DEVELOPMENT OF METAL MATRIX COMPOSITE CASTINGS THROUGH MICROWAVE PROCESSING

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in
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by
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July, 2015

CERTIFICATE

I hereby declare that the thesis entitled "**Development of metal matrix composite castings through microwave processing**" is an authentic record of my study carried out as requirements for the award of the degree of **Master of Engineering** in **Production Engineering** at **Thapar University, Patiala** under the supervision of **Dr. Dheeraj Gupta** and **Dr. Vivek Jain**, Assistant Professor, Mechanical Engineering Department, Thapar University, Patiala during July 2013 to July 2015. Year. The matter embodied in this report has not been submitted in partial or full to any other university or institute for the award of any degree.

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Abstract

The application of microwave energy is not new in material processing, lots of developments in material processing have been carried out through this technology. This technology has been implemented in sintering, joining and coating/cladding with metal powders. After getting the tremendous advantages in material properties as well as economy, the domain has been extended towards the melting of metal powder and to cast the metal matrix composite castings. None of the material fulfils all its requirements for specific application/purpose. So, to make it perfect for different applications, the two materials are added so that the combined advantages of two materials can be obtained and known as metal matrix composites castings.

One of the common example of MMC's are copper-graphite composites, in which copper provides excellent electrical and thermal conductivity and graphite acts as solid lubricant and have lower coefficient of thermal expansion. These composites are widely used in electrical appliances, generator bushes, bearing materials etc. because of excellent properties of graphite and self-lubrication due to graphite content. In this dissertation report, Cu-graphite composite castings have been developed through Microwave Hybrid Heating (MHH) technique. In this dissertation, Copper-graphite MMCs were fabricated by mixing 2 and 5wt. % graphite, into copper powder followed by microwave processing route and compared with the cast of pure copper through same processing route. In the characterization phase, XRD spectra showed the presence of Cu and carbon in the form of graphite. The smaller peaks showed the formation of oxides with the copper but the lesser amount of oxides helped to increase the hardness of composites. The maximum Vickers micro-hardness value of around 85 Hv has been achieved for Cu-5wt% graphite composite castings and it was around 75 Hv for 2wt% graphite composite castings, which is comparatively higher than pure copper. The micrographs of Cu-graphite shows that graphite particles are uniformly distributed into copper matrix. From wear study, it is concluded that the wear resistance of the composite increases with increase in graphite content due to the lubricating properties of graphite and decrease in wear rate with addition of graphite is also calculated.

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Abbreviations

EDS Energy Dispersive X-Ray Spectroscopy.

EM Electromagnetic Spectrum.

HAZ Heat Affected Zone.

MHH Microwave Hybrid Heating.

MW Microwave.

SEM Scanning Electron Microscopy.

XRD X-Ray diffractometer.

SS Stainless Steel

Chapter-1

INTRODUCTION

In the modern era of growing and developing technologies, the lots of advancements are being noticed in the field of engineering. The industries are looking forward for new and improved processing techniques. The wide range of advanced materials including ceramics, metals, non-metals and composites can be processed easily and effectively through these technologies. With the development of technology, new materials came into existence and to process those materials high energy efficient methods were developed. But the problems associated with these technologies such as environmental degradations, high energy consumption, high manufacturing cost etc. have lead the researchers to focus on all those processing techniques which can minimise these losses upto an extent. The microwave energy lead the researchers to explore the field of microwave processing towards different areas of materials manufacturing. Recently, lot of research has reported in the field of sintering, joining, cladding etc. and applications are still increasing towards the melting of metals. This chapter will illustrate the basic overview of microwave heating of materials and applications in various fields.

1.1 Introduction to Microwaves

The microwaves are the part of the electromagnetic spectrum in which the electric and magnetic waves travels perpendicular to each other having frequency of range 300 MHz to 300 GHz. The wavelength in case of microwaves varies from 1mm to 1m as shown in Fig 1.1, which represents the EM spectrum. The frequency range of microwaves is specific for different applications, which includes communication systems, food processing, medical purposes, industrial heating and material processing [1]. The primary applications of microwaves were noticed in the communication systems including RADAR, satellite communications and television broadcasting etc. But the use of microwaves for heating purpose was discovered accidently by Spencer (1945) while he was eating ice-cream on a highway and he noticed its melting and concentration of electromagnetic field towards heating and got first patent. Over the time this energy became an important source of household product and used in food processing because of its advantages such as high heating rate, lower processing time and lower energy consumption. The frequency on which domestic microwave ovens operates is 2.45 GHz, which is primarily used for heating food items. The electromagnetic spectrum with various frequencies and wavelengths are shown in Fig. 1.1. The furnaces have been developed for material processing purposes which are used in many industrial applications and they work on higher frequencies ranging from 915 MHz to 18 GHz [2].

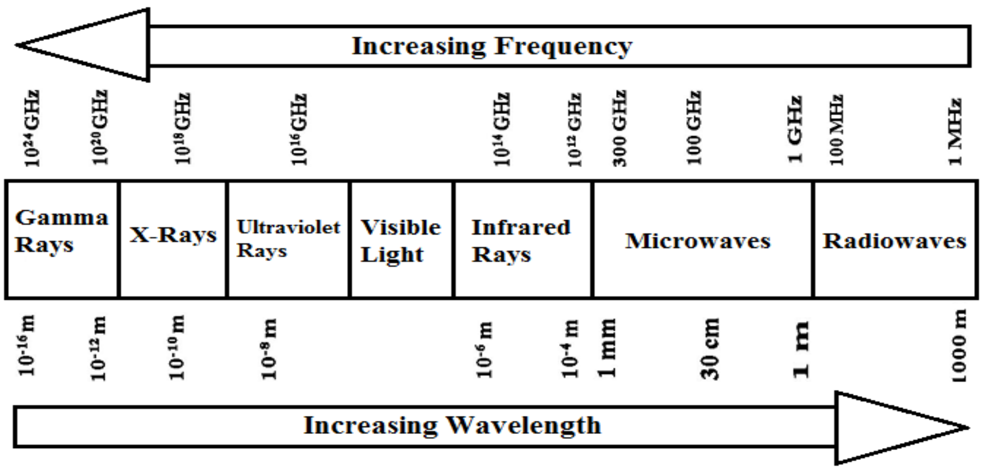


Figure 1.1: Electromagnetic spectrum carrying frequencies and wavelength of microwave band

(http://www.emf-safety.com)

The use of microwave energy was further developed in the field of material processing to gain the advantages of higher heating rates with having lower processing time [3] i.e. used tires recycling, metallic materials and ceramic processing etc.

1.2 Characteristics of Microwave processing

The microwave heating has various characteristics due to which it has become popular for heating low temperature applications as well as high temperature applications. The absorption of microwaves directly at the molecular level of coupled materials with the microwaves corresponds to volumetric heating of that material, which consequently leads to the rapid heating and reduced thermal gradient inside the materials being processed. The volumetric heating feature in microwave processing leads to high heating rate and this feature overcomes the limitation of conventional heating and consumes less processing time, which directly results in less energy consumption. An outstanding feature of selective heating in microwave processing is that microwaves directly impacts at the focused region, which interns helps to reduce heat affected zone up to an extent and reduces the defects simultaneously. The work carried out by several authors in this field reported huge amount of savings in processing time which corresponds to less power consumption during microwave processing of materials [4]. These are some of the common characteristics as shown in Fig. 1.2

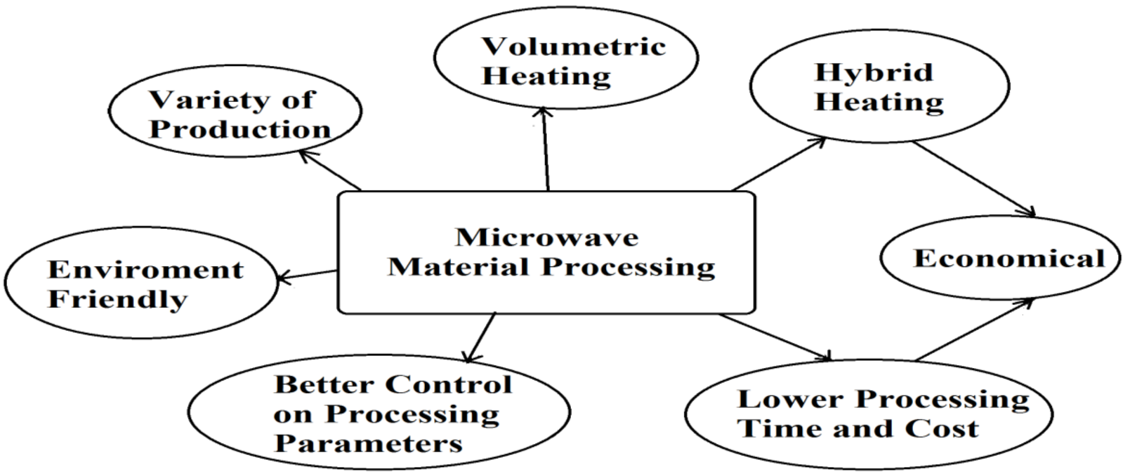


Figure 1.2: Some common characteristics of material processing through microwaves

The sintering of the ceramics through microwave processing route have been carried out easily and effectively, with enhanced diffusion rates and improved properties. After having a tremendous achievements in field material processing through microwave ovens, this technology is being implemented at the industrial level for melting purposes through microwave furnaces.

1.3 Microwave/Material Interaction

Heating/Melting is the most common process in each and every manufacturing industry but there are lots of methods to carry out these processes. As for as microwave heating is concerned, its commonly used for heating the food products as a kitchen appliance and with passage of time the use of this technology is being noticed in different fields such as engineering as well as in chemical and textile industries. There are lots of electric based heating technologies, which utilize the specific bands of electromagnetic spectrum such as induction, infrared, ultraviolet and microwave heating. Microwave technology is well known for food and rubber processing, but there is a growing interest towards the industries to properly utilize its potential for various manufacturing processes (dealing with metallic materials) as well as for the treatment of various waste-streams etc. Microwave processing leads to energy saving while it is compared with the conventional ones. Moderately, microwave processing leads to 10 – l00 times less energy consumption and operates 10-200 times fast. The material heating takes place by direct absorption of microwaves throughout the volume of material. This characteristic (volumetric heating) of microwave heating leads to enhanced diffusion, shorter processing times, finer microstructures, better mechanical properties, eco-friendly, energy savings, compact system, higher efficiencies etc. over other conventional material processing technologies. However, the heating capabilities of materials through microwaves mainly depends on the physical properties of materials and these properties play an important role in deciding the process ability of materials through microwaves. Different materials can interact in a different manner with the microwaves. Three type of materials are exposed to the microwaves and their results are represented in Fig. 1.3.

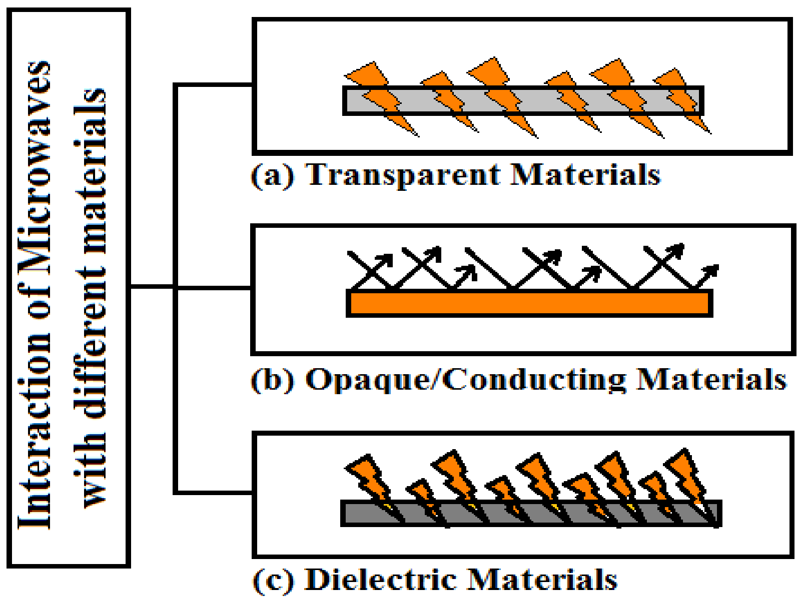


Figure 1.3: Interaction of microwaves with (a) transparent materials, (b) conducting opaque and (c) dielectric materials

The transparent materials such as glass does not have an ability to absorb microwaves and it directly allows them to pass through without having any type of loss and hence heating does not take place when rays are impacted on it. On the other hand bulk metallic conductor materials do not allow microwaves to pass and neither absorbs but causes reflection when these opaque surfaces are exposed to microwaves. This leads to the plasma formation and causes surficial heating of the body. However, third class of materials are known as dielectric materials, which tends to absorb the microwaves and heating is obtained by the conversion of radiation into heat and this principle of heating is known as microwave heating. The dielectric property play an important role in absorption of microwave and heating profile of the materials.

1.4 Concept of Conventional Heating and Microwave Heating

The generation of heat is a complex phenomenon which occurs mainly because of dipolar loss or re-orientation mechanism. When material is subjected to microwaves then electric and magnetic fields alternates 2.45 billion times in a second and this causes rotation of dipoles from the +ve to the –ve and travels from –ve to +ve. The presence of cohesive forces between the dipoles hinders the rapid reversals and causes frictional heating. The internal resistance of material also causes resistance heating on application of alternating electric fields. This process starts to take place instantaneously within the whole body and leads to the volumetric heating of whole mass subjected to microwaves. The main highlight of microwave heating is that heat is produced within the material and has inverted profile i.e. from inward to outward surface; whereas in conventional heating surface (outer zone, closer to heat) is heated first and then heat travels inward [5-6]. The difference between the heating phenomenon of conventional and microwave heating is shown in the following Fig. 1.4.

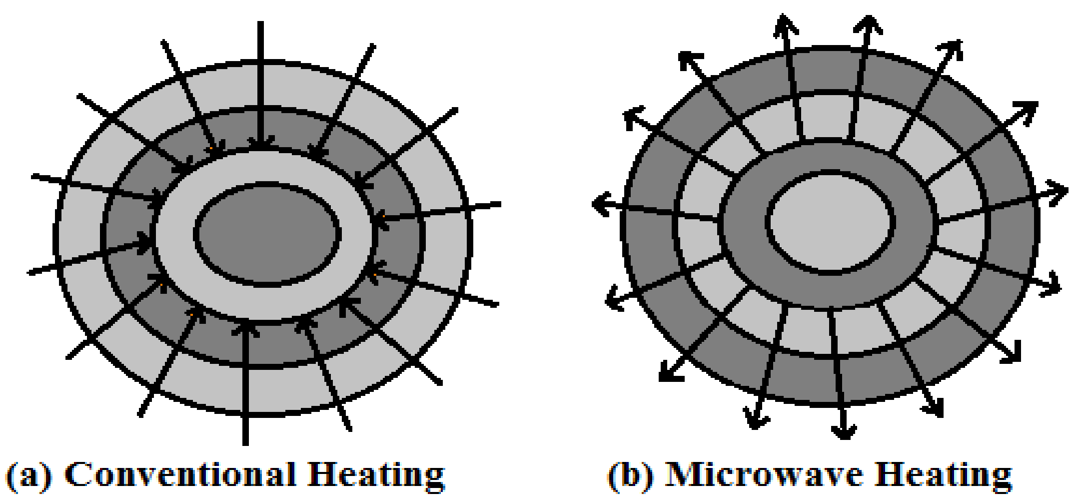


Figure 1.4: Comparison of heating mechanism for conventional and microwave heating

As shown in Fig. 1.4, the concept of conventional heating states that the heating takes place from outside to inside (e.g. boiling water on commercial oven). Basically, in conventional heating, there is a large temperature gradient between the inside temperature and outside temperature of the material. This temperature difference leads to uneven heating profile of the product. Microwave heating overcomes this limitation of conventional heating, because the heating profile generates at the centre in this type of heating and moves towards outside area. Microwave heating is a volumetric heating that why in this type of heating temperature gradient is very less as comparative to conventional heating [7]. After understanding the concept of conventional heating and microwave heating, point comes to understand the concept of microwave hybrid heating (MHH).

