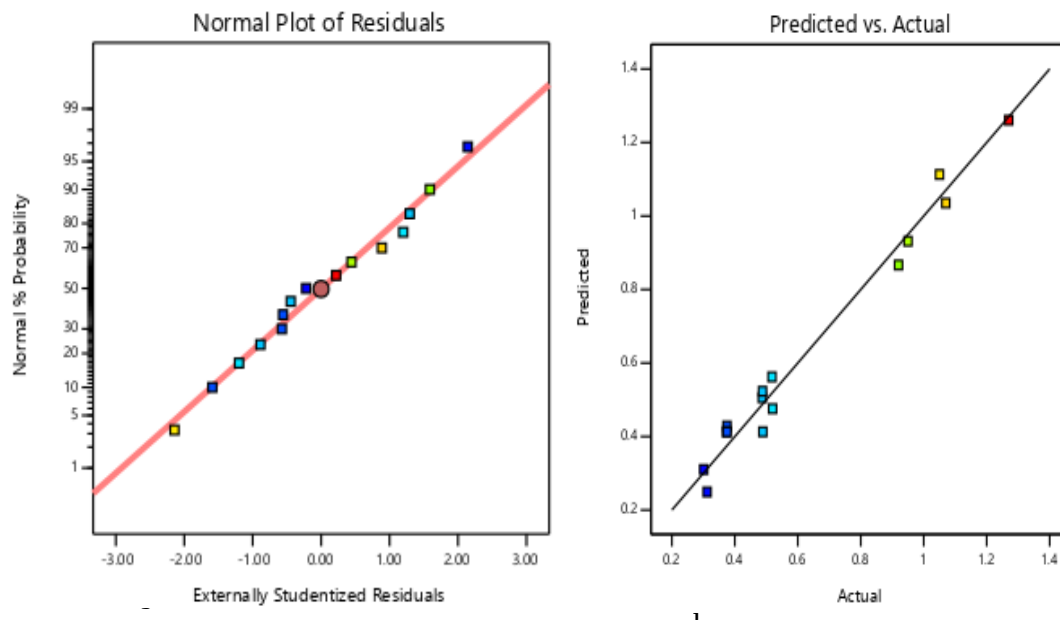


Figure 5.17 a) graph of Normal probability plot versus external standardized residuals, b) graph of predicted versus actual

4.14.3. Effect of extrusion ratio on damage

As a result, the percentage contribution for each parameter to the damage percentage during the extrusion of the copper matrix composite was evaluated. Largest value of extrusion ratio 64.84% as calculated from ANOVA table 4.20. The of result percentage contribution of temperature was calculated from ANOVA 9.42%, while ram speed had the lowest percentage contribution of ram speed 4.23%. Closely relate the value of ram speed and temperature percentage contribution and small value and did not effect on damage. But extrusions ration effect on damage response. The ANOVA with p values of extrusion ratio ($P < 0.001$) and R-square value is 97.97% with extrusion ratio is significant factors effect on damage. . Under this condition the percentage contribution of residual is 2.02% as calculated on table 4.20, this result indicated improved experimental result and model more fit. Below figure 4.18 and 4.19 shows those interaction effects of extrusion ratio and ram speed on damage and extrusion ratio effect on damage at different extrusion ratio respectively.