1.4.1 Microwave Hybrid Heating

The processing of non-coupled materials through the microwave energy is really a challenging task. To deal such types of materials through the microwave energy, the research was carried out and microwave heating came out with a different form to process these materials, named microwave hybrid heating [8-9]. This type of heating phenomenon considers the concepts of conventional heating as well as the concept of microwave heating. The concept of microwave hybrid heating with its heating profile is shown in Fig. 1.5.

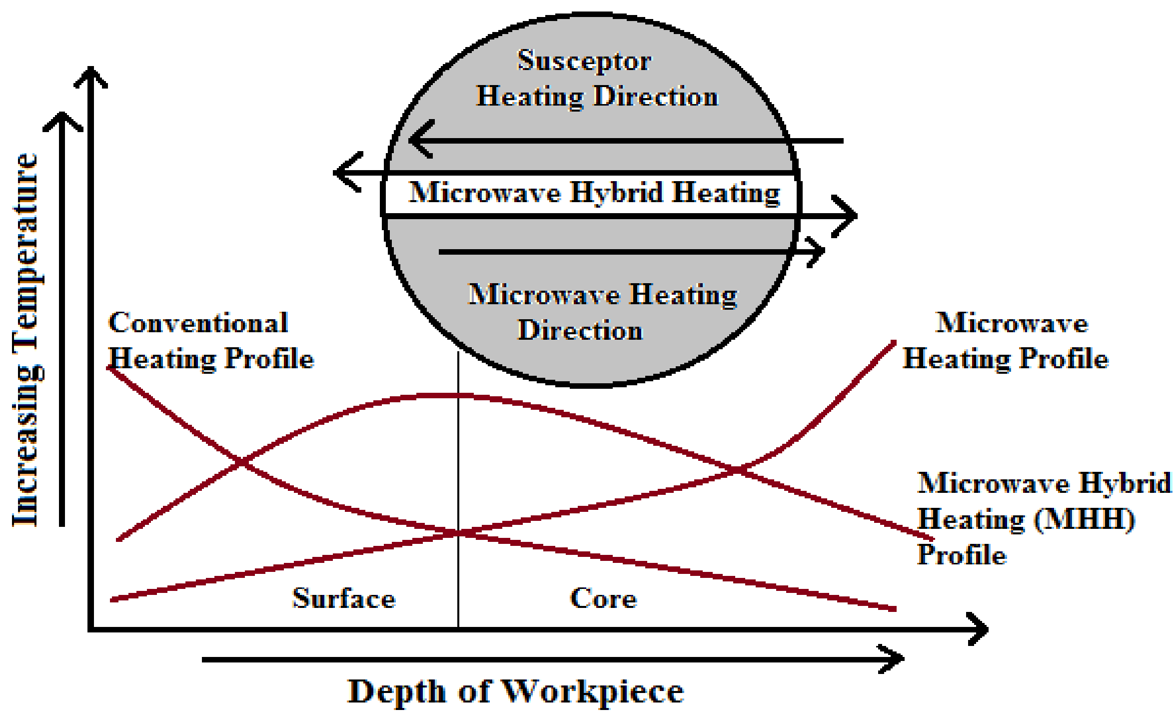


Figure 1.5: Heating Profiles for various types of heating method

In conventional heating of materials, surfacial heating occurs first and then heat transfer takes place throughout the remaining material from the outer surface to the inner surface with having a varying temperature gradient, which corresponds to the microstructures having poor surfaces and it may lead to the overheating of the surface in contact or sometimes the metallic powder may get fused. In context of microwave heating, this type of heating mode may leads to the poor microstructure of core because of carbon content at the core surface, which can cause excessive brittleness and due to burning of core cracks may generate. It's because of heating from inside to outside of the material. To reduce the thermal gradient between the surface and core and to make the limitations of these processes as an additional advantage, a new approach was introduced after lots of research in the field of material processing through microwave named it as two directional heating or Microwave Hybrid Heating, such that heating can take place from the outside towards inside as well as from the inside towards outside during processing. The heating phenomenon leads to reduced temperature gradient and high rate of heating [10].

1.4.2 An Overview of Material Processing Development through Microwaves

Basically the microwave technology was firstly used for the purpose of telecommunication in the year of 1940 and it was well known technology for the low temperature application and after this in 1950-65, technology used for the processing of rubber curing, food processing, wood curing etc. It was the time, when microwaves were used to treat the materials up having temperature of 450° C. After the passage of time with the continuous research, the range of control was achieved up to 1000° C and this technology was implemented in ceramic processing, processing of nitrides and glasses from the period of 1970-99. With the development of technology, the microwave technology used for material processing in the year of 2000 and after that claddings, sintering, melting and joining of metallic materials carried out.

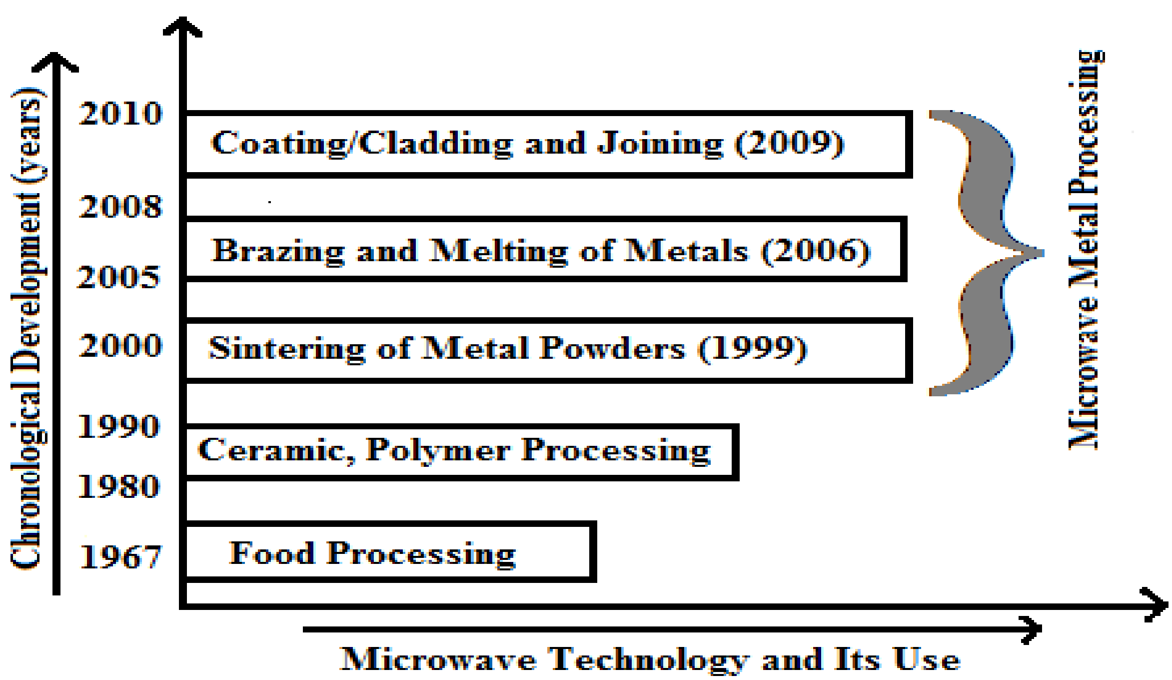


Figure 1.6: Chronological developments in material processing through microwaves

1.4.3 Basic Principle of Microwave Heating

International Electro-technical Commission defines, "Microwave heating of materials is carried out mainly through their molecular motion and their ionic conduction by the action of electromagnetic waves of 300 MHz to 300 GHz to heat dielectric materials". The basic principle behind the microwave heating is shown in Fig. 1.7.

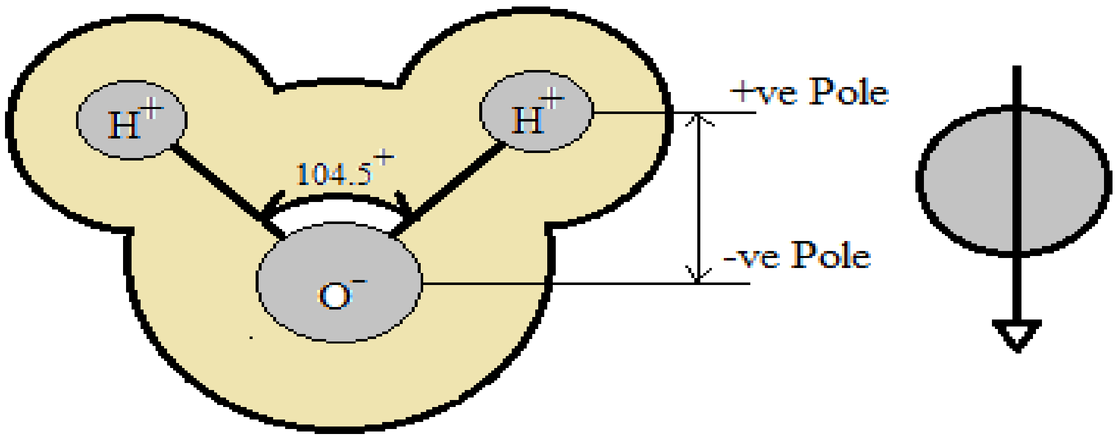


Figure 1.7: The structure of water molecule and image of permanent dipole

(http://www.microdenshi.co.jp/en/microwave)

The structure of water molecule as shown in Fig. 1.7, which consists of two hydrogen atoms and one oxygen atom. It doesn't have electric charge as total, an oxygen atom is bounded with two hydrogen atoms at an angle of 104.5°. Those two take a little charge of each plus (+) and minus (-) to form a dipole.

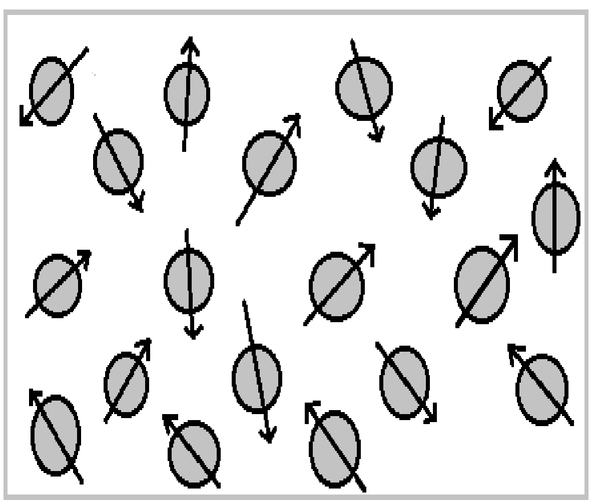


Figure 1.8(a): The structure of water molecule without electric field

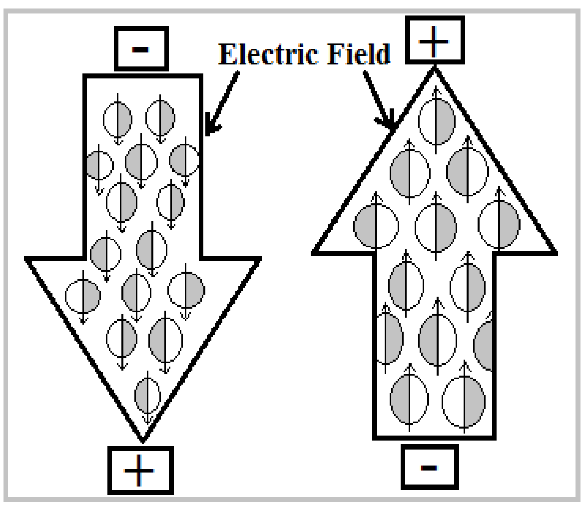


Figure 1.8(b): The structure of water molecule with external electric field

Another principle explained that when there is no external field it has set a balance as shown in Fig. 1.8 (a) but when subjected to external electric field, dipole will turn to electric field as shown in Fig. 1.8 (b).

1.5 Dielectric Properties of Materials

The physical properties of materials plays a significant role in effective heating of materials through the microwave radiations. The dealing phenomenon of microwaves (interaction of microwaves) with different materials depends upon the properties of the materials. In general, materials are classified into three main groups as described in Fig. 1.3 and microwaves deals in a different manner with all these materials. There are some materials which are known to absorb the microwaves, are known as absorbers (microwave coupled materials) and convert these radiations into heat. The properties of materials that cause the absorption of microwaves are the complex relative permittivity and loss tangent represented by the following equation [10];

$$\mathbf{P = K \cdot \varepsilon_r \cdot \tan\delta \cdot f \cdot E^2\ [W/m^2]}$$

Where,

P is the power absorbed by the material.

K = 0.056 × 10^{-10} (Constant)

ε_r = Dielectric material’s specific inductive capacity

tan δ = dielectric power factor of the material

f = frequency (Hz)

E = electric field strength (V/m)

The greatest significance of material property during microwave processing of a dielectric are the complex relative permittivity $\varepsilon = \varepsilon' - j\varepsilon''$ and the loss tangent $\tan\delta = \varepsilon''/\varepsilon'$, where ε' is called the dielectric constant mostly regulates that how much energy enters the material interface and how much how much energy is reflected back from the material. The most significant property in processing of a dielectric through microwave energy is loss tangent tan δ or dielectric loss which tells about the capability of the material to convert the microwave energy into heat. For best microwave energy coupling a reasonable value of ε' to allow adequate penetration and should be combined with high values of ε'' and tan δ to convert microwave energy into thermal energy. In microwave processing heating of material is internally and depth of penetration varies from material to material. The depth is coordinated by dielectric properties. Penetration depth is defined a depth at which approximately 1/e (36.79%) of the energy has been absorbed.

1.6 Prospects of Microwave Material Processing

Every technology when gets invented, it's not perfect from every aspect for which it's made or the purpose for it is used to be. There are lots of parameters and specifications, lots of challenges have to be controlled while dealing with newer technologies. These are lots of advantages and little bit limitations, because of which this technology has been emerged in material processing.

1.6.1 Advantages of Microwave Material Processing

There are lots of advantages of material processing through microwaves, because of which this technology has been implemented in the field of metal processing. As the research is regularly being carried out in microwave energy field for material processing, this technology is supposed to be get implemented in industries very soon. The most common advantages of microwave processing are;

- Rapid Heating
- Volumetric Heating
- Variety of production
- Environment friendly
- Energy and Cost Effective

Rapid Heating: Microwaves reach the object as same as speed of light. Firstly, these travels towards an object as waves and gets absorbed after that object generates heat. Rapid heating is possible in microwave heating because in this type of heating the heat is generated by the object by its own with penetration of microwaves by the object.

Volumetric Heating: Volumetric heating is observed during microwave heating, so there is no temperature gradient as observed in conventional heating as the temperature varies from core to surface. In microwave material heating, temperature remains same throughout the material which leads to a better quality product.

Variety of Production: There is a variety of production in microwave material processing, because of range of the temperature control and different heating parameters. This leads to the microwave technology in the field of metal processing through microwaves and having variety of production in different areas of production like sintering, cladding/Coating, Joining of metallic materials.

Environment Friendly: Microwave energy is said to be completely environment friendly as compared to conventional energy methods of material processing. Conventional methods for heating the metals in the form of furnace heating generates fumes, dangerous gases, smoke etc. which leads to environment degradations and this thing can be avoided in microwave processing so its environment friendly from this point of view.

Energy And Cost Effective: Microwave Heating leads to energy saving as well as cost saving. The energy requirement to rise the temperature in furnace for melting a material is very high but this requirement of energy can be fulfilled very easily and vastly with microwave energy. Energy saving directly or indirectly leads to cost effectiveness, which also tends to save the time because of its high efficiency.

1.6.2 Limitations of Microwave Material Processing

As the coin has two sides, having head and tails, similarly every process has some pros and cons but how to minimize or to control these depends on the skills of the operator. The most common limitations of microwave processing are;

- Unknown temperature inside the microwave cavity.
- Poor coupling between metallic materials and microwaves.
- Controlling the rate of heat transfer with respect to different materials.

All these parameters need to be get optimized because overheating may get fuse the metallic powders and radiations may harm the inefficient worker, so it should be handled/operated carefully.

1.7 Introduction to Casting

Casting is a manufacturing process, in which the metal in its liquid form is poured into the mould and after cooling, solidification takes place inside the mould. Solidified part is then ejected out of the mould. Casting is used to produce intricate shapes of the products, very large dimensional pieces, and for the mass production. There are different types of casting materials (pure metals, their alloys, composites) used as per the requirements of the industries. With the development in technology, the requirement of new materials diverged this domain towards composite materials.

1.7.1 Metal Matrix Composites (MMC)

The particulates reinforced metal matrix composite (MMC) is a new approach in the field of casting. These are newly developed structural members and a gradual development has been noticed during last two decades because of their outstanding properties and seems to be an important part of future products. For several years, fabrication of metal matrix composite is the main focus of the researchers because neither matrix nor reinforcement alone can't fulfil all the requirements [11]. So various materials have been mixed with each other and fulfilled the required properties, which are different from their base materials and became the inbuilt properties of the material like stiffness, strength, corrosion resistance, wear resistance and elastic modulus etc. The fiber-reinforced metal matrix composites (MMCs) provides the following advantages than compared to the base metal alone are high specific stiffness, wear resistance and high temperature performance, corrosion resistance and elastic modulus etc. These properties are required for each and every product being manufactured, but the cost to manufacture the MMCs and difficulties which comes into play during fabrication are the main reasons behind their limited use for commercial application. In last several years, with the development in technology, several casting techniques (squeeze casting, Investment casting, die casting etc.) came into existence and MMCs now can be easily manufactured. MMCs can also be made with ceramic reinforcement, commonly used ceramics are (silicon carbide, boron carbide, alumina etc.) which may improve the mechanical properties of the materials up to an extent.

1.7.2 Properties of metal matrix composites

Metal matrix composite (MMC) castings are the types of castings through which the combined advantages of two materials can be easily obtained. In MMC's one of base metal is metal and other being a reinforcing material (ceramic). These advantages includes [12];

- Extremely lightweight
- Strong and corrosion resistant
- Better mechanical and thermal properties

MMC castings exhibits reduced material loss and reduced machining time and ultimately resulting in a more efficiently manufactured component for complex shapes and higher volumes. There are different types of processes to cast the metal matrix composites, which are shown in Fig. 1.9.

1.7.3 Types of Metal Casting Processes

These are some of the most common processes used to manufacture the castings and MMCs.

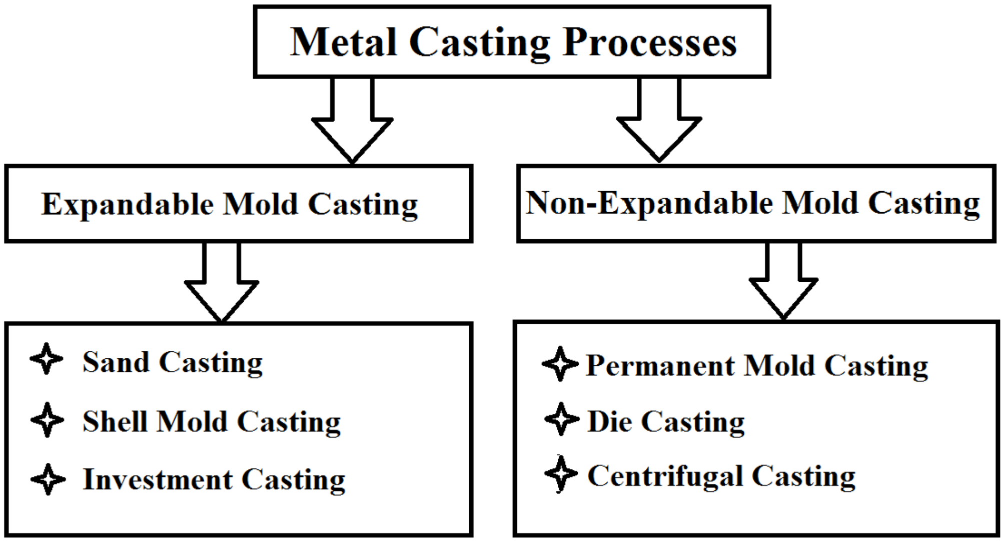


Figure 1.9: Types of Metal Casting Processes

With the development in technology, more and more problems faced by the scientists and technologists in the field of manufacturing and material processing for new materials and different metal alloys and composites these casting techniques came into existence.

1.5 Fabrication of Metal Matrix Composites

For the fabrication of metal matrix composites two materials are needed, one for matrix (base metal) and the second one is as a reinforcement material. The matrix materials are commonly metals and its alloys (aluminium, copper, magnesium, titanium and nickel alloys) except all these materials several many more matrix materials have also been used including super alloys [13]. The reinforcement materials may be metal and ceramics or glass fibre reinforcement. The choice of a reinforcement mainly depends on the end use of the product, for which purpose it's manufactured. The main parameters, which influence the selection of a particular fabrication process are:-

(i) Type of matrix and reinforcement,

(ii) The orientation and distribution of reinforcements,

(iii) The thermal and mechanical properties of matrix and reinforcement,

(iv) Finally its end use and cost-effectiveness.

Fabrication methods to manufacture the metal matrix composites are very complex and diverse in nature. Major problems are concerned with the densification parameter, which may sometimes get influenced because of the excessive or improper chemical bonding throughout the composite materials such as between matrix and reinforcements. The fabrication processes are generally subdivided into two groups based on its physical state i.e. solid state processing and liquid state processing. In case of solid state processing, the matrix is in the form of powder, sheet or foil. To manufacture the metal matrix composites through solid state processing, two types of processes comes under this category are power metallurgy and diffusion bonding. On the other hand side, in liquid state processing, casting of metals comes into the role. Solid state processing overcomes the limitations comes into play while carrying out the casting (liquid infiltration processes). These can be summarized as lower processing temperatures, slower diffusion rates, and the chances of reaction between the matrix material and reinforcements are very less. The other manufacturing processes comes under the metal forming such as forging, rolling, drawing and extrusion are also important, but lots of care is needed to reduce the damage in case of reinforcement.

So, in the present study the work is being carried out to cast the metal matrix composite castings through microwave processing and this is the form of solid phase processing. As far as the research is concerned to fabricate the composites through microwave technology, no work yet has been carried out regarding the casting. So, it’s a very challenging task to cast the metal matrix composites through this technology.

Chapter-2

LITERATURE REVIEW

2.1 Introduction

In this chapter, the literature on the basis of different metal processing techniques through the microwave processing route are elaborated. Literature contains various metal processing techniques, which clarifies the fundamentals regarding the working principle and parameters includes sintering, cladding, joining and melting of metals through microwave processing. This chapter also contains the literature survey in the field of casting to understand the concept of manufacturing, various methods to cast the MMCs and change in their properties. Brief observations are highlighted from the authors work and are presented for better understanding.

2.2 Perspectives of Microwave Processing

Das *et al.* (2009) [14] represented various prospects of microwave energy**,** in which the use of microwaves in various fields of material processing were highlighted. In which, they revealed that microwave heating is fundamentally different while its comparison is done with the conventional one. During microwave processing, electromagnetic energy gets converted into thermal energy and helps to heat the material in the form of microwave heating. The heating of material can be carried out by two forms of heating, it may be radiation and/or convection or the combination of both i.e. transferred to the bulk material through conduction mode. Various areas in which microwave application is commonly seen when point comes to the material processing are, sintering of ceramics and metal powders, processing of polymers, joining of metals, melting of metal powders, coating/cladding development etc. In this study they also revealed the advantages of microwave processing, which includes unique microstructure and properties when compared to conventional ones, less energy consumption, reduced manufacturing cost etc. They highlighted the feature of selective heating of material in microwave processing, this feature heats the material at the molecular level and leads to save the processing time and can enhance the product quality. In case of microwave heating, transfer of energy is noticed at the molecular level and due to volumetric heating, it corresponds to a reduced thermal gradient but in conventional heating to reduce a steep thermal gradient, low heating rates are selected which can be considered as a compromise between processing time and product quality.

Agrawal (2010) [15] did a study to find out the latest developments in material processing, in which he spotted a light on different areas of microwave material processing. Microwave technology is an innovative technology because of its additional advantages over conventional processing, which leads to reduction in processing cycle time and which indirectly helps to reduce the cost associated to manufacture component. This technology helps to achieve the improved microstructure and no environmental issues are related to this technology. Most recent application of microwave technology was noticed for steel making and recycling of tires. Selective heating feature of microwave technology was also utilized for brazing and soldering of metal parts/components. In this study, the concept of melting and sintering of ceramics was explained and its comparison was done with respect to the conventional sintering as shown in Fig. 2.1.

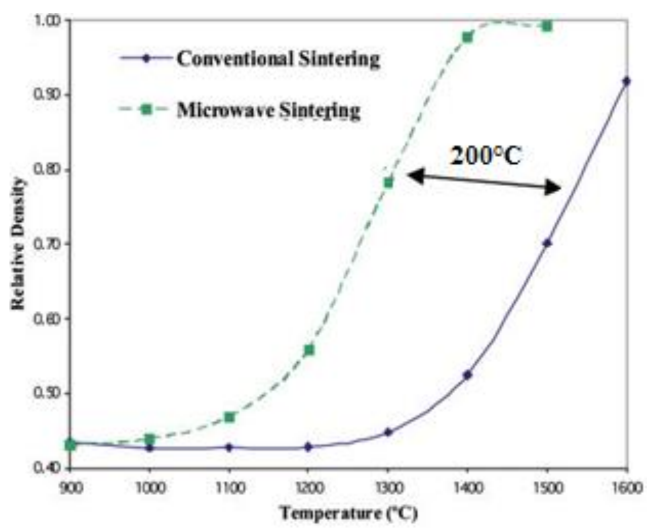


Figure 2.1: Comparison between Conventional sintering and Microwave Sintering

As shown in Fig. above, microwave processing produces relatively dense structures comparatively to conventional processes and at lower temperatures than that of conventional processes. The main reason behind the limited research in case of metals processing through this technology, is the misconception towards people that most of the metals reflect the microwaves. After facing lots of problems and research in this field, it has been proved that metallic materials can absorb the microwaves in the powder form. Steel making contributes to the formation of CO_2 in green-house effect because of the fumes produced through conventional furnaces, so a process with less CO_2

emission was required for the steel manufacturing. Hwang et al. got an outstanding achievement by combining electric arc furnace with the microwave processing and developed a new technology, which leads to a remarkable reduction of CO_2 generation up to 50% as compared to conventional steel making technology and 25% less energy consumption over the basic furnace technology.

Clark and Sutton (1996) [16] reviewed the capability of microwaves to process metallic materials. The material being processed through microwaves, interacts for a short duration of time with the microwaves rather than radiant heat exposure in conventional processes. In this technology, the heating is volumetric and selective or rapid heating can be achieved because the heat is generated by material itself. The rate of heating is proportional to the power. These features leads to uniformity and variety of production. The equipment facility requires less floor area and faster production throughout the process which corresponds to proper utilization of the facilities. Authors summarized the advantages of microwave processing over the conventional ones and different areas in which this technology should be explored. The microwaves at the atomic level can be absorbed, transmitted or reflected by materials and these properties may vary from material to material. All three states (solid, liquid and gases) can interact with microwaves and can be heated. In comparison to conventional heating processes, microwave processed materials commonly exhibits higher temperature at the center than on the outside surface because heat travels from inside to outside. Various aspects of the microwave processing have been also highlighted i.e. heating and drying rates, electric field distributions, temperature profiles, power absorption and hybrid heating.

2.3 Microwaves in Surface Engineering

In the year 2010, Gupta and Sharma developed a novel processing technique for deposition of metallic materials on metallic base metal by using a domestic microwave oven of frequency 2.45 GHz to improve its surficial properties and mechanical strength compared to the substrate and the process was claimed for Indian Patent [17].

Gupta et al. (2011) [18] did an experiment to investigate the dry sliding wear behaviour of WC10Co2Ni clads processed through microwave radiation**.** Wear analysis is important from the engineering point of view, because most of the component gets fail due to wear and corrosion, it may be due to aggressive working conditions. The common fields, in which these problems generally arises are the components of hydro-power plants and gas turbines. Generally, to design these type of material two methods are adopted.

1st method- Design a bulk material having properties of wear and corrosion resistance.

2nd method-Modification of surfaces which generally comes in contact with the sliding conditions.

Designing a bulk material leads to high investment cost to manufacture it, so generally modification of surfaces by mechanical means is generally adopted, to provide a high wear resistance and high hardness to the substrate material (Austenitic SS). So, the microwave cladding was carried out and after producing it, the mechanical properties of the cladding were measured. There was a huge difference noticed before and after the micro-hardness of the material, the hardness was increased upto 1064±99 HV at the top of the surface and at the interface it was around 800 HV, which was far-far more than the substrate alone. Microwave cladding gave a higher wear resistance, which was about 84 times higher as compared to the substrate material (SS-316) having velocity 0.5m/s.

Sharma et al. (2012) [19] did an experiment on surface engineering and its effects on the microstructure and flexural strength of cladding developed through microwave processing. In this research, metal-ceramic composite cladding was developed on the substrate. As a substrate material, austenitic steel was used and Ni based EWAC having 20% WC10Co2Ni as a reinforcing agent was used for composite cladding. Nickel was chosen because of its toughness property and corrosion resistance at elevated temperatures and WC helps to increase the hardness as well as contributes to higher wear resistance. Thus, a cladding produced with this composition is supposed to have tensile and compressive stress bearing capability. Through microwave processing, a defect free cladding with 0.89 porosity was manufactured. This reinforcing composition on a substrate helped to increase the hardness of SS from 200HV to 416±20HV and flexural strength of clads 629±8N was achieved.

Gupta et al. (2014) [20] represented a new technique of coating through microwave, it was the new approach in surface engineering. In this cladding approach they used WC10Co2Ni powder on SS-304 through microwave hybrid heating, the partial dilution of the substrate (austenitic steel) with the cladding material made a metallurgical bonding between the substrate and deposits. Austenitic steels are known for their corrosion resistance properties, this is the main reason of their high demand in industries. But, when the point comes on wear, these materials show poor tribological properties. To enhance this property surface engineering in the form of microwave processing is a well-established technology over the other surface engineering techniques like nitriding, cyaniding and carburising etc. The basic working principle with experimental set up as shown in Fig 2.2.

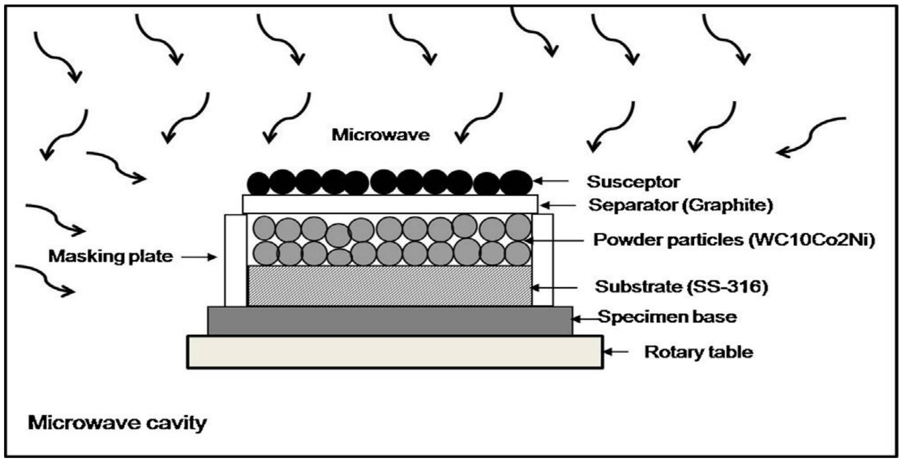


Figure 2.2: Experimental setup for Cladding/Coating through microwave processing

Because of its excellent feature of dilution between the clad layer and substrate material and surficial finish, which leads to a better metallurgical bonding and improves the mechanical properties. In this study, the ease of processing and benefits over the laser cladding have also been discussed, which leads to lower setup cost, maintaining cost as well as lower operating cost and higher deposition efficiency. The cracking tendency generally induces in laser cladding processes due to high cooling rate and localised thermal distortion and residual stresses are induced on the substrate, these things can be avoided upto an extent due to furnace cooling. The cladding produced through microwave processing showed an excellent resistance to abrasion and erosion wear due to formation of carbides of tungsten and nickel. WC helps to improve the hardness and wear resistance but having a low toughness, this balancing requirement was fulfilled by nickel content, so this combination became a superb cladding material by fulfilling all the required properties of the substrate material.