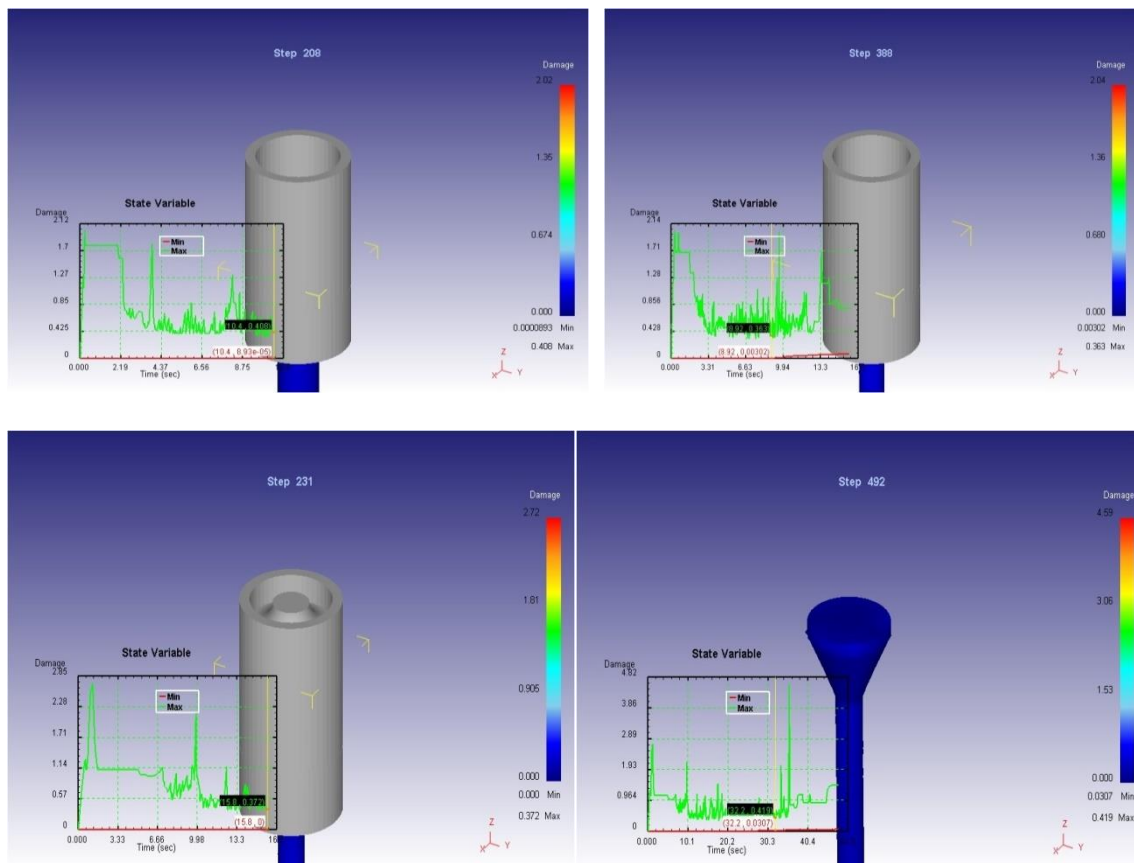


Figure 5.18 Damage value distribution

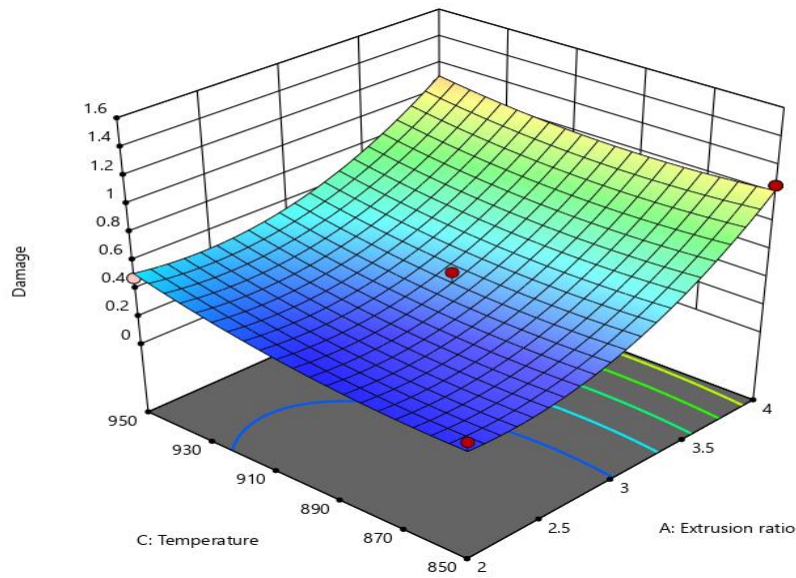


Figure 5.19 the interaction effects of extrusion ratio and temperature on damage.

Table 5.22 Constraints for optimization of extrusion process parameters and responses

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A	is in range	2	4	1	1	3
B	is in range	1	3	1	1	3
C	is in range	850	950	1	1	3
Damage	minimize	0.301	1.27	1	1	3
Load	minimize	215	2452	1	1	3
Stress	minimize	985	3693.41	1	1	3

The extrusion process is important for deform materials and improved properties of materials to selected parameters (extrusion ratio, ram speed and temperature) and responses (load, damage and effective stress) must be optimized by taking into consideration a number of significant constraints. To obtain the desired product quality and performance, these constraints must be carefully managed when optimizing the extrusion process. Damage =0.301 up to 1.27, Load = 215N up to 2452N and effective stress = 985MPa up to 3693.41MPa

Table 5.23 Solution of optimized extrusion parameters for extrusion process

No	Extrusion ratio	Ram speed	Temperature	Damage	Load	Stress	Desirability	
1	2.485	2.884	875.186	0.265	146.194	915.964	1.000	Selected
2	2.066	2.705	887.498	0.263	109.240	934.639	1.000	
3	2.141	2.437	889.630	0.252	191.139	818.535	1.000	
4	2.455	2.850	874.595	0.260	132.732	915.668	1.000	
5	2.242	2.363	866.488	0.231	136.404	951.739	1.000	
6	2.177	2.188	868.195	0.234	180.899	929.621	1.000	
7	2.222	2.778	893.333	0.260	171.286	807.601	1.000	
8	2.457	2.425	864.079	0.243	206.271	945.901	1.000	
9	2.438	2.720	868.548	0.251	130.321	960.270	1.000	
10	2.389	2.692	878.587	0.245	140.895	838.271	1.000	

4.15. Multi objective optimization of extrusion parameters by GA

Regression equation obtained from Design-Expert was converted into MATLAB for multi-objective optimization process. MATLAB function, the function was the input of toolbox used as the objective function. Objective function to choice based on optimized parameters which the parameters minimization and maximization with upper and lower bounds were specified. Pareto optimal solution involved to multi-objective optimization algorithm technique is used to search optimal value.

Table 5.24 Pareto Optimal Solution results obtained from MATLAB

Run	Damage	Load	Effect	Extrusion	Ram speed	Temperature
			stress	ratio		
1	-0.88532	370.0421	687.1013	2.473168	2.449163	901.1822
2	-1.46428	96.82511	918.3052	2.35022	2.999861	880.3321
3	-1.43031	-74.4912	1522.525	2.0002	2.999964	857.3424
4	-1.21523	230.0303	743.7695	2.459728	2.766704	891.0907
5	-1.42339	500.558	730.177	2.527468	2.998138	915.6687
6	-1.0059	329.0804	691.8922	2.438093	2.568703	900.5964
7	-0.75389	275.619	720.1618	2.372547	2.282311	887.6426
8	-1.19858	309.2656	714.739	2.376823	2.761372	903.2756
9	-1.33422	436.4493	715.882	2.481277	2.907438	912.0423
10	-0.92314	174.3233	787.5692	2.263801	2.458072	884.0725
11	-1.32588	47.65457	1000.441	2.108266	2.8761	881.5118
12	-1.35252	-32.1904	1253.38	2.086168	2.906151	865.0332
13	-1.39698	-58.2264	1447.354	2.052605	2.960256	857.5202
14	-1.14091	421.4603	708.2184	2.423794	2.720869	911.3934
15	-1.33081	196.4318	800.3795	2.484724	2.878286	886.0733
16	-1.43316	16.05761	1128.446	2.023895	2.990397	880.2959
17	-1.40336	-54.0035	1413.742	2.069708	2.9638	858.5257
18	-1.44225	17.11734	1061.02	2.176009	2.983495	875.4251

Pareto optimal used to finding a collection of Pareto-optimal solutions to a multi-objective optimization problem is the aim; in this type of problem, no objective can be enhanced without losing at least one other target.

Table 5.25 Pareto optimal value result obtained from GA

Parameters	Value of parameter	Value of responses		
		Extrusion ratio	Ram speed	Temperature
Damage	0.753	2.37	2.28	875.64
Load	17.11	2.176	2.98	875.48
Effective stress	687.101	2.437	2.449	901.18