Prasad and Gupta (2013) [21] did a microwave processed cladding of nickel based lanthanum oxide composite powder particle having particle size of 40 μm on mild steel. The substrates were cut into average dimensions of 10mm × 10mm × 5mm. Then they were polished with emery paper to obtain the artificial texture. In this study, mild steel was used as substrate and composite powder was placed on it. The sample was placed in microwave oven with 2.45 GHz frequency, 900 W power and exposed for 240 sec. The approximately 500 μm clad thickness has been developed on mild steel. The vicker's micro-hardness of developed clad sample was obtained around 319 Hv and clads showed a better wear resistance and mechanical properties.

2.4 Microwave Joining of Metals

Joining of metal can be done by many techniques to make the high strength joints but microwave energy played a vital role in this field to join the metals with improved mechanical properties. There are many techniques to join the metals like welding, brazing, soldering, laser welding etc. but these all techniques consumes lot of energy and time. Microwave assisted joints are clean and more efficient than conventional ones. Following literature represents the use of microwave energy in joining of metals.

Srinath et al. (2011) [22] explained a methodology for joining of metallic materials through microwave processing. In this research, joining of copper was carried out using domestic microwave having frequency 2.45 GHz and power 900 W. Copper was selected as a material for joining through microwave heating because copper is one of the most commonly used metal in manufacturing of electrical devices and components used in aerospace industry. The experimental setup for joining and pre-placement of joining sample is shown in Fig. 2.3.

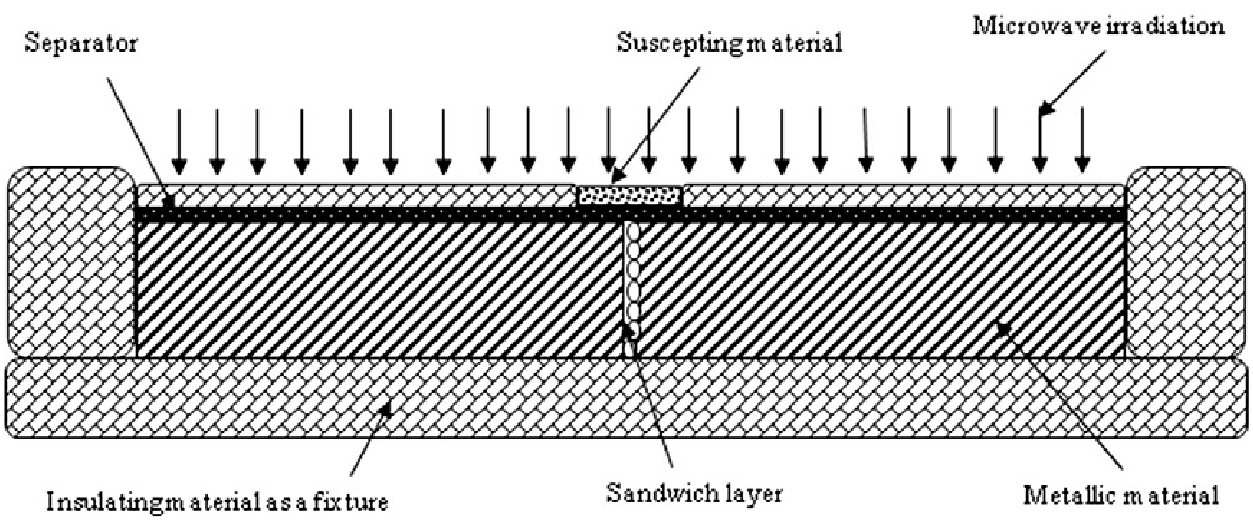


Figure 2.3: Schematic view of the experimental process for microwave joining [9]

As the microwaves are reflected by the metals, so susceptor was used to enable heating. The fine charcoal was used as susceptor and copper in the form of plates and coins was joined successfully through microwave processing in 15 min. The copper powder was filled in a gap between the two preplaced samples having a gap of 0.5mm between each other. Due to the complete melting of metal powder at the interface due to microwave exposure lead to the metallurgical bonding of metal pieces. Characterization of the joints was carried out. A uniform microstructure with better metallurgical bonds between the sandwich layer and interface was formed. The hardness was achieved upto 78±7 Hv and porosity was controlled upto 1.92 %. Microwave processed copper joints possessed significant tensile strength with high elongation.

Srinath et al. (2011) [23] investigated the mechanical as well as microstructural properties of similar or dissimilar joints fabricated through microwave processing. The joining of SS-316 with mild steel in bulk was carried out successfully using microwave multimode applicator at 2.45 GHz and power 900 W. Microwaves can't couple with the metals directly at room temperature, so a susceptor medium was used to initiate the coupling of microwave with metals and joining was carried out by using microwave hybrid heating. To form a joint, EWAC (nickel based) powder was used to fill the gap between two metal pieces. The characterization of developed joints was carried out using FESEM, XRD, micro-hardness and universal testing machine. The volumetric heating nature of microwave processing resulted in complete fusing of the interface layer, which developed a metallurgical bonding with bulk interfaces. Formation of metallic carbides has been observed through microwave heating. Bulk joint has a Vickers micro-hardness 133 Hv and porosity of bulk joint was observed to be0.58 %. The dissimilar joints processed through microwaves possessed the tensile strength upto 346.6 MPa with an elongation of 13.58 %.

Bansal *et al.* (2013) [24] carried out the mechanical and metallurgical characterization of MS-MS joint fabricated through microwave heating using nickel powder as interfacing material. Joining of the bulk metallic materials is the most challenging task with the help of microwave energy, because of poor coupling of microwaves with the metallic materials and microwaves are generally reflected by the metals. In this study, microwave hybrid heating (MHH) technique was used for joining. A nickel-based powder of particle size 40 μm was used as the interface layer for the fabrication of the joint. The bulk MS plates of were placed in such a manner that a butt configuration and a gap of approximately 0.5 mm was maintained between both the samples. The gap was filled with the slurry, having the mixture of EWAC (Ni base powder) and epoxy. Because of the microwaves reflection phenomenon by metallic materials, it was very difficult to deal with the microwaves at room temperature. To make it possible that microwaves get absorbed into the material, an insulator mask of fine charcoal was formed around the joint and by doing so, metal was not directly exposed to the microwaves.

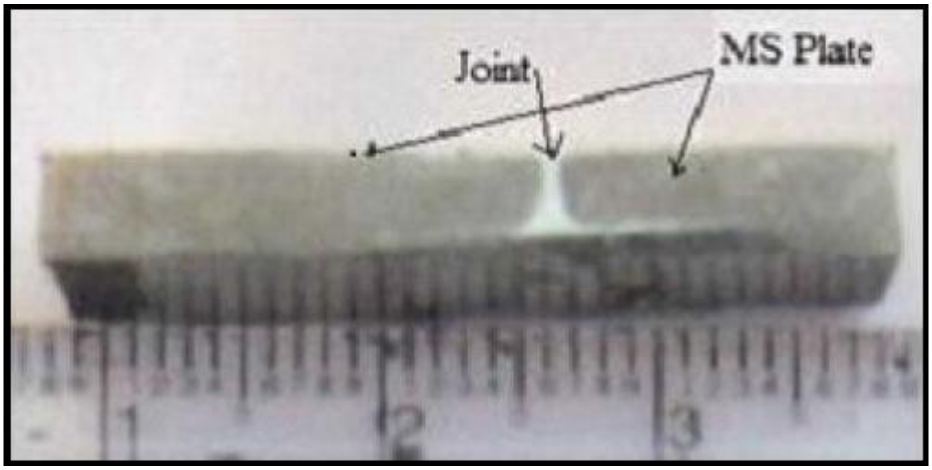


Figure 2.4: A view of joint formation through MHH [24]

Micro-hardness of the joint as well as base metal was measured using a load of 50gm for 20sec. The size of indentations were smaller on the joint zone and larger on the base material (mild steel). This size of indentations clarifies that the joint formed has higher hardness as compared to the base metal, which is desired for better quality of joint.

2.5 Microwave Sintering of Metal Powders

Sintering is the manufacturing process, in which product is fabricated in the form of solid mass by applying heat and pressure. Sintering is the operation in which the product is not allowed to reach melting state. Sintering as a manufacturing process is used with ceramics, plastics, metals and other materials. Sintering can be carried out by using conventional methods but the microwave assisted sintered materials have possessed higher properties. Following literature represents the use of microwave energy in sintering.

Rajkumar et al. (2012) [25] represented the chemistry of cu-graphite sintered composite through microwave processing by calculating its wear behaviour. The most common applications of cu-graphite composite are found in case of those areas where highly conductive material is required as well as lubrication phenomenon. Both of these requirements can be fulfilled by Cu-Graphite composite. The main application of these composites are in electrical appliances such as electrical brushes in motors and generators, where sliding contact comes into play. Because of excellent properties of graphite to increase the wear resistance and coefficient of friction due to self-lubrication, it's generally employed as a reinforced component into matrices. The wear rate of the sliding component is influenced by many factors, such as temperature, pressure, sliding speed, and material properties. So the accelerated life testing (ALT) was carried out to analyse the wear properties of the material.

The wear testing of composite was carried out using pin-on-disc tribometer with varying temperature and pressure at different sliding velocities. In this study, copper-10vol% graphite composite was prepared by powder metallurgy, which is a good one combination for better sliding properties. The mixing powders were blended for 2 hours in electrical agate pestle mortar with the speed of 20 rpm. Before the compaction, powders were preheated at a temperature of 150^{o}C to evaporate the volatile constituents. Sintering in the form of cylindrical components was carried out by microwave processing after compaction with hydraulic press and furnace cooling was adopted. Hardness of the composites came out to be 98 HV, which was higher than conventionally prepared composite. After wear testing, it was analysed that temperature and pressure affects the wear rate of the composite upto an extent. The most outstanding feature of the result outcome was that the microwave sintered composite exhibited 3 ½ times better mean life than electro-graphite sliding contact material.

Agarwal (1998) [26] developed a sintering of ceramics and W/Cu composites using microwave oven. The use microwave energy was progressively being explored to create better and cheaper products mainly ceramics at that time and sintering was limited upto ceramics. During this research it was proved experimentally that in the case of W/Cu composite, the cycle time was reduced to 1/10th using microwave energy compared to conventional process and resulted in better properties. Sintering carried out through microwave processing leads to reduction in sintering time, sintering temperature and total cycle time. The mechanical properties such as density, average grain size, and hardness were improved. The microwave coupling results extremely rapid reaction in the occurrence of a defect and this reaction results in new formed materials at much lower temperatures than that of conventional heating process. It has been anticipated that with above mentioned significant advances in the field of microwave processing of ceramics, there is a great future of microwave technology for fruitful commercialization for specialty ceramics.

Agrawal (2006) [27] represented the metal processing such as sintering, brazing and melting of metals through microwave energy. In this study, an importance of microwave processing over the conventional one and the common advantages were highlighted such as very short cycle time (resulting into energy savings upto 90%) over conventional methods, rapid heating rates, finer microstructures which directly leads to improved mechanical properties. In this research, microwave energy was utilised for brazing of bulk metal pieces, super-alloy based turbine blades which are very difficult to join through conventional processes. Joining of cast iron and stainless steel was carried out with the help of braze powder. Sintering of many of the commercial used powders have been successfully sintered using microwave processing such as Al, Cu, Ni, W, WC etc. upto near full densification. The microwave sintering of various metals and metal alloys including ceramics have been sintered in very short period of time (75-90% less power consumption than conventional processes) with having high mechanical properties like modulus of rupture (MOR) and hardness achieved was higher than that of conventional ones.

Gupta and Wong (2005) [28] used microwave hybrid heating to sinter the metallic materials and improved the overall mechanical properties of the manufactured product. Al and Mg and lead free solder were chosen to carry out the sintering process. The sintering process was carried out by using domestic microwave oven with 2.45 GHz frequency and 900 W power level. SiC was used a susceptor for enable coupling between microwave and the material. The sintering was carried out through microwave hybrid heating (MHH) because microwave heating has a feature to heat the material from inside to outside and on the other hand susceptor (SiC) supported the sintering from outwards to inwards. The sintering of all three selected materials was successfully carried out using microwave heating. A better combination of tensile properties was obtained in microwave sintering as a result of the reduced level of high temperature thermal exposure.

Wong and Gupta (2007) [29] carried out the sintering of Mg/Cu Nano-composites through microwave rapid heating. In this research, powder metallurgy process including microwave hybrid heating was used to prepare magnesium composites with varying amount of Nano sized Cu particulates. The sintered specimen was hot extruded and characterization was carried out in terms of microstructural as well as mechanical properties. The process was carried out by using microwave energy with 2.45 GHz frequency and 900W power level. SiC was used a susceptor, so that microwaves can couple with the material. Microstructure of prepared composite revealed minimum porosity because microwave processing has a capability to manufacture the products with high density. The Nano sized Copper particles were uniformly distributed over matrix. Cu particulates lead to an increase in hardness, 0.2%yield strength, elastic modulus, ultimate tensile strength and work fracture of matrix. Tensile properties were increased using microwave energy.

Upadhyaya et al. (2006) [30] carried out the sintering using W – Ni – Fe alloy through microwave processing. In this research, the composite was sintered and the effect of heating mode on the microstructure and mechanical properties of composite (92.5W–6.4Ni–1.1Fe) was studied and compared with conventional process. The compacts were sintered using microwave oven having frequency of 2.45 GHz and conventional furnace at 1500°C and in 2.45 GHz. Microwave sintered composite took about 75% less time than conventionally processed composite, which corresponds to less W coarsening. The hardness of sintered composite was carried out by Vickers micro-hardness tester. XRD was used for phase determination. From the experimental results, it was observed that micro-hardness hardness and tensile properties of microwave sintered composite was better than that of conventional ones. The mechanical and microstructural properties of microwave sintered alloys were higher than conventional sintered composites.

Rajkumar and Aravindan (2009) [31] have successfully sintered copper-graphite composites through microwave processing route. These metal matrix composites are increasingly being used in tribological applications because of their higher wear resistance. Microwave processing is a novel technique for synthesis of metals when compared with conventional heating appliances. For the experimentation, copper and graphite powders were mixed together thoroughly. An industrial microwave having frequency of 2.45 GHz was used for processing.

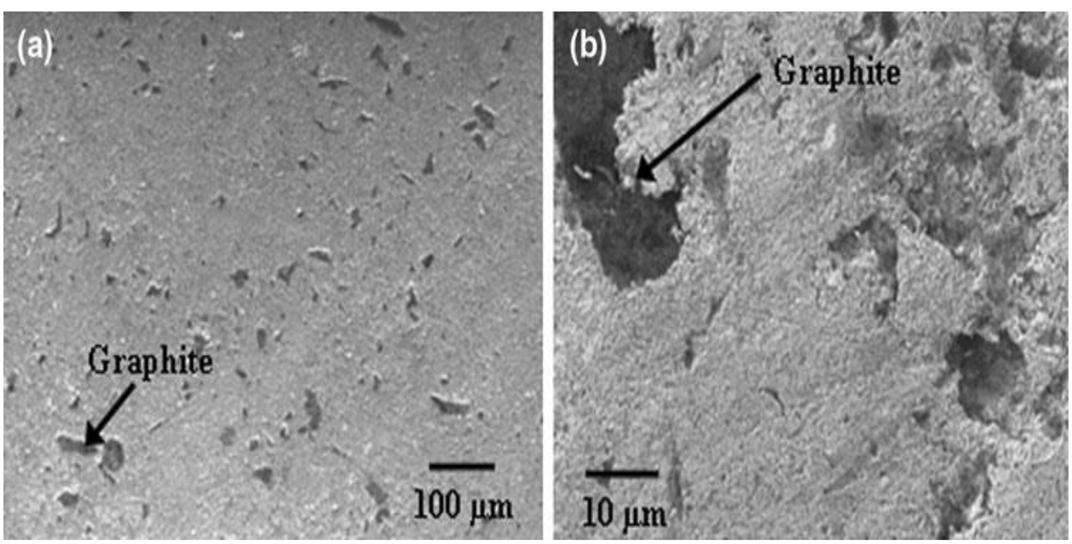


Figure 2.5: SEM image of sintered composites through microwave processing [31]

Hybrid setup was formed in such a manner that it was having two layers of elements such as first one was transparent to microwave and the second one was the absorber of the microwaves, known as susceptor. The micro-hardness and microstructure of developed samples were analyzed.There was a significant improvement in the micro-hardness of microwave processed (sintered) copper – graphite composite in different volume fractions and the developed samples were free from cracks. The finer microstructure with moderate porosity level caused due to microwave heating increases the performance of the composite.

Rajkumar et al. (2013) [32] reported the work on the composites of Cu-nanographite manufactured through microwave processing. In this work, the properties of cu-graphite composites which they delivers, the effect of particle size, loading conditions and effect of sliding speed on the wear performance were highlighted. The composites were sintered through microwave processing. The copper-graphite composites are mainly used in electrical motors/generators, bush and bearings in industries. So these materials should have self-lubrication property so that sliding wear can be minimised upto an extent. Gibson et.al have reported that improvement of wear-resistance in case of aluminium can be done by having an addition of 2% graphite in it, but higher graphite content upto 8% graphite weakens the alloy by increasing its softness and which corresponds to higher wear rate. In this study, pure copper as a matrix material (99.98%) was used as a matrix material and graphite in the form of nano-graphite as a reinforcement material was selected. The composites were manufactured through powder metallurgy by varying graphite content (5%, 10%, 15% and 20%) vol. of graphite. Homogeneous distribution of nano-graphite in copper matrix reveals very less porosity, high density, high hardness and very high electrical conductivity Cu-nanographite 5 vol% composite showed a high value of hardness and highest thermal conductivity when it was compared with the other varying graphite proportion of composites.

Mondal et al. (2010) [33] carried out the sintering of refractory metals/alloys through microwave processing. Refractory metal/alloys are well known because of their mechanical properties but their melting temperature is very high. Because of refractoriness, it is very difficult task to process these metals/alloys, first of all the problem comes to achieve the temperature in conventional furnaces under moderate conditions. During this research, refractory materials such as W, Mo, W-Cu, W-Ni-Cu, W-Ni-Fe refractory materials having wide range of temperature difference between each other were taken for the analysis. Tungsten (melting temperature 3420°C) and Mo (melting temperature 2620°C) was sintered in microwaves for 30 minutes in H_2 atmosphere. The composites W-30 Cu alloys, W-7Ni-3Cu alloys, W- Ni-Fe alloys were sintered for 30 minutes in H_2 atmosphere. Microwave sintering of these metals delivers about 80% reduction in total processing time. Finer grain size and better mechanical properties were also achieved through microwave sintering which can be considered as an additional advantage which increased the performance of materials.

Rajkumar and Aravindan (2011) [34] performed an experiment for copper - carbon nanotubes composite through microwave sintering and evaluated the tribological behavior of composites. Copper coated carbon nano tubes with 5-20 vol% with copper powder were mixed thoroughly and sintering was carried out using microwave heating. Electrolytic copper powder was used as a matrix and multiwall carbon nano tubes were used a reinforcement. Hybrid heating setup was to process the material inside the microwave furnace with 2.45 GHz and 3.2 KW. Developed samples were characterized using XRD, SEM and SEM-EDS. Pin on disc setup was used to evaluate the tribological performance of the copper-carbon nano tubes composites under dry conditions. The percentage increase in volume of carbon nano tubes (CNT) helps to reduce the coefficient of friction and wear rate of composites. Self-lubrication property of carbon film was accounted for reduction in coefficient of friction, which provides the property of higher wear resistance to the sintered composites.

Chandrasekaran *et al.* (2011) [35] did an experimentation to melt the metals through microwave processing. It was the first time in microwave material processing, when the melting of metals was carried out through microwave oven/furnace. Melting metals through conventional furnaces leads to very high amount of energy as well as time consumption. First of all, its very time consuming process to achieve the required temperature for melting metals in conventional furnaces, and there is a possibility of material and energy losses. In conventional furnaces some safety risks are also associated with it. In order to overcome the limitations associated with the conventional melting, some advanced melting technologies came into existence such as plasma melting, electron beam melting, infra-red melting, microwave melting, solar melting etc. are preferred over the conventional ones because of their high quality production for some special application and requirements. Microwave heating is little bit more attentive because of its major advantages such as high heating rates due to hybrid heating concept, which directly corresponds to optimize the processing time, less consumption of power and less environmental hazards. Microwaves can't deal properly with metals and cause sparking when comes in contact with the metallic materials. The microwaves are reflected by the metals, because their skin depth is of few microns. Microwave melting of low temperature materials such as lead, tin, aluminium and copper was carried out with the aid of susceptors. Aluminum and copper samples were melted in the atmosphere of argon/nitrogen (inert gas) to minimize the risk of oxidation. On comparing it with the melting through the conventional furnace, microwave melting was found to be twice faster, which directly leads to less energy consumption and less time consumption.

2.6 Conventional Casting Methods

Conventional casting methods which are used for the fabrication of metal matrix composite includes friction stir processing, squeeze casting, die casting, centrifugal casting, powder metallurgy, stir casting etc. Various methods have additional advantages and some limitations, which are highlighted by various researchers, and are explained below;

Kang *et al.* (1996) [36] fabricated the metal matrix composites through die-casting and evaluated the mechanical properties of composites. A slurry was prepared, which contains alumina fibres with silica binder and impeller was used to ensure the proper mixing of the ingredients. The main processing parameters of the fabrication were the stirring time, amount of binder and amount of water etc. should be taken care of while preparing composites through this technology. A conventional aluminium alloy was used as a matrix material. Injection velocities having range from 0.1 to 3.6 m/s were used to fabricate Al_20_3-short-fiber reinforced MMCs. The preheated preform was settled in the preheated die cavity and molten metal was poured into the mould. After closing the dies, the pressure was applied until the complete solidification took place. Finally, the MMC part was ejected. To study the metallurgical properties of the composites, the samples were polished through automatic polisher. Metallographic preparation of composite samples was achieved by polishing on an automatic polisher. Microstructural analysis of the composites were carried out by scanning electron microscope. It was observed from the experiments that with decrease in injection velocity, the infiltration length of the composite was increased. The actual pressure as compared to the applied pressure was very small. From the result point of view, the conclusion came out that injection velocity is the main parameter to control the infiltration length. With increase in fibre length, the physical and mechanical properties also got increased, such as tensile strength, elastic modulus and harness of the composite.

Beffort *et al.* (2006) [37] represented the microstructure and mechanical properties of SiC reinforced aluminium composites by squeeze casting process and studied its alloying effects. In this processes, the matrix material was used as Aluminium alloy (99.90%) and Silicon Carbide (SiC) was used as reinforcement material. The steel castings were used to pack SiC particles, with graphite based coating and preheating was carried out at 750°C and were mixed with the melted matrix material. After addition, the pressure of 1400 bar was applied for two minutes. The microstructure was analysed by using optical microscopy, SEM, EDS etc. The result showed that the hardness factor was increase up to 10-20% and bending strength was achieved upto 700 MPa along with elastic modulus 200 GPa. Which states that percentage of SiC content should be varied as per the constituents of the alloying elements and the required mechanical properties of the MMC's**.**

Akbari *et al.* (2013) [38] performed an experiment to manufacture aluminium composite reinforced with Al_2O_3 and evaluated its properties. A356 alloy was used as matrix material and Al_2O_3 nanoparticles as reinforcement material. A resistance furnace with a graphite stirrer was used in resistance furnace to mix the molten aluminium alloy with the Al_2O_3 powder wrapped in aluminium foils. The prepared slurry was stirred for 12 min, having temperature 850^{o}C at 450 rpm and then it was poured into cast iron moulds and cylindrical shaped composites were carried out after solidification. The scanning electron microscope (SEM) was used to analyse the microstructural characterization. In results, SEM micrographs showed uniformly distributed reinforcement of Al_2O_3 throughout the matrix. The mechanical properties such as hardness, tensile and compressive strength decreased with the increase in milling time and the porosity of the composite increased proportionally.

Huang *et al.* (2011) [39] conducted an experiment to manufacture aluminium alloy pistons using SiC as reinforcement material through centrifugal casting. An aluminium alloy was used as a matrix material and the reinforcement of SiC particles with the diameters of 15 and 30µm. The same proportion of the SiC particles having 50% each was used for both the different sizes of particle compositions. The melting of Al–Si alloy was carried out in a graphite crucible. After preparation of melt, a stirrer was introduced into the molten metal and then it was mechanically agitated. The slurry prepared was then poured into the rotating mould of the vertical centrifugal machine consisting of iron mould with two parts and finally, the Al alloy-based composite pistons were fabricated by centrifugal casting reinforced with SiC particles. These type of pistons carries huge amount of SiC particles, where the most required properties are such as hardness and wear resistance etc. Three different zones exists in the piston from piston head to the piston skirt. The hardness values along the axis of the piston gradually decrease from the head to the skirt, which leads to the change of structure throughout the length of the piston. The pistons were successfully fabricated through centrifugal casting having a temperature of the slurry 850˚C and temperature of mold 600˚C. The rotational speed of the mold was 800 rpm, possesses the highest hardness value, i.e., 93.9 HRB respectively.

The results showed that with the increase in nano CuO content, Micro-hardness of the samples increased. The maximum hardness is observed for the sample with 2 wt% nano CuO. The mechanical properties of the composites improved with increase in weight% of nano Cuo particles.

Abdizadeh *et al.* (2014) [40] conducted a study to evaluate the mechanical properties and microstructure of Al composites reinforced with nano MgO fabricated through stir casting process and powder metallurgy methods. During this experimentation, A356 (Al alloy) was used as the matrix material and MgO nanoparticles as the reinforcement. The fabrication steps in the form of detailed information, for both stir casting and powder metallurgy methods, have been shown in Fig. 2.6 in the form of flowchart.

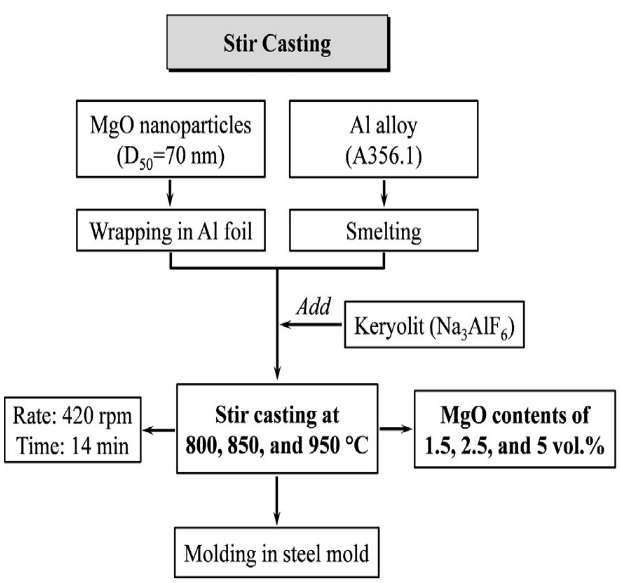


Figure 2.6: Flowchart representation of stir casting and powder metallurgy methods [30]

In stir casting, Al was melted and MgO nanoparticles (which were wrapped in Al foil) added to it and the process was carried out using three different casting temperatures of 800, 850, and 950^{o}C. On the other hand, in powder metallurgy route, Al powder alloy was directly mixed with MgO nanoparticles, and then conventional powder metallurgy was employed to produce the samples. Finally, the composites were sintered at different temperatures of 575, 600, and 625^{o}C. Various proportions of MgO contents having 1.5, 2.5, and 5 vol. % have been considered for each processing temperatures. The crystal structure and phase investigation was performed using x-ray diffraction. Density measurement revealed that the density of stir casted composites was higher than the sintered composites. The reason behind the densification parameter was the formation of micro-pores and cracks in sintering method which increase via increasing of the MgO contents. The optimum processing temperatures to achieve better mechanical properties were 625^{o}C for powder metallurgy and 850^{o}C for stir-casting respectively.

Kovacik et al. (2008) [41] did a study to friction coefficient of composites carrying copper as a matrix and graphite as reinforcing agent by varying the compositional percentage of graphite. The hot-isostatic pressing process was used to prepare the composite and % age of graphite was varied from 0-50 vol%. On the second hand, copper coated graphite having 30-50 vol% of graphite was used. With increasing concentration of graphite in copper, first wear rate of coated and uncoated samples at first decreases and the coefficient of friction of composite becomes independent on the composites, when it reaches the threshold value. Copper powder shows a dendritic shape and graphite has a flake shape in SEM images. The result showed the threshold value for very fine graphite powder (16μm) was 12 Vol% and for coarse powder it was 23 vol%.

Ramnath *et al.* (2014) [42] conducted an experiment for Al-Al_2O_3-B_4C and evaluated the mechanical properties of the prepared composites. In this work for preparing metal-matrix composite, aluminum alloy was used as base material, and as a reinforcing material alumina and boron carbide in powder form are used. The prepared mixture was filled into the die and solidification was carried out. The first process in the experiment was preheating. In which, the empty crucible and the reinforcement powders, namely boron carbide and alumina powders are heated separately. The melting of the aluminum alloy (95%) was carried out in the graphite crucible inside the furnace. Then, the crucible with aluminum alloy was heated to 830^{o}C while the preheated powders are mechanically mixed with each other below their melting points. This metal matrix is then kept into the furnace at the same temperature. This experiment was repeatedly done by varying the compositions of the composite powder. In this paper three samples were used with varying percentage as shown below in the form of Sample1, 2, and 3 as shown below.

Sample 1 carrying aluminum alloy–95%, alumina–3% and boron carbide–2%,

Sample 2 carrying aluminum alloy–95%, alumina–2% and boron carbide–3% and

Sample 3 carrying: aluminum alloy only.

After the preparation of MMCs the following tests were conducted on the specimens, which includes tensile test, flexural test, impact test etc.

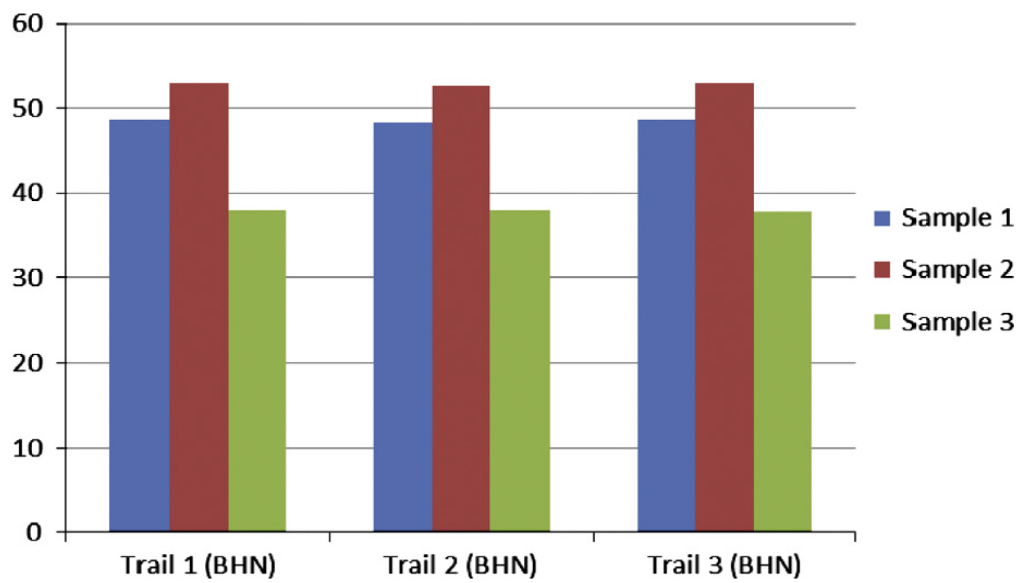


Figure 2.7: Comparative analysis of hardness with varying compositions

The tensile strength of sample 3 was obtained marginally higher than other two samples because of its aluminum content. But, the sample 1 has higher tensile strength (54.60 MPa) than sample 2 (51.75 MPa). Also, the Brinell hardness of sample 1 (48.53) is marginally lower than that of sample 2 (52.80) but higher than that of sample 3 (37.83).