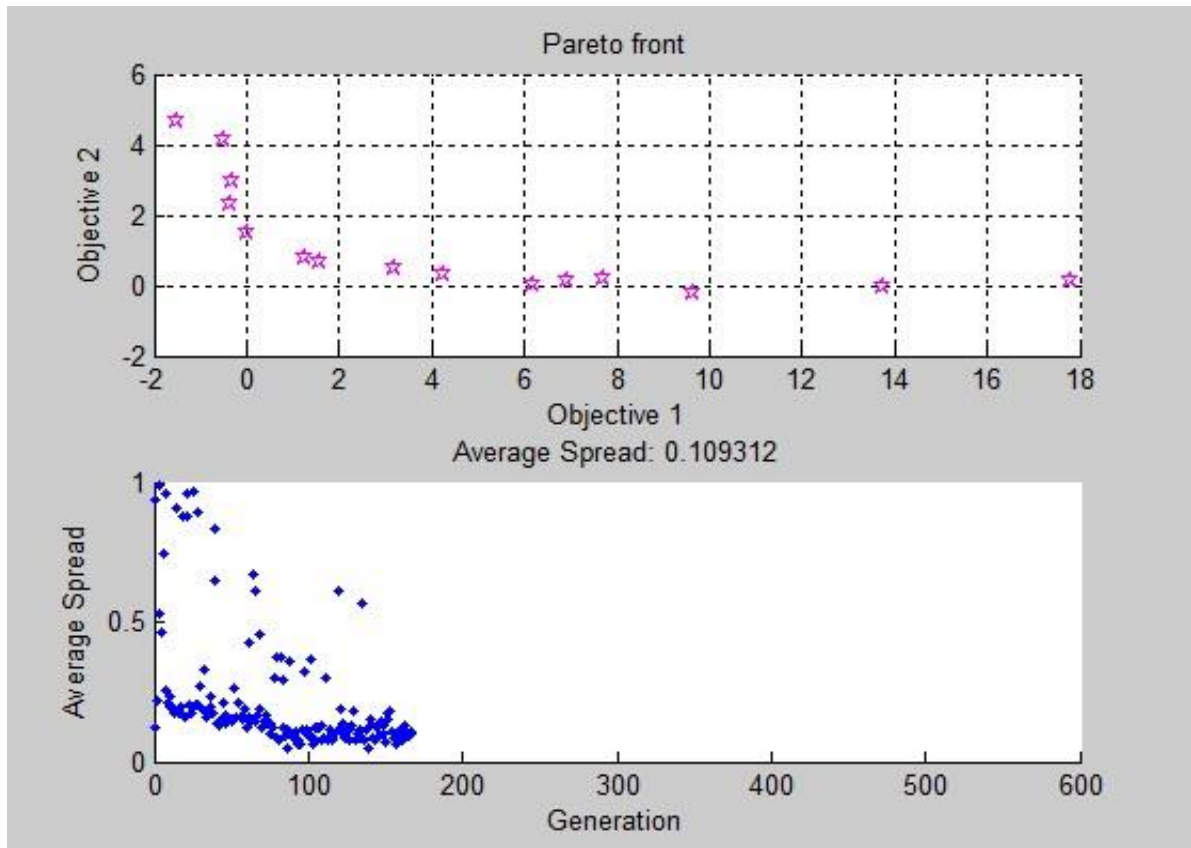


Figure 5.20 Pareto spread of average and parent optimal front

4.16. Confirmation experiments

To combined to taken optimal value selected as goal of target as based on minimized responses of load, effective stress to obtained from table 4.23 and results obtained from pareto optimal solution on table 4.25 compared with confirmation experiment of simulated in Design-Expert software selected optimal value was taken better value for secondary operation (hot extrusion process). So closely related both optimal value in Design- Expert software and GA confirmation test was accepted. After decided those combined optimal values was done hot extrusion values and was checked mechanical properties after extrusion process and selected optimal value.



Figure 5.21 extruded samples

4.17. Result test of the secondary operation

4.17.1. Density analysis after hot extrusion process

Density was calculated with porosity of 81% Cu-7.5SiC-7.5CeO₂-4%Gr copper composite. when compared the result of actual density copper matrix composite fabricated through stir casting and extruded composite through hot extrusion process as shown in table 4.15 increased from 8.0932g/cm³ to 8.109g/cm³ which shows Significant improvement in porosity percentage from 0.26% to 0.07%. Decreasing value of porosity percentage helps to improve mechanical and physical property of composite. Heated to a temperature higher than its recrystallization temperature, the composite material is pushed through a die shaped as desired while under intense pressure during the hot extrusion process.

Table 5.26 results of theoretical density, actual density and porosity after extrusion

Sample	Theoretical density	Actual density	Porosity (%)
S2	8.115	8.109	0.073

4.17.2. OM test after hot extrusion process

Tested and compared the sample (81% Cu-7.5SiC-7.5CeO₂-4%Gr) the reinforcements particles distributed in matrix material uniformly in copper matrix via stir casting was discussed and observed after extrusion process by using optical microscopy. After

deformed some presence change of structure and grain size, thus effect was improved mechanical property of 81% Cu-7.5SiC-7.5CeO₂-4%Gr and grain size was 36.5 μ m.

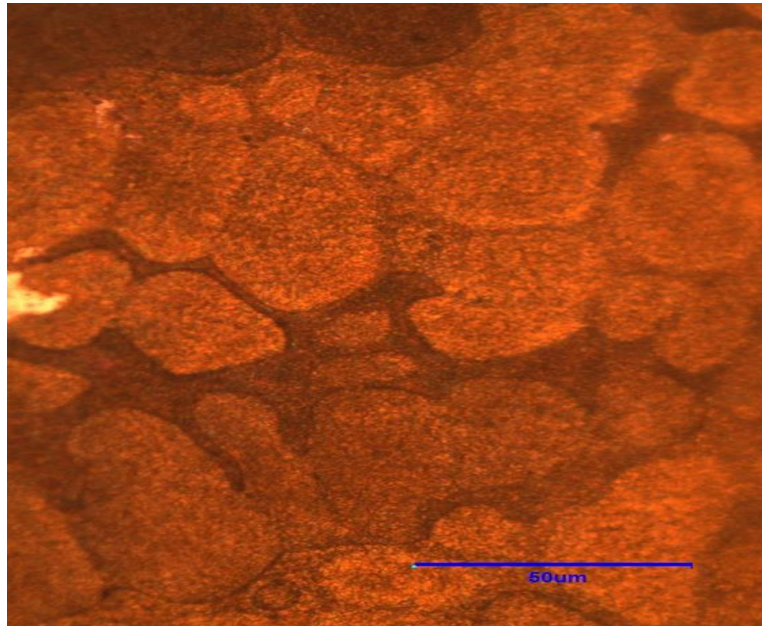


Figure 5.22 result OM of 81% Cu-7.5SiC-7.5CeO₂-4%Gr after extrusion

4.17.3. Hardness of sample after hot extrusion process

Dislocation density rises with heated deformation, leading to dynamic recrystallization and a decrease in grain size. Smaller grain size materials and higher dislocation can improve mechanical properties like hardness, compressive strength. A number of variables, including the extrusion temperature, speed, die design, and the particular composition of the copper composite, will affect how much the hardness is improved.

Table 5.27 result of hardness after hot extrusion

Sample	Trail 1	Trail 2	Trail 3	Average	% improvement
S2	132.56	145.35	146.34	141.41	70.74

Hardness of copper composite fabricated through stir casting improved 51.73% and at secondary operation (extrusion process) more improved by 70.74%.

4.17.4. Compression test of sample after hot extrusion process

Result obtained from test after hot extrusion process shows too improved in compressive strength when compared with sample before extrusion process increased from 393.758MPa to 411.23MPa. During hot extrusion process, copper heated at 0.5 of melting point can grain size as well as pressure and temperature effect on properties of material. When the

grain size of material to reduced, hot extrusion process formed due to recrystallization and improved compressive strength.