Kumar *et al.* (2013) [43] fabricated metal matrix composites of A359/Al_2O_3 through using electromagnetic stir casting process. An alloy of aluminum (A359) was used as the matrix material and reinforced with varying %age of Al_2O_3 having 2wt%, 4wt%, 6wt%, 8wt% of average 30μm powder size for the fabrication of the composites. The muffle furnace was used to carry out the melting at temperature of 730±20°C. After addition of the preheated Al2O3 particles, the melt was electromagnetically stirred. The processing of the composite was carried out at a temperature of 750°C with a stirring speed of 300 rpm. Experiments were carried out for a wide range of particle weight percentage varying from 2% to 8% in steps of varying the composition by 2% of reinforcement. Finally, the mechanical properties A359–Al_2O_3 composites are compared with the unreinforced A359 matrix alloy. The microstructural characteristics and hardness of the composites were evaluated.

Zhan et al. (2006) [44] did an experiment to find out the significance of graphite particle in the wear of hybrid composites. In this study, powder metallurgy method was used to fabricate the composites by using copper as matrix material and SiC and Gr using reinforcing agents. The main use of copper matrix composites are in those applications, in which high electrical and thermal conductivity with having better corrosion and wear resistance is needed. Major elements used to increase the mechanical properties of the matrix material (copper) are ceramics the ceramics i.e. SiC, Al_2O_3, TiC, WC etc. Previous work in this field revealed that graphite content helps to improve the wear resistance at room temperature as well as at high temperature with having high conductivity. During experimentation, two compositions were selected as Cu-SiC 15 vol% and second combination with Cu-SiC 15vol%-Gr 8 vol% using powder metallurgy with hot pressing method. The result showed that with addition of Gr as second reinforcement helped to decrease the wear rate of the composite. The friction coefficient of copper hybrid composite showed more stability and was lesser than SiC/Cu composite. Surface roughness of SiC+Gr/Cu was far-far less than SiC/Cu composite.

Wang et al. (2013) [45] experimentally tried to find out the bending and tribological properties of Copper-Graphite composites, by using expanded graphite through ball milling. The result showed that with decreasing the particle size coefficient of decreases and increasing graphite content until the condition of threshold is achieved. On the other hand, bending strength of composite decreases with increasing amount of graphite. So, to balance bending strength and friction coefficient, many of the different techniques were used by different authors to prepare these MMCs with retaining its properties. Rajkumar et al. used copper coated graphite powders having advantage over uncoated graphite, but it's very difficult to coat very small size particles, when the point comes for the mass production. In the study, expanded graphite (EG) was the new invention with a new type of carbon material, usually fabricated by rapid heating of graphite to a high temperature. During this study, composites were fabricated through cold compression moulding and sintering by using electrolytic copper powder and ball milled expanded graphite (EG) as reinforcing constituent. Result showed that bending strength decreases significantly at 3wt% having 226.31 MPa to 85.61 MPa having 5wt% of EG.

2.7 Summary of the Literature

The applications of microwave energy in various fields of engineering are increasing day by day due to significant improvements in mechanical properties, reduced processing cost and times. The concept of microwave hybrid heating has explored its potential upto the mark of industries. The lower energy consumption with higher rate of heat generation due to volumetric heating accelerated the research in this field of processing materials. The most significant feature of selective heating is one of the main reason behind its main use for metals processing. First of all, to understand the concept of microwave processing, various manufacturing processes being done through this technology were studied i.e. sintering, cladding, joining and melting of metal powders etc. Microwave hybrid heating resulted in improved properties than conventional methods were reported due to formation of new stronger phases and uniform grain growth during MW processing.

The various methods were used to prepare the MMCs with the objective to get more and more improvements in their mechanical properties and their comparison with other methods of manufacturing the same. Effect of the reinforced materials with their varying percentage were studied. The change in reinforcement not just affected the properties but it also changed the microstructure and grain growth of the composites. For the manufacturing of MMCs, the conventional processes such as die casting, stir casting, friction stir processing and common methods to prepare it were studied. After summing up, it was concluded that all these properties can further be enhanced by fabricating the MMCs through microwave hybrid heating.

Chapter3

RESEARCH GAP AND PROBLEM FORMULATION

This chapter illustrates the basic idea that how the concept generated to fabricate the composite castings through microwave processing, various gaps found in the field of microwave processing and its limited use in material processing. By keeping all the things into mind, the problem was formulated and plan was proposed that how the things will be carried out.

3.1 Gaps in Literature

Initially the main use of microwave energy was noticed for the communication purpose. Afterwards, this technology was proposed for heating purpose in food processing. With the development of technology its heating capability was used for the processing of polymers, ceramics, minerals, inorganic materials etc. But the main limitation of microwave radiations at the room temperature can't interact with the metals, so it's very difficult to heat metals with this technology. Hence, research during last two decades revealed that microwaves can couple with metal by an intervening medium called susceptor and separator. With the help of this approach various manufacturing processes like sintering, cladding, joining and melting of metal powders have been carried out using microwave technology. Microwave processed materials revealed remarkably excellent properties and remarkable reduction in processing time but a very limited research has been noticed in the field of casting and melting of powders, so lot of scope is still left in the field of casting and melting. In the other metal processing operations carried out through this technology, the mechanical properties, microstructural properties and tribological properties were improved as compared to the products compared with conventional processing. All these properties are desired in metal casting also and to make casting process as a green manufacturing system with reduced energy consumption, the decision was taken to cast it through microwave hybrid heating.

3.2 Limitations in the existing literature

Lots of development have taken place in the area of manufacturing, with the development in technology, but no one has enlightened on these following parameters while manufacturing the MMCs, these parameters are;

- Which one is the best method to manufacture the MMCs, with maintaining its mechanical properties (strength, hardness, wear resistance etc)
- Based on energy consumption, which method is best and what are the initial requirements of the processes on the basis of its common use.
- In all the manufacturing processes, the concept of volumetric heating or melting is not introduced, which is one of its main limitations.
- All of the manufacturing processes require a separate furnace for melting and premixing of constituents, which adversely affects the cost of production.
- Most of the metal matrix composite (MMC) casting processes are very time consuming methods. In every casting processes there is a need of 8-10 hrs. Melting procedure for the base metal and then post-curing etc., which leads to a very time consuming process.

In case of microwave processing, the lots of developments have taken place. For example, in case of microwave sintering, brazing and joining, surface treatments, microwave cladding, etc. But no work has done yet in the field of casting through microwave**.** Various field of microwave processing for manufacturing are shown in the fig below.

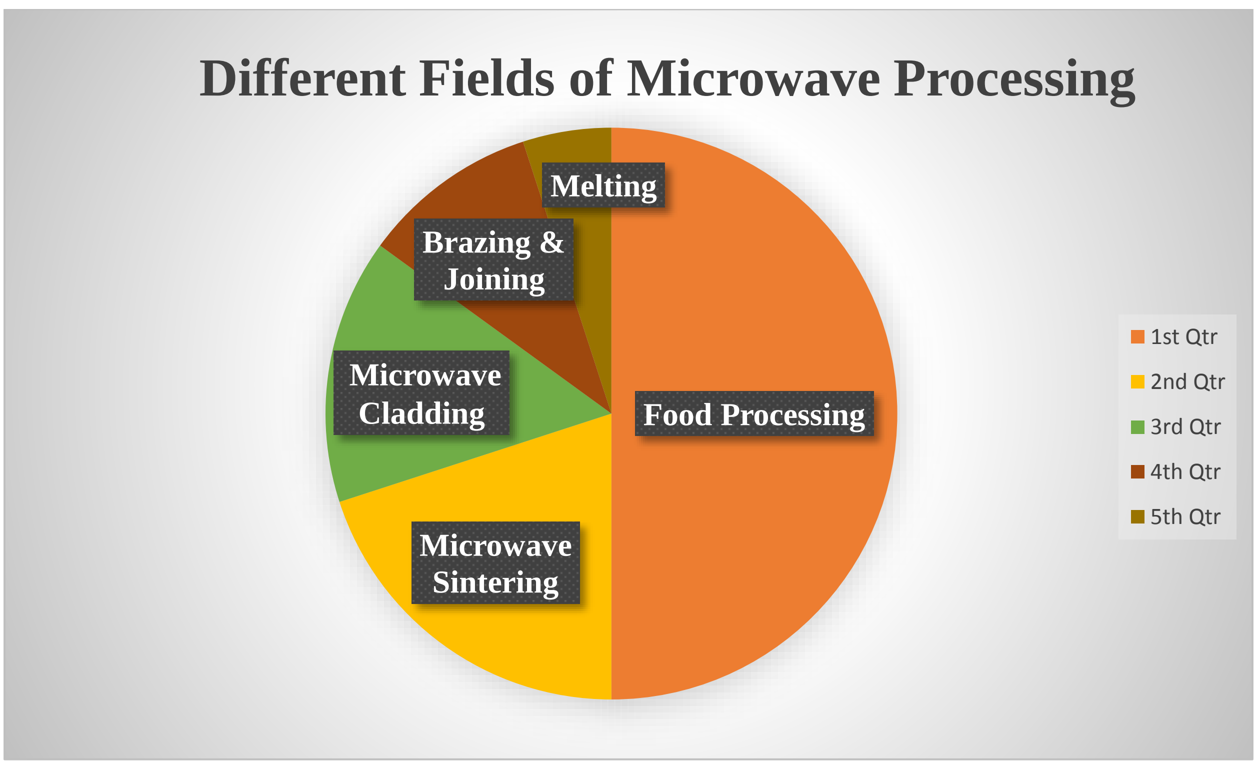


Figure 3.1: Wheel depicting different fields of microwave processing

3.3 Problem Formulation

In the age of cut throat competitions in the field of manufacturing, designing specific materials for desired application is not a cost effective solution in present scenario. The main objective of the present research is to develop a new cost effective as well as green technique for producing metal matrix composite using a non-conventional source of energy (microwave) which offer variety of advantages as compared to the conventional ones. The microwave as a heating source can be a substitute for the existing sources and can overcome those limitations. The main characteristics of microwave heating includes;

(1) Radiation depth of penetration, (2) rapid and volumetric heating, (3) hybrid heating, (4) selective heating of materials.

These inherent characteristics of microwave heating provides more rapid and uniform heating in a green environment and offers better product quality. The manufactured products through this technology have unique microstructures, better mechanical properties and additional advantages from economic point of view such as reduction in processing times which also corresponds to reduced manufacturing cost. However, heating metals and subsequently cladding using microwave is a challenging task due to poor penetration depth of microwave at common frequency 2.45 GHz at room temperature.

3.4 Objectives of the Research

After going through the literature in various fields of microwave processing by different authors, research gaps were formulated. The main objective of every research is to minimize the limitations of the existing processing upto an extent and to enhance the required properties. The main objective of the present research are;

1. To fabricate a metal-matrix composite casting through microwave heating.
2. To carry out the microstructural and metallurgical characterizations of microwave developed composites castings using SEM (scanning electron microscopy), EDS (energy dispersive X-ray spectroscopy), Optical microscope and XRD (X-ray diffraction) techniques.
3. To study the mechanical properties of casts object in terms of Vicker’s micro-hardness and dry sliding wear behavior using pin-on-disc tribometer.
4. To study the fractographic analysis of the worn-out samples, fractured area and to understand the wear behavior with and without the reinforcement.

3.5 Plan of the Present Work

The work to be carried out is subdivided into two phases such as phase 1 and phase 2.

Phase 1--- During the phase 1, proper selection of the matrix material and reinforcement will be carried out and the selected material will be used to cast the metal matrix composites through microwave processing.

Phase 2---During the phase 2, characterization of the composite in terms of mechanical as well as metallurgical will be carried out.

The proposed plan of the present work is illustrated in the flow chart as shown in Fig. 3.2.

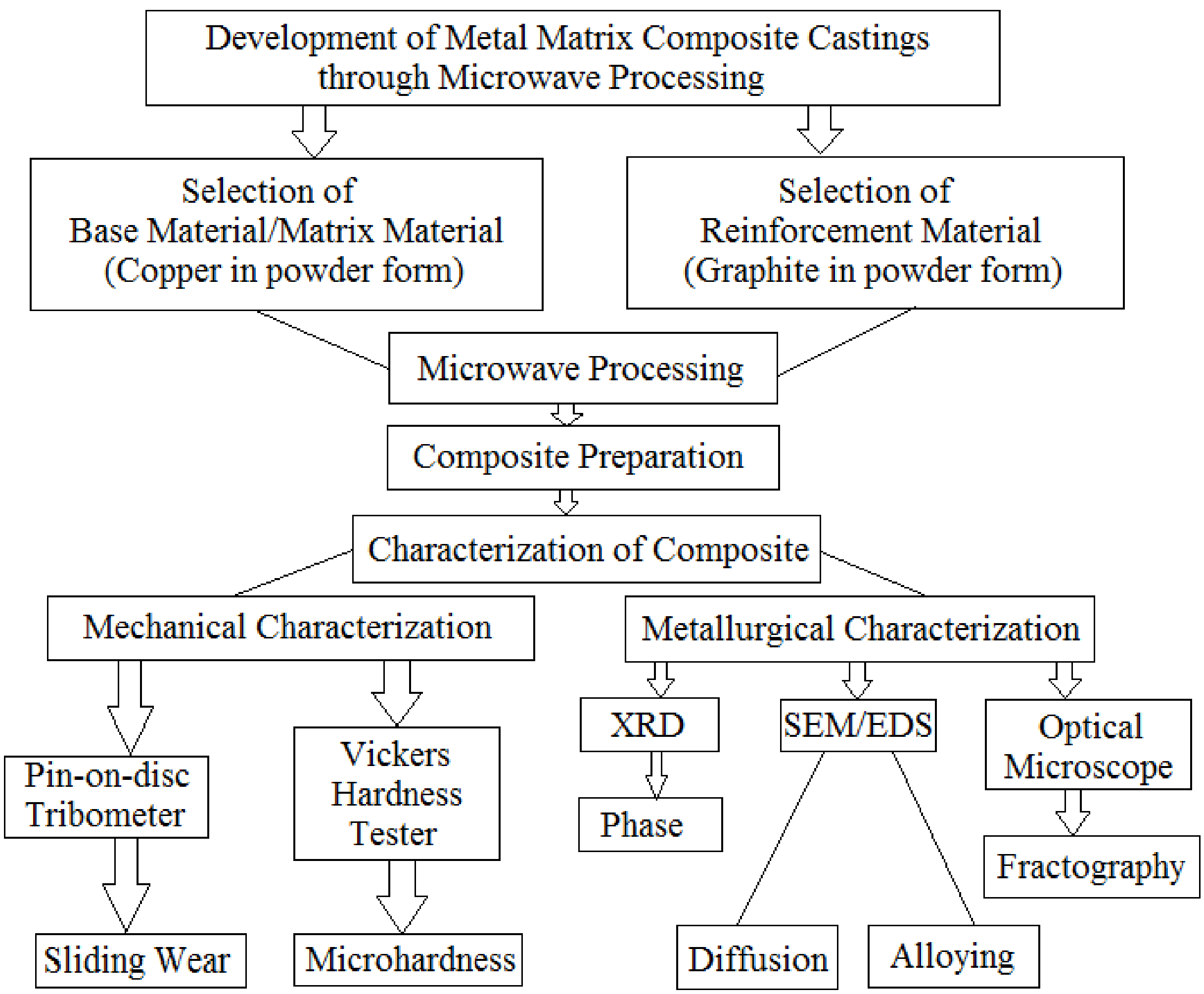


Figure 3.2: Flow diagram showing the plan of the present work.

Chapter 4

EXPERIMENTAL PROCEDURE

The development of metal matrix composites is a challenging task by using the microwave radiations. The main reason behind this challenge is the poor coupling between microwaves and metals and very limited amount of research has been carried out in case of melting of metallic materials. In the present work, wear resistant composite castings having copper with varying percentage of graphite reinforcement have been developed by using domestic microwave oven of frequency of 2.45 GHz has been used as a heating source. The following sections briefly describes the procedure for development and characterization of composites.

4.1 Material selection and its applications

4.1.1 Matrix material: To cast the metal matrix composite castings, copper was used as a matrix material. The main reason behind its selection as matrix material was it's used in electronics industry and electrical appliances.

4.1.2 Reinforcing material: Graphite was used as a reinforcement material because of its properties. Graphite acts as a solid lubricant between the mating surfaces hence due to this outstanding property, it's used with those matrix materials which have to be in contact regularly with the sliding parts or have to slide its own.

4.1.3 Applications of copper with different reinforcements

Table 4.1: Various applications of copper base composite materials

Matrix material	Reinforcing material	Application in different fields
Copper (Cu)	Tungsten (W)	Electrical contacts, Electrodes of resistance welding.
Copper (Cu)	Graphite (Gr)	Electrical brushes, Sliding contacts, Generator bushes.
Copper (Cu)	Titanium Carbide (TiC)	Electrodes of resistance welding.
Copper (Cu)	Cobalt and other agents.	Sliding rings, Composite materials on walls of nuclear reactors, Electronics industries etc.

4.2 Synthesis of Copper-graphite Metal Matrix Composite Castings

The copper-graphite metal matrix composite castings were fabricated through microwave hybrid heating. MMCs were prepared with varying the percentage of graphite (reinforcing material) into the copper used as matrix (base material). To study the difference between the pure copper casting and reinforced graphite composite castings, the difference in their microstructures and mechanical properties of the fabricated castings were studied.

4.2.1 Microwave Hybrid Heating and Selected Parameters

Prior to the microwave heating, the material selection part was done, in which three compositions were decided to cast through microwave processing. Pure copper, Cu-2 wt. % graphite and Cu-5 wt. % was selected as a composite materials to cast. To cast the metal matrix composites, the powders were preheated at 120°C in the graphite crucible for 360 seconds through the convection mode of heat transfer and then allowed to stay at room temperature for a specified period of time. After preheating, samples were premixed in finite quantity as per the material decided in the test tube and after mixing, the powders were poured in the graphite cavity. The cavity was placed on the refractory brick and it was covered with the graphite sheet used as a separator and after that the complete experiment was covered with the susceptor material as shown in the Fig. 4.1.

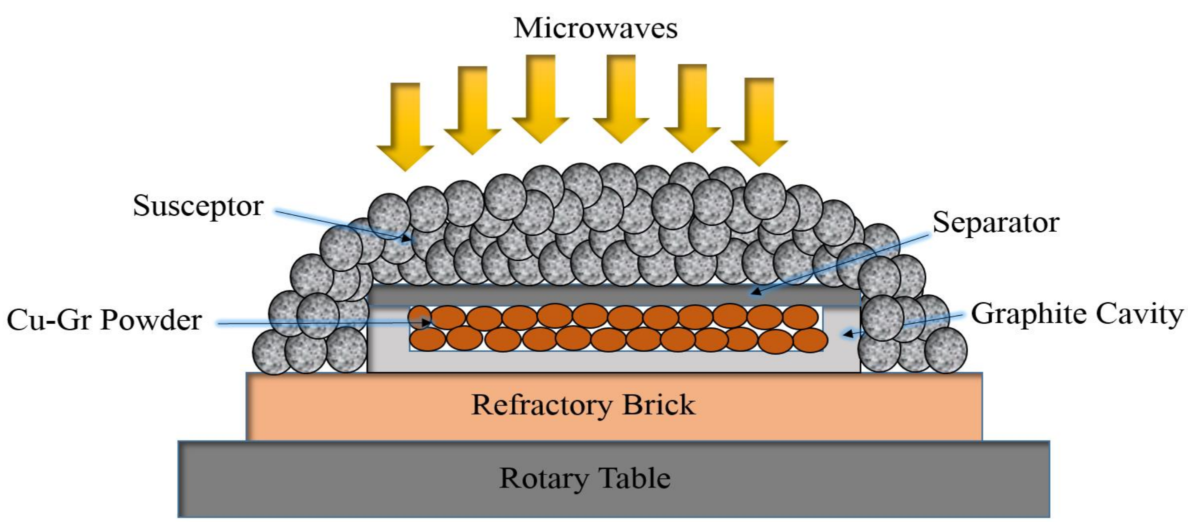


Figure 4.1 Experimental Setup to Cast metal matrix composites through microwave processing

After the experimental setup of the casting equipment, the setup was exposed to microwave heating having frequency 2.45 GHz and power rating 900 watts. The average time taken to melt the casted composite was around 900 seconds and after this the setup was allowed to cool at the room temperature. The setup material and requirements during experimentation are shown below in the Table 4.2.

Table 4.2: Various Microwave Processing Parameters and Material Requirements

Process Parameters	Descriptions
Microwave applicator	Domestic multimode microwave (Made: LG, Model: Charcoal)
Working frequency and maximum power rating	2.45 GHz and 900 watts
Exposure time	Preheating (Convection)= 360 seconds Microwave Heating= 900 seconds
Work-piece material	Copper as matrix Graphite as reinforcing agent
Powder Size	Copper powder (Average particle size 40 µm)
Susceptor material	Fine grained charcoal powder
Separator material	99.9% pure thin graphite sheet

4.3 Characterization of Casted Composites (MMCs)

Composites were washed fully with acetone before carrying out the characterization. The low speed diamond cutter was used to cut the composites cross-sectionally. The samples were first polished by emery papers and after polishing, composites then exposed under a jet of water and then after it was exposed to a blower to soak the water particles.

4.3.1 Diamond Cutter

A diamond cutter is basically a blade on which diamonds in the form of abrasives are fixed on the surface of the cutting edge. It’s basically a cutting in the form of grinding because a blade cuts the material through the abrasives mounted on the cutting periphery. It cuts the material very slowly but the quality of the product based on the surface finish is very high. There are different types of diamond blades for different applications i.e. for cutting stones, concrete/ glasses or ceramics at the industrial level but here it was used for metal cutting. It is the product of Ducom (material characterization systems) and named as low speed saw unit.

Figure 4.2 Low speed diamond cutter (Courtesy: Fluid machinery lab, Thapar university)

4.3.2 Polishing machine

The first step is to polish the samples thoroughly with low grades (coarse) of emery papers such as 180, 220, 320 and 600. After polishing it with coarse grades, the samples were then polished on a rotating wheel by mounting the emery papers of fine grades on it such as 1000X, 2000X, 3000X and 4000X and a high quality of surface finish was obtained. On the other hand, second wheel is covered with a soft cloth (velvet), on which the sample was polished with diamond paste (1µm alumina paste) used with spray to give it super high (mirror like) and scratch free finish for characterization. After polishing, the samples were washed thoroughly under the jet of water and were dried under the jet of water to soak the water particles completely.

Figure 4.3: Polishing Machine (Courtesy: CNC Lab, Thapar University)

4.3.3 Hardness Testing Machine

The micro-hardness of the casted composite samples was evaluated using a Vicker's micro-hardness tester. The samples after cutting and polishing were prepared for evaluating the micro-hardness. In Vicker's micro-hardness test procedure, an indenter creates the indentation at the variable loads. By measuring the indentation the hardness value is calculated. After the indentation is completed, the two diagonals are measured and the average value is considered. The indenter used for indentation can be used for all materials irrespective of hardness of materials.

Figure 4.4: Vicker's Micro-hardness Tester (Courtesy: A.M. Lab, Thapar University)

4.3.4 Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS)

The analysis of microstructures and chemical compositions are generally carried out by using Scanning electron microscope and energy dispersive X-ray spectroscopy. The scanning electron microscopy (SEM) is used to scan the surface area and is used to determine the microstructures of samples at higher magnifications which uses an electron beam to scan the sample surface. The electrons interact with the atoms of specimen and produce signals that contain information about the microstructural analysis produce images. It can produce very high-resolution images of the specimens surface, revealing details less than one nano meter in size. SEM as shown in Fig. 4.5. (Make: JEOL JSM-6510LV, Oxford Instruments) is available at ***SAI Labs, Thapar Technology***

Campus, Patiala is used for analysis. It is a high-performance and low vacuum SEM for fast characterization and imaging of fine structures having magnification from 5X to 300,000X. Generally EDS is also attached with SEM, because it tells about the elemental composition peaks for that microstructural images. EDS provides the quantitative analysis of its elemental compositional % age having a depth upto 1-2 microns and data is represented in the form of peaks and distribution of reinforcement at various points.

Figure 4.5: Scanning Electron Microscope (Courtesy: SAI Labs, Thapar University)

Figure 4.6: X-Ray Diffractometer (XRD) (Courtesy: SAI Labs, Thapar University)

4.3.5 X-Ray Diffraction (XRD)

X-ray diffraction technique is one of the most powerful tools for qualitative and quantitative analysis of materials. The analysis of this technique provides the average bulk composition of materials. The basic principle behind the XRD is that the monochromatic radiation is produced by the X-rays, which are generated through a cathode tube and concentrated on the samples. In the present work XRD is used to determine the elemental composition of the manufactured composites, produced through microwave processing. The XRD results were obtained at room temperature using X-ray diffractometer as shown in Fig. 4.6 (Make: *X'Pert PRO, PANalytical*) in

SAI Labs, Thapar Technology Campus, Patiala. with Cu kα x-ray. The scanning rate was fixed at 1° $min^{-1}$and the range was from 5° to 100°.

4.3.6 Wear Study (Pin-on-disc Tribometer)

A pin on disc tribometer (Ducom India) was used to calculate the dry sliding wear behavior of the composites. Composites were cut cross-sectionally into the dimensions having (8mm × 8mm × 6mm) and samples were held in the square clamp (V-shaped) against the rotating disc. The rotating disc was prepared from EN31 steel and its hardness was maintained at about 108 HRB. The rotating disc as well as samples were cleaned prior to start the testing.

Figure 4.7: Experimental setup of wear and friction monitor (Courtesy: Machine tool Lab, TU)

Trials were carried out at different speeds and by varying the loads. Tribological behavior was studied for sliding distances (500m, 1000m, 1500m, and 2000m), 3 different loads (0.5 Kg, 1Kg and 1.5 Kg) and velocities of 0.5 m/s, 1m/s and 1.5 m/s without any lubrication. Before measuring the weight loss of worn out samples, they were cleaned with the acetone and dry air. Weight loss of samples was measured by electronic balance with least count 0.001gm after every 500 m of sliding distance. After carrying out the tribological testing of the specimens, weight loss of the composites was calculated, which helped to find out the wear rate of the composites at different sliding speeds and at different loads. The standard test conditions and all related parameters are shown in the Table 4.3 below.

Table 4.3: Details of Sliding wear parameters for wear study

Parameters	Description
Testing specimens and setup	Pin-on-disc (a) Pure copper (b) Cu-2 wt.% graphite (c) Cu-5 wt.% graphite Thickness= 8mm
Counter disc	Material of disc=EN-31 Hardness of disc= 108 HRB Diameter of disc=90 mm
Sliding distance (m)	500, 1000, 1500, 2000
Sliding speed (m/s)	0.5, 1.0, 1.5
Normal Load (Kg)	0.5, 1.0, 1.5
Lubrication condition	Dry condition
Temperature (°C)	Atmospheric Temperature

4.3.7 Fractographic Analysis (Optical Microscope)

The samples after carrying out the wear study were seen under the microscope to find out the worn out surface, formation of tribolayer and other defects due to dry sliding. The optical microscope is used to study the microstructural images having magnification range generally between 10X to 50X.

Figure 4.8: Optical Microscope (Courtesy: A.M. Lab, Thapar University)

Chapter-5
Result and Discussion

Metal matrix composite (MMC) castings of copper with varying percentage of reinforcement (graphite) were successfully developed through microwave processing by using the concept of microwave hybrid heating (MHH). The following section describes the characterizations of the developed composites.

5.1 Hardness Measurement

The variation in hardness at different positions and at various points have been measured with and without the graphite reinforcement and results are shown in Fig. 5.1. The micro-hardness of the pure copper castings as well as graphite reinforced composite was carried out at the load of 50 g and dwell time of 10 s. The general hardness trend should be that with the addition of graphite, the hardness should reduce upto an extent because of the soft graphite nature. However, there were lots of variations obtained in the data during hardness measurements because the hardness taken on the graphite particle has different hardness and the position where the copper was in huge amount, the hardness values were different, so the data obtained was uneven and scattered.

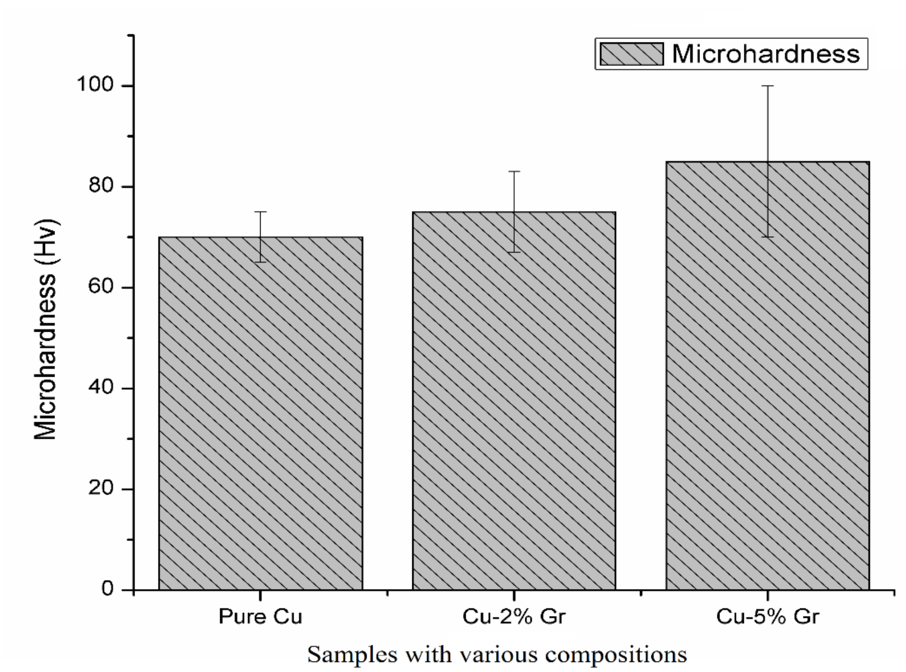


Figure 5.1: Micro-hardness of the microwave processed metal composite Castings

The Vickers hardness value of around 70 was obtained for pure copper cast samples, which is little bit higher than compared to the casted copper with the conventional one (62 Hv). With the addition of graphite upto 2wt%, the hardness obtained was also around 75Hv but a huge difference was seen when graphite content was increased upto 5wt% and hardness obtained was around 85Hv, which is very high as compared to pure copper's hardness value.