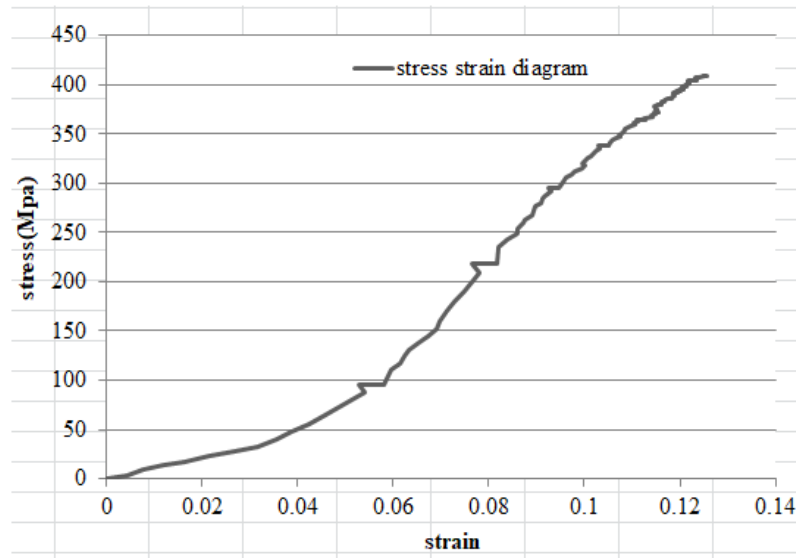


Figure 5.23 compressive strength of CMMC after hot extrusion

4.17.5. Corrosion test of samples after hot extrusion

The material's resistance to corrosion can be enhanced by a high degree of deformation and grain refinement at higher extrusion ratios. Under hot extrusion the developed sample deformed grain size particle more fine and improved hardness, porosity and corrosion resistance. The finer grain structure can create a more homogeneous microstructure, reducing the likelihood of galvanic coupling and localized corrosion.

Table 5.28 value of electrochemical corrosion in corrosion test after hot extrusion

Sample	$E_{\text{corr}}(\text{V})$	$\beta a(\text{V})$	$\beta c(\text{V})$	$I_{\text{corr}} (mA/cm^2)$	CR (mm/year)
Extruded sample	-0.212	0.054	-0.078	-0.069	0.00321

Before hot extrusion process, the corrosion rate of copper metal matrix composite was 0.0088 but, after hot extrusion corrosion rate reduced to 0.00321 with increased corrosion resistance.

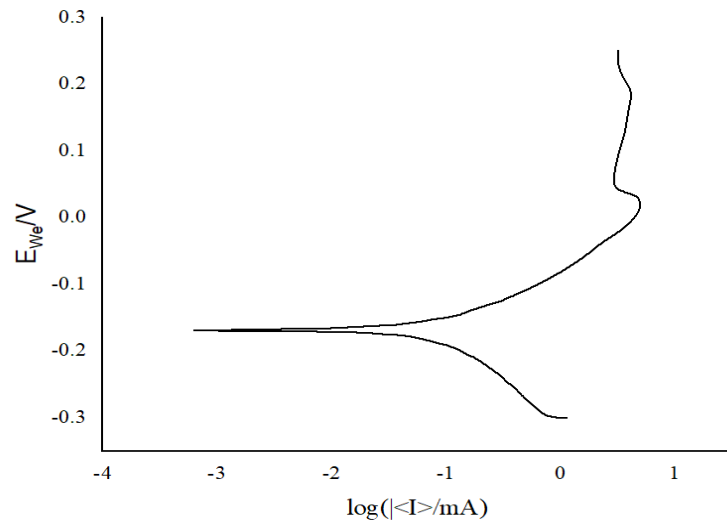


Figure 5.24 corrosion test after hot extrusion process

CHAPTER FIVE

CONCLUSION AND RECOMMENDATIONS

5.1. Conclusions

Copper matrix reinforced with SiC, CeO₂ and Gr was fabricated through stir casting. At 1100°C pure copper was heated in the furnace and reinforcements preheated at 800°C mixed with predetermined stirring time. The investigation of characterization and the effect of weight percentage on mechanical properties like hardness, compressive strength, corrosion rate, wear rate, microstructure and phase were carried out. Based on the results of experiment done on developed sample, as per S/N ratio, the sample 2 was selected (81%Cu

-7.5%CeO₂-7.5%-4%Gr). The selected material was deformed and deformation behavior were explored using 3D DEFORM software. 3D DEFORM numerical simulation was carried out to determine the parameters of extrusion process (extrusion ratio, ram speed and temperature of work piece) and responses (effective stress, load and damage values). Extrusion parameters: extrusion ratio (2, 3, 4), ram speed (1, 2, 3) and work piece temperature (850, 900, 950) °C was combined by BBD method to supported simulation process were studied. These parameters effect on load, effective stress and damage values of copper metal matrix composite deformed were explored through hot extrusion. Multi-optimization process i.e. genetic algorithm method was used to determine optimum parameters for minimum load, damage and effective stress. These optimum input extrusion parameters were used to extrude the selected sample through hot extrusion process. The conclusions drawn were as follows:-

- The results of optical microscopy indicated minimum porosity percentage of 0.78% for (81%Cu-7.5%CeO₂-7.5%-4%Gr). The hardness for this sample was improved by 19% as well as uniformly distribution of reinforcement particles were observed to greater extent.
- SEM showed homogenous mixed of particle of reinforcement in matrix and studied fractography after compression and wear morphology after wear test.
- Density was measured and calculated porosity indicated improved by 0.86% (reduced porosity).
- The reinforcements was added on pure copper matrix to increased compressive strength 393.758MPa and improved hardness by 51.73%

- Increased wear resistance and also increased corrosion resistance of CMMC improved after added reinforcements 19%.
- ANOVA results analysis by Design-Expert software indicated that extrusion ratio is significant and extrusion parameters effect on three response those' increased when extrusion ratio increased.
- In ANOVA analysis the most significant influence on damage was extrusion ratio had contribution percentage also followed had influence ram speed and temperature.
- Multi-objective optimization of extrusion parameters by using GA in MATLAB software was studied the effect of extrusion parameters was effect on responses is conducted rather than optimizing individual values.
- 81%Cu-7.5%CeO₂-7.5%SiC-4%Gr hybrid copper composite after extruded in hot extrusion process enhanced porosity from (0.86 to 0.073), hardness improved from (51.73% to 70.74%) after extruded
- OM observed after extruded and the grain size reduced from to (42.08 μ m to 36.5 μ m) so it indicated improved mechanical properties.

5.2. Recommendation

- Based on results of test and values obtained from results to recommend for other research work as follow:
- Ultrasonic Stir casting can be done to avoid agglomeration during stir casting operation.
- Fabrication of samples can be done as per some experimental design.
- Numerical Simulation of Stir casting can be carried out to explore the fabrication process in detail.
- More testing of materials like Fatigue, Creep and Flexural can be done to explore additional engineering applications.

SPECIAL ACKNOWLEDGMENT

This research work is founded by Adama Science and Technology University under the grant number ASTU/SM-R/917/23, Adama, Ethiopia.

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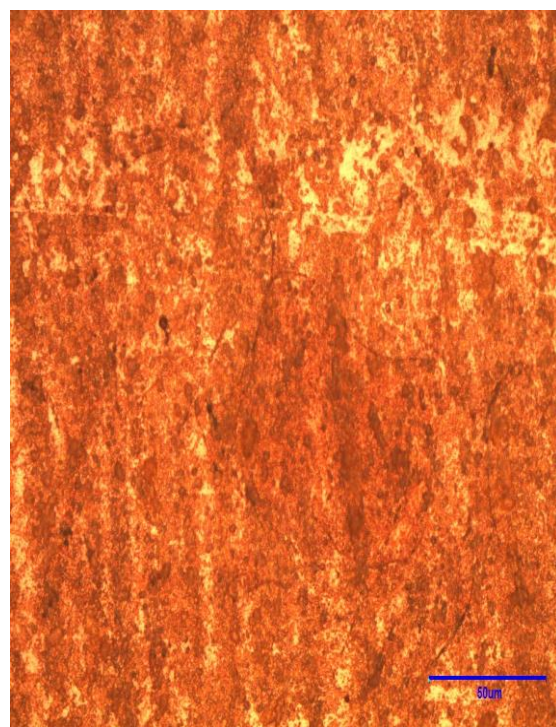
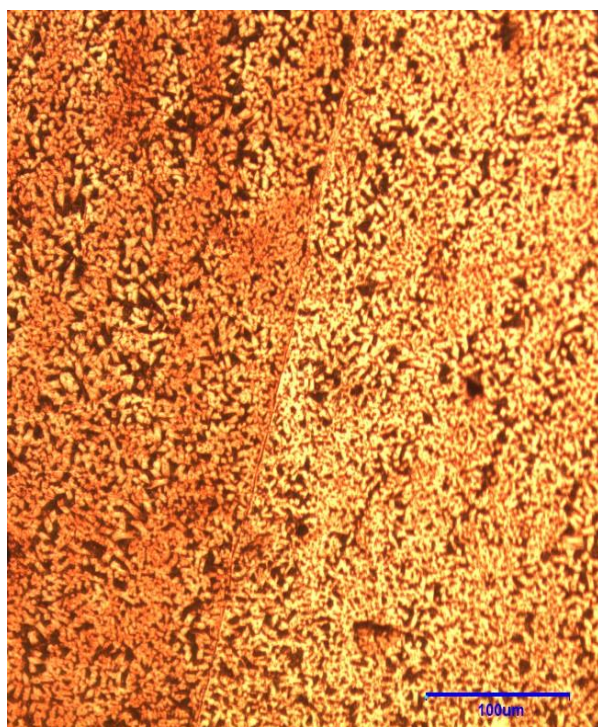
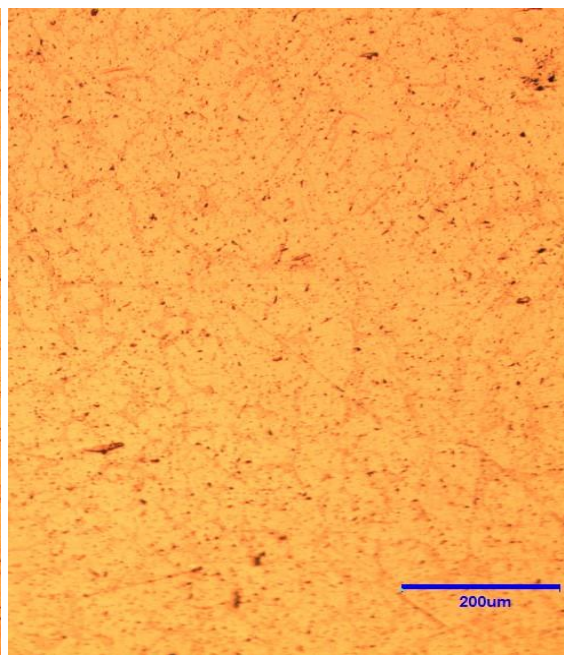
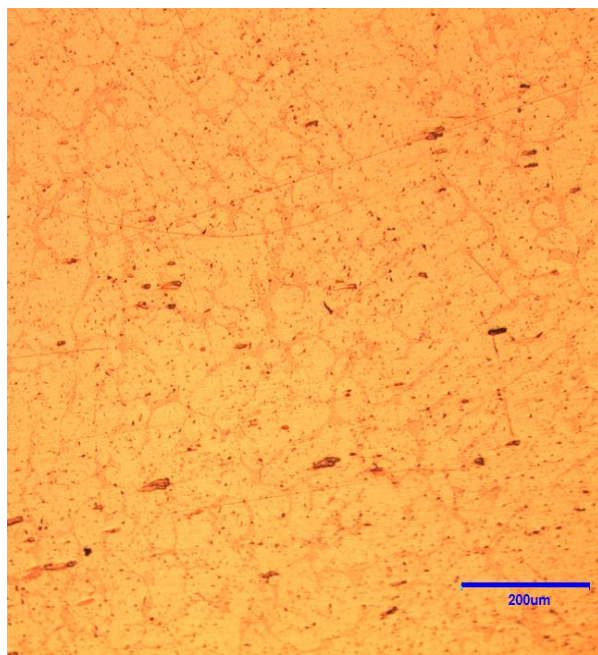
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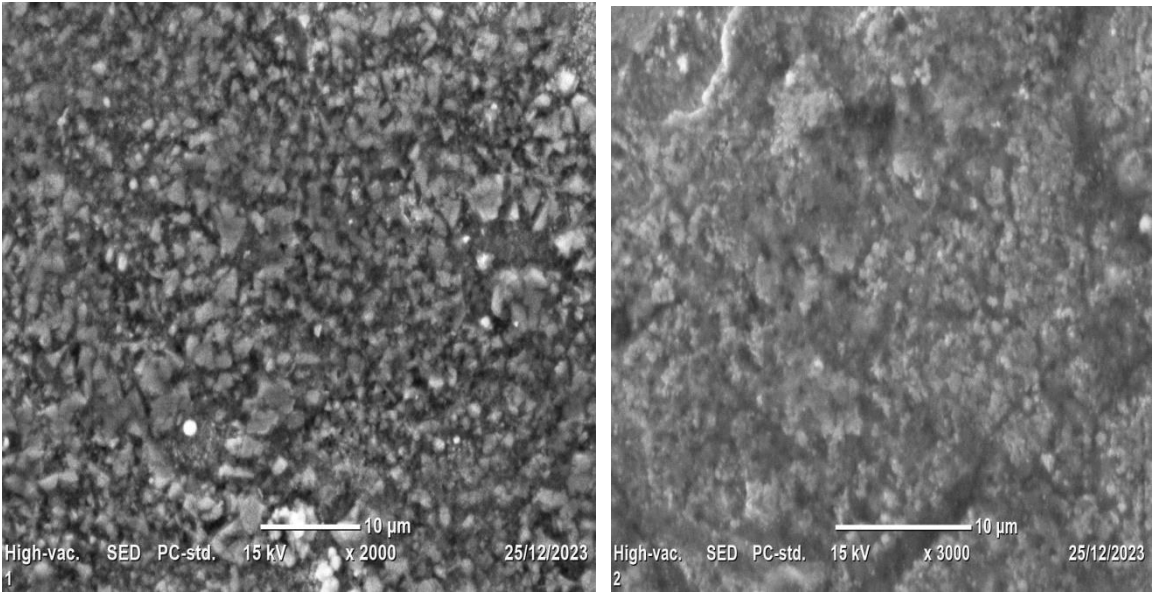
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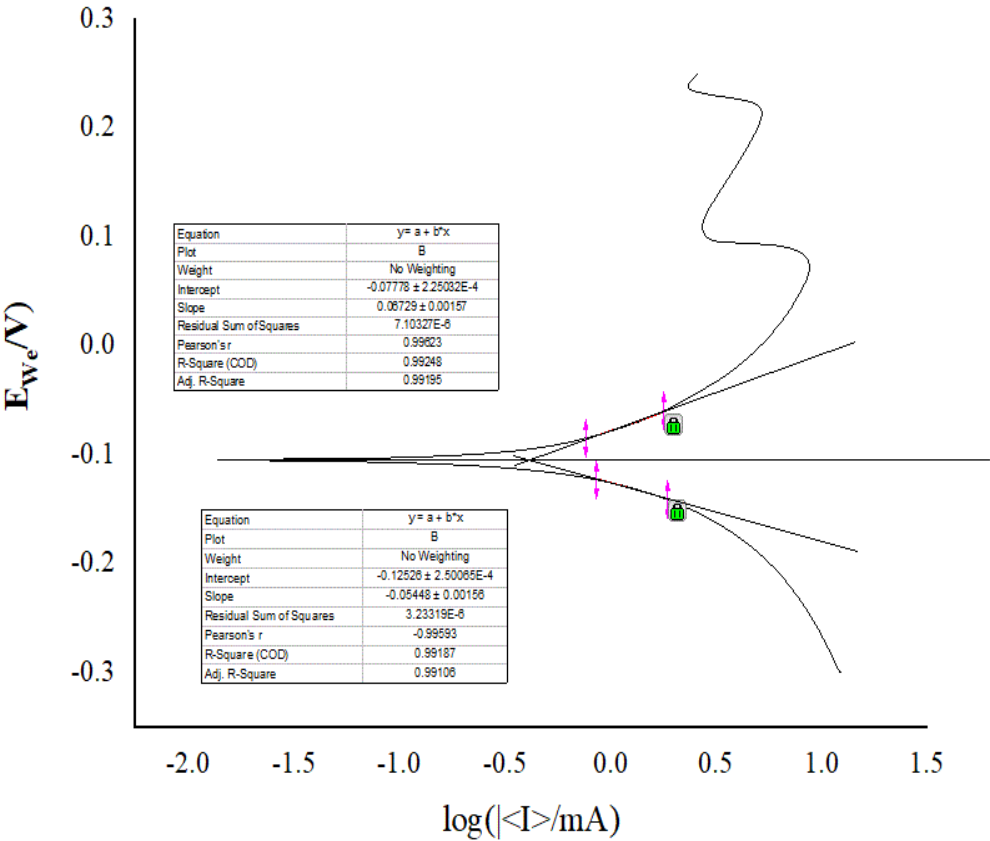
APPENDEX I: microstructural image of S1 and S2 after and before etchant at $200\mu m$ and $50\mu m$



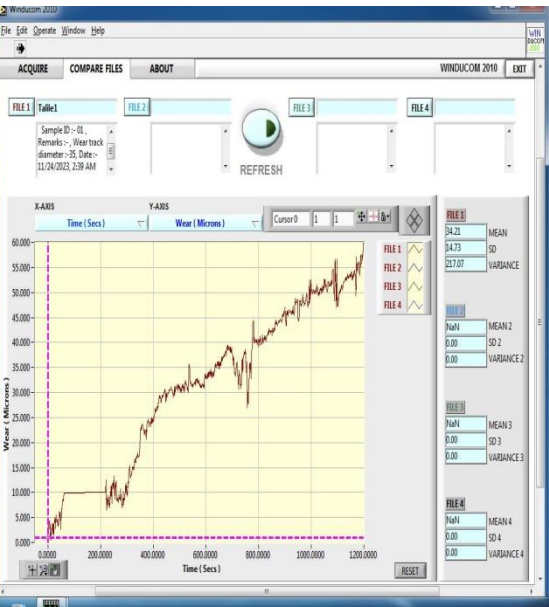
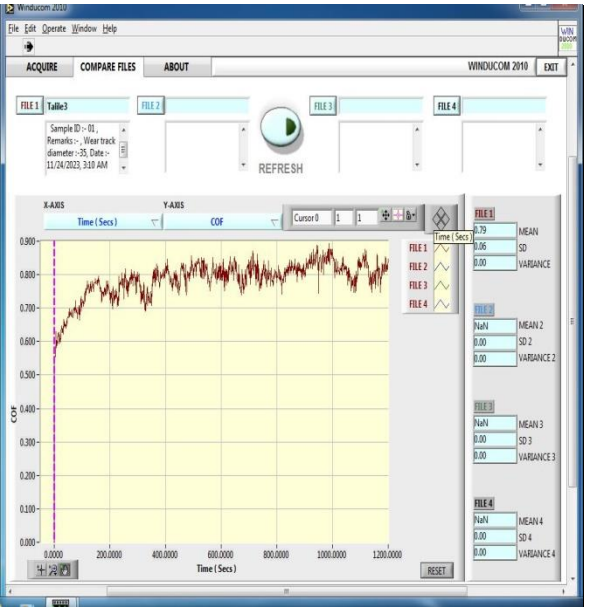
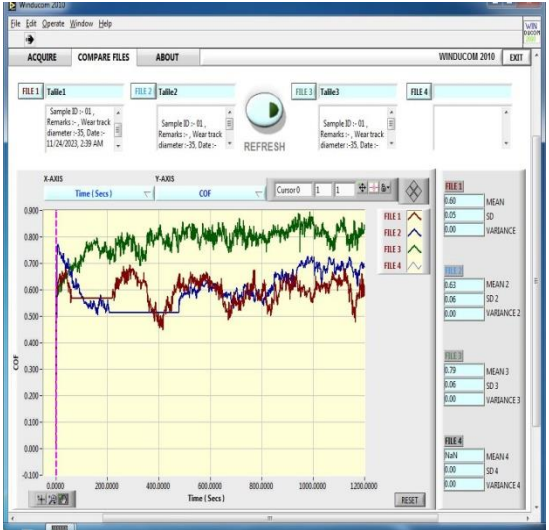
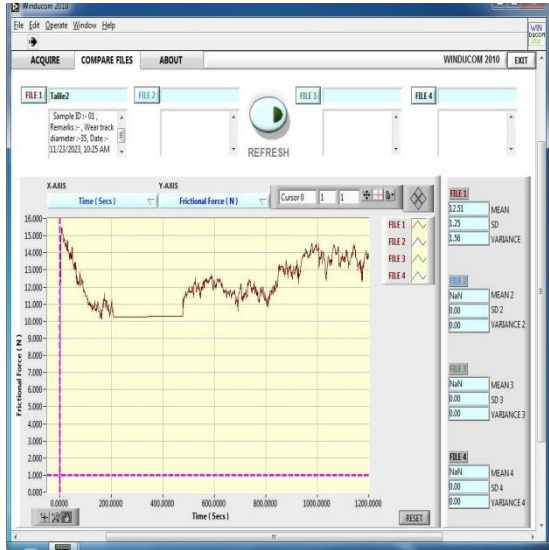
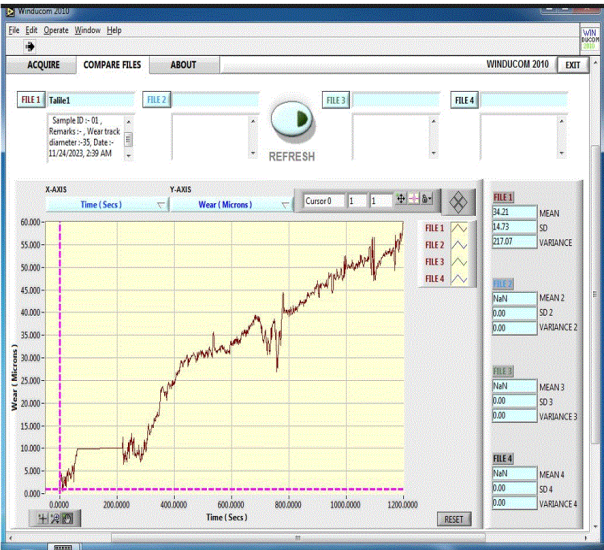
APPENDEX II. SEM image of S1 and S2 at 10 μ m.



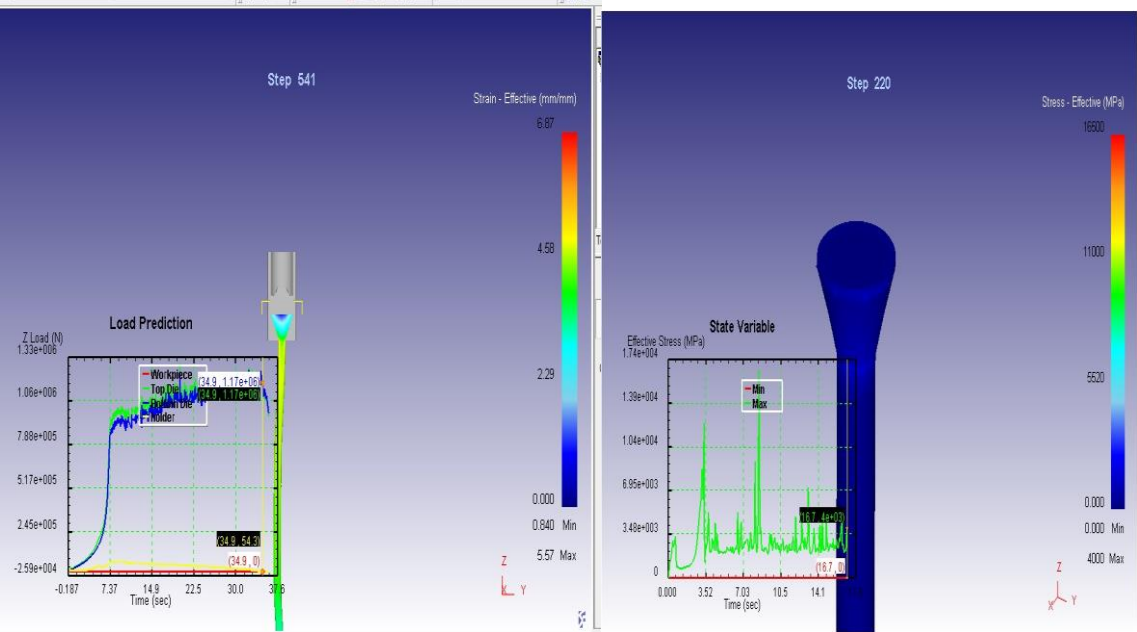
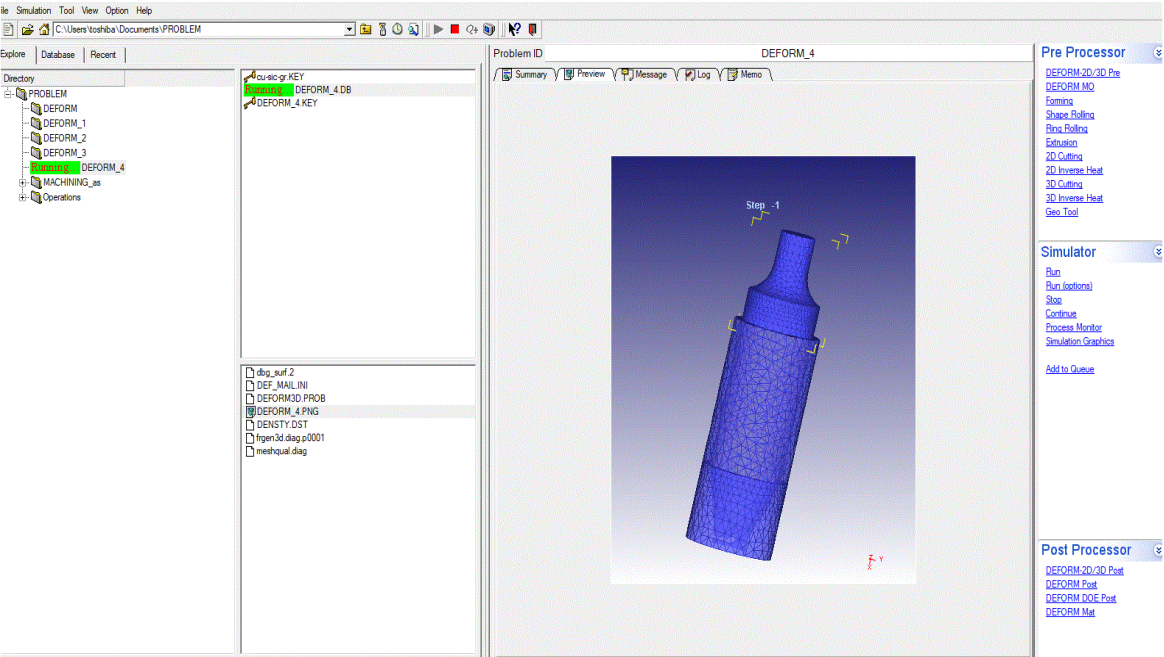
APPENDEX III. Calculation of current density, potential voltage, cathode and anode from Tafel plot

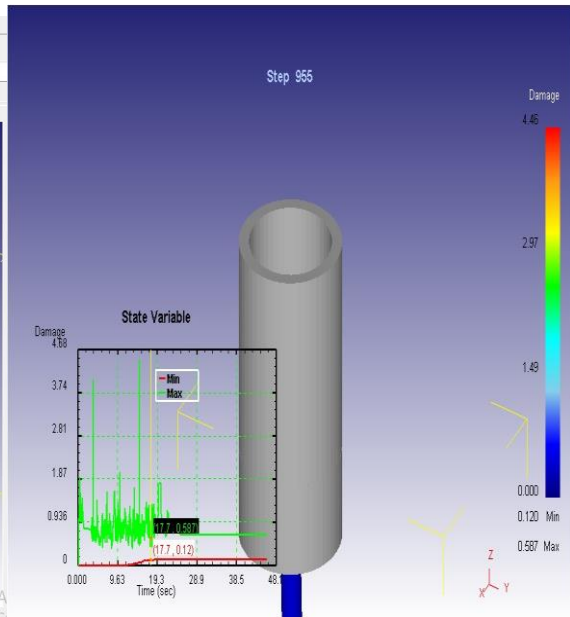
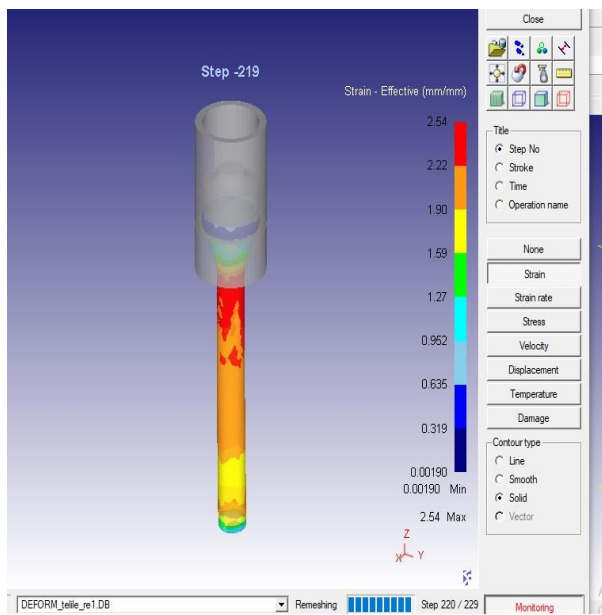


APPENDIX IV. Wear test of samples on a pin on disc for S1, S2, S3.

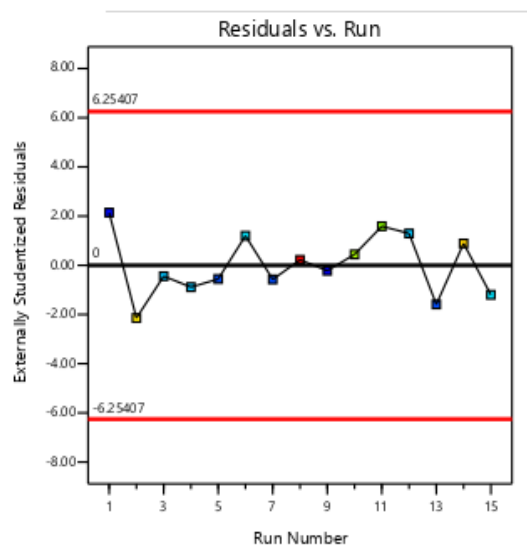


APPENDEX V. simulation process by 3D DEFORM software.

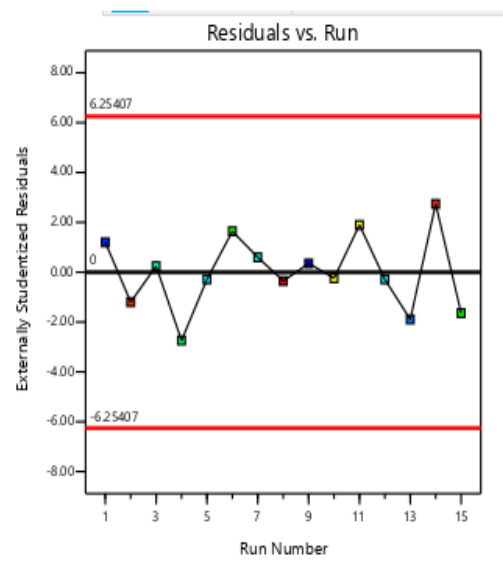




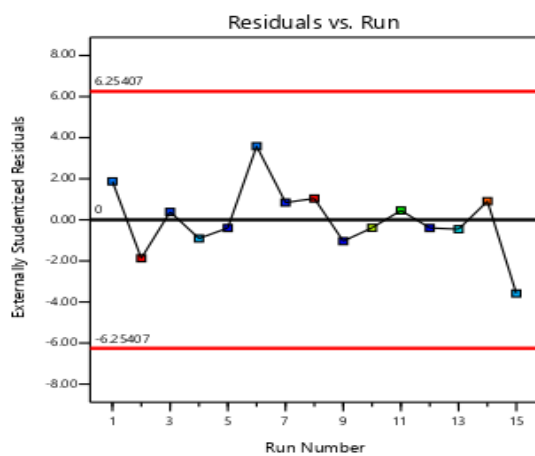
APPENDEX VI. Graph of residuals versus run number with effect of the damage (a), load (b) and effective stress (c).



a)



b)



c)

APPENDEX VII. Report table were used to produce the diagnostics graphs.

Ru n	AV	P V	Residu al	Lever age	IS R	ESR	Cook's Distance	DFFIT S	St O
1	0.3120	0.2491	0.0629	0.750	1.635	2.143	0.802	3.711	5
2	1.05	1.11	-0.0629	0.750	-1.635	-2.143	0.802	-3.711	8
3	0.4870	0.5056	-0.0186	0.750	-0.484	-0.444	0.070	-0.768	1
4	0.4880	0.5229	-0.0349	0.750	-0.907	-0.887	0.247	-1.537	7
5	0.3750	0.4127	-0.0377	0.333	-0.600	-0.557	0.018	-0.394	15
6	0.5200	0.4758	0.0442	0.750	1.150	1.200	0.397	2.078	12
7	0.3740	0.4127	-0.0387	0.333	-0.616	-0.573	0.019	-0.405	13
8	1.27	1.26	0.0094	0.750	0.244	0.219	0.018	0.380	2
9	0.3010	0.3104	-0.0094	0.750	-0.244	-0.219	0.018	-0.380	3
10	0.9500	0.9314	0.0186	0.750	0.484	0.444	0.070	0.768	4
11	0.9200	0.8665	0.0535	0.750	1.391	1.589	0.580	2.752 ⁽¹⁾	11
12	0.4890	0.4127	0.0763	0.333	1.215	1.295	0.074	0.916	14
13	0.3750	0.4285	-0.0535	0.750	-1.391	-1.589	0.580	-2.752	10
14	1.07	1.04	0.0349	0.750	0.907	0.887	0.247	1.537	6
15	0.5180	0.5623	-0.0443	0.750	-1.150	-1.200	0.397	-2.078	9