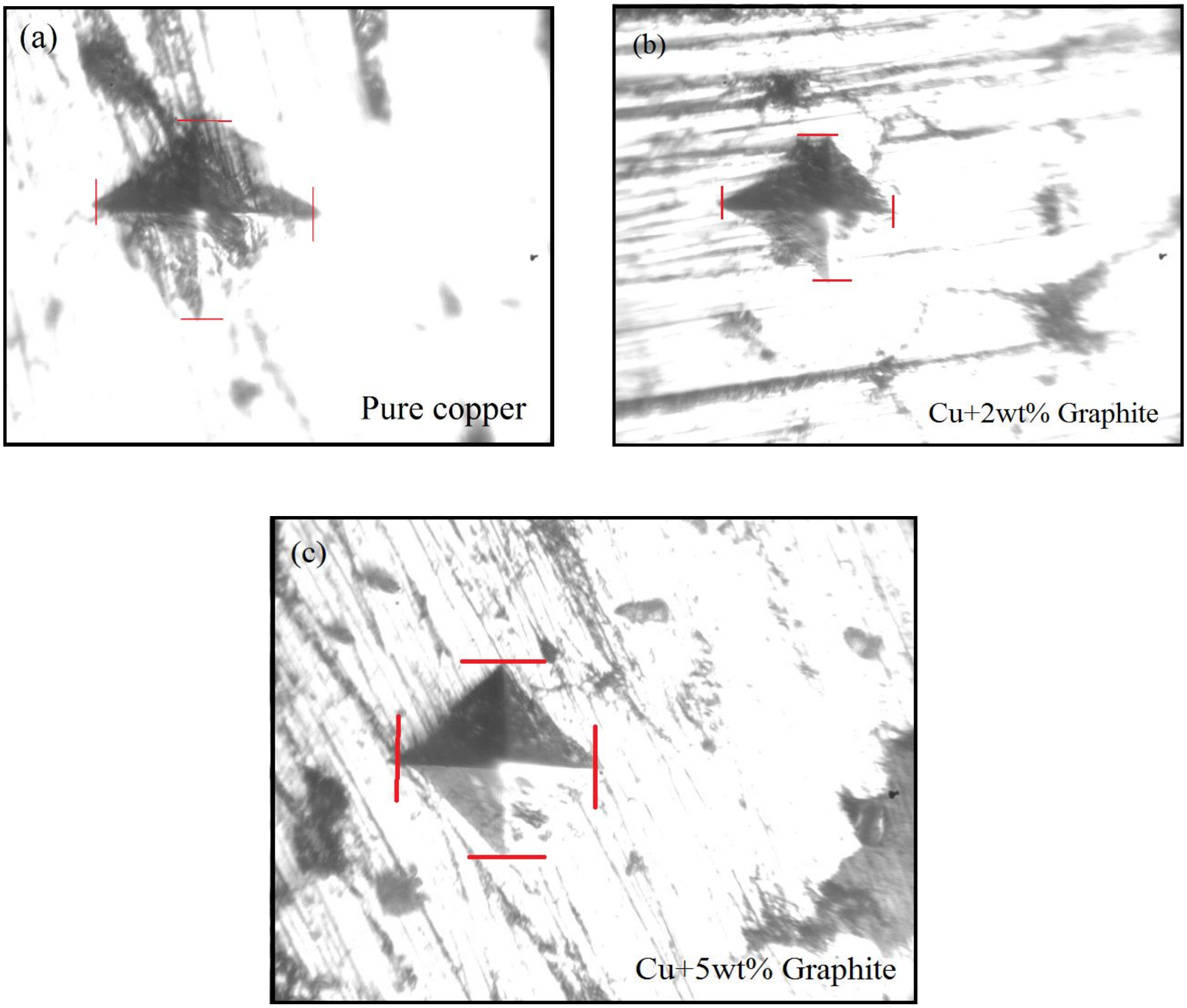


Figure 5.2: Micro-hardness indentations on samples (a) Pure copper, (b) Cu+2wt%Gr, (c) Cu+5wt%Gr

Another reason of higher hardness is fine grain size in case in both the cases having copper as well as graphite. The better rate of graphite dispersion over the matrix and grain size refinement are the main reasons which are responsible for increase in hardness of Cu-graphite composites. Graphite being a main form of carbon increased the hardness of the composite.

5.2 Composite microstructural observations

The microstructure of microwave casted sample is shown in Fig. 5.3(a), which shows the SEM micrograph. It can be seen from that proper melting and fusion of copper particles is achieved throughout the casting.

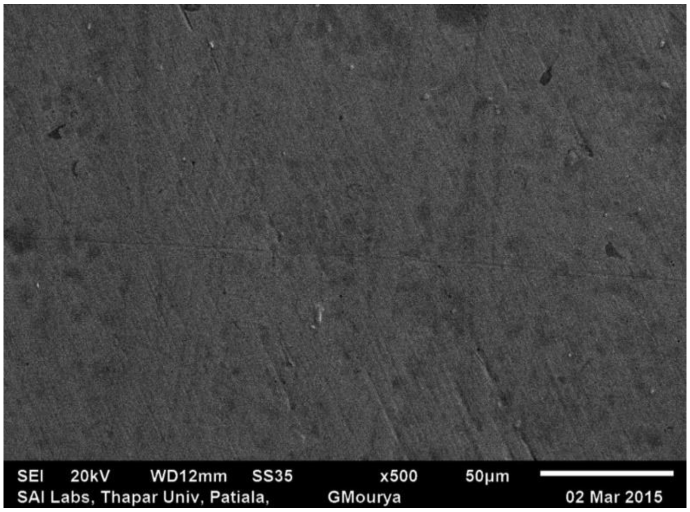


Figure 5.3(a): Microstructure of pure copper casting fabricated through microwave heating

Fig. 5.3(b) shows the SEM micrograph and distribution of Cu and graphite particles throughout the casting. Graphite being used as a reinforcement was properly mixed with copper used as a matrix even after the poor bonding between both the powders and graphite was uniformly dispersed on the matrix.

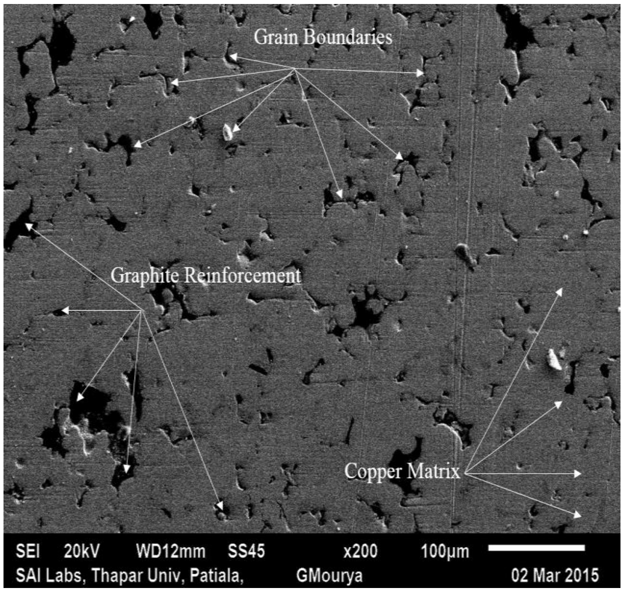


Figure 5.3(b): Microstructure of Cu+2wt% Gr

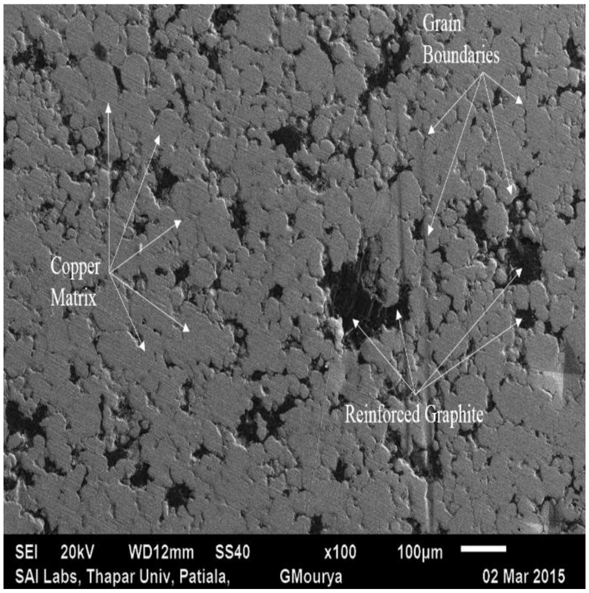


Figure 5.3(c): Microstructure of Cu+5wt% Gr

Fig. 5.3(c) shows the SEM of copper-graphite composite having graphite as 5% composition by weight. In this micrograph, grain boundaries were clearly visible and graphite was uniformly distributed over the matrix. Graphite when added into metals, it generally acts as a porous material and microwaves have capability to cast the material with high density, which was achieved upto an extent from visibility of micrographs.

5.3 Elemental Study of Composites

The elemental study was carried out by Energy Dispersive X-ray Spectroscopy (EDS). The Scanned microstructures were analysed to check the elemental composition and distribution of alloying elements throughout the micrographs. The EDS spectra shows the peaks of Cu and graphite and its interactions with atmospheric contaminants. Fig. 5.4 (a) shows the EDS spectra of the pure copper cast product.

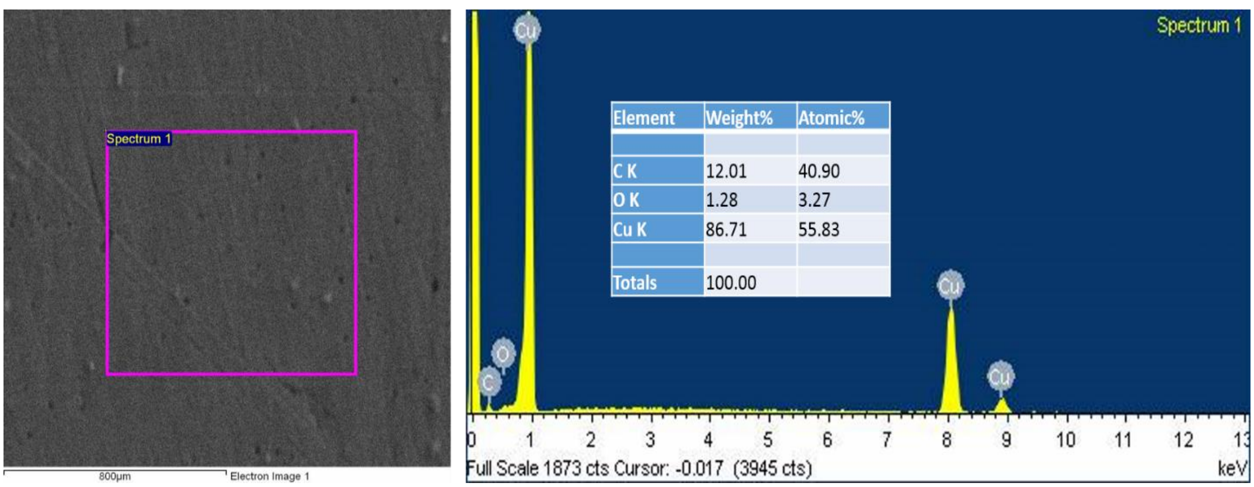


Figure 5.4(a): Microstructure and elemental composition of pure copper casting

Copper was the main constituent, so the peaks of copper were seen. Carbon was seen about 14% and it was just because the casting/ melting of copper powder was carried out in the graphite cavity and there was an interaction between the seperator material and the powder to be cast so, the carbon peaks also came into play. Copper oxide was formed, which is generally considered as impurity because at high temperature when copper comes in contact with the atmospheric oxygen, it’s very prone to make copper oxide (CuO). But sometimes the formation of oxides is favourable because it provides little hardness to the product which is generally required when soft materials/ductile materials comes into contact with forces or sliding contact with other materials. Due to this high carbon percentage and oxide formation, the hardness of the casted copper was increased upto 70 Hv, which is higher than that of conventionally cast copper.

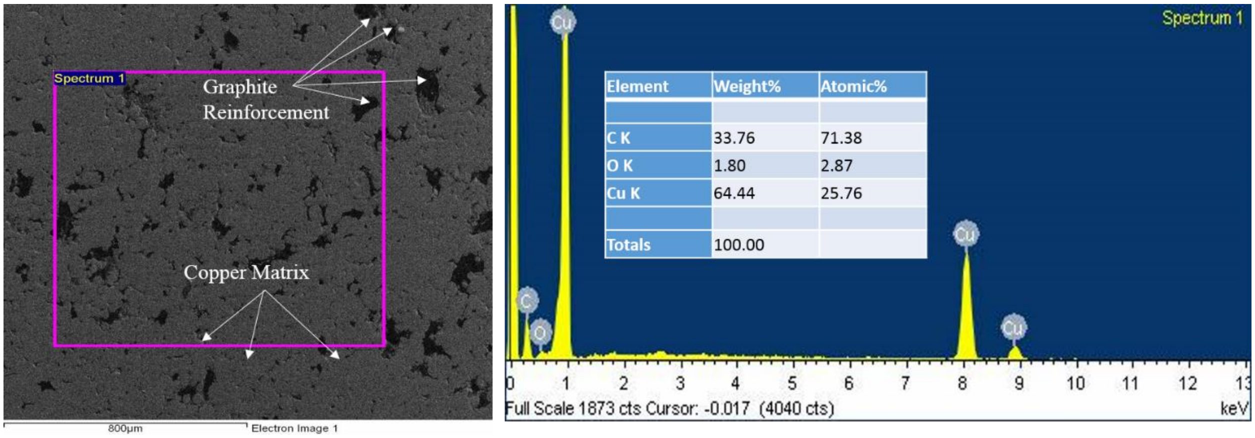


Figure 5.4(b): Microstructure and elemental composition of Cu+2wt% Gr composite

Fig. 5.4(b) shows the EDS spectra of the composite having copper as a matrix material and graphite used as a reinforcement with 2wt% composition. Carbon percentage was more than pure copper so, a peak was seen carrying carbon as 33% composition and hardness achieved upto 75Hv was just because of carbon percentage. The microstructure and elemental distribution for Cu+2wt% Gr is shown as in Fig 5.4(b) above.

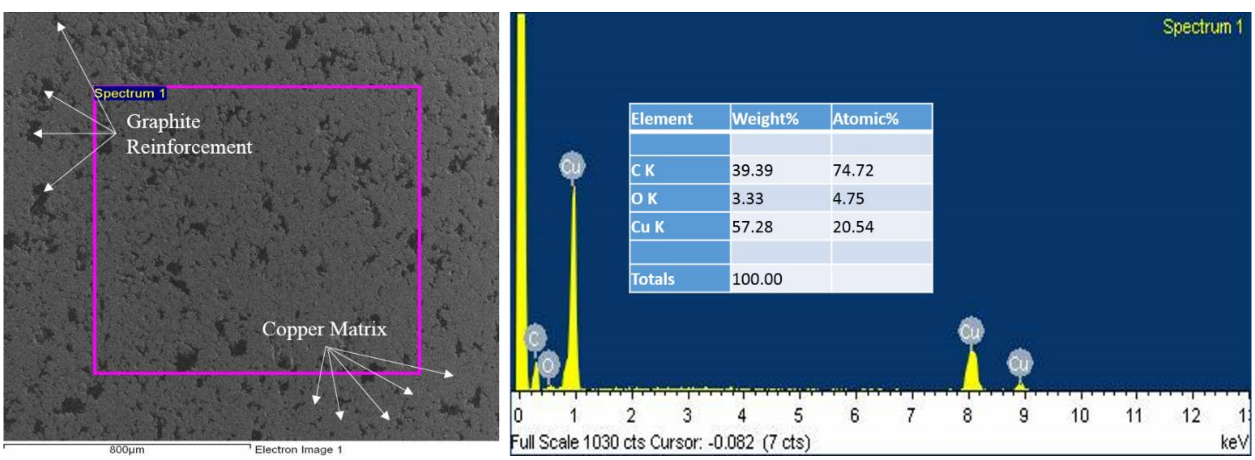


Figure 5.4(c): Microstructure and elemental composition of Cu+5wt% Gr composite

Fig. 5.4(c) shows the EDS spectra of the composite having copper as a matrix material and graphite used as a reinforcement with 5wt% composition. Graphite with 5wt% addition has observed the best value as a reinforcement in copper matrix because adding more graphite than 5wt%, it starts to have an adverse effect on matrix. The carbon percentage approached to near 40% and this was the best outcome for copper graphite composite casting because micro-hardness was achieved upto 85 Hv with this combination.

5.4 X-ray diffraction studies

A typical XRD spectrum of the pure copper developed through microwave hybrid heating is presented in Fig. 5.5(a), 5.5(b) and 5.5(c). Presence of copper is clearly identified as a major constituent and formation of oxides are seen in the XRD pattern as shown in Fig. 5.5(a). It is attributed to possible decomposition at higher temperature during MHH from Cu powder to CuO.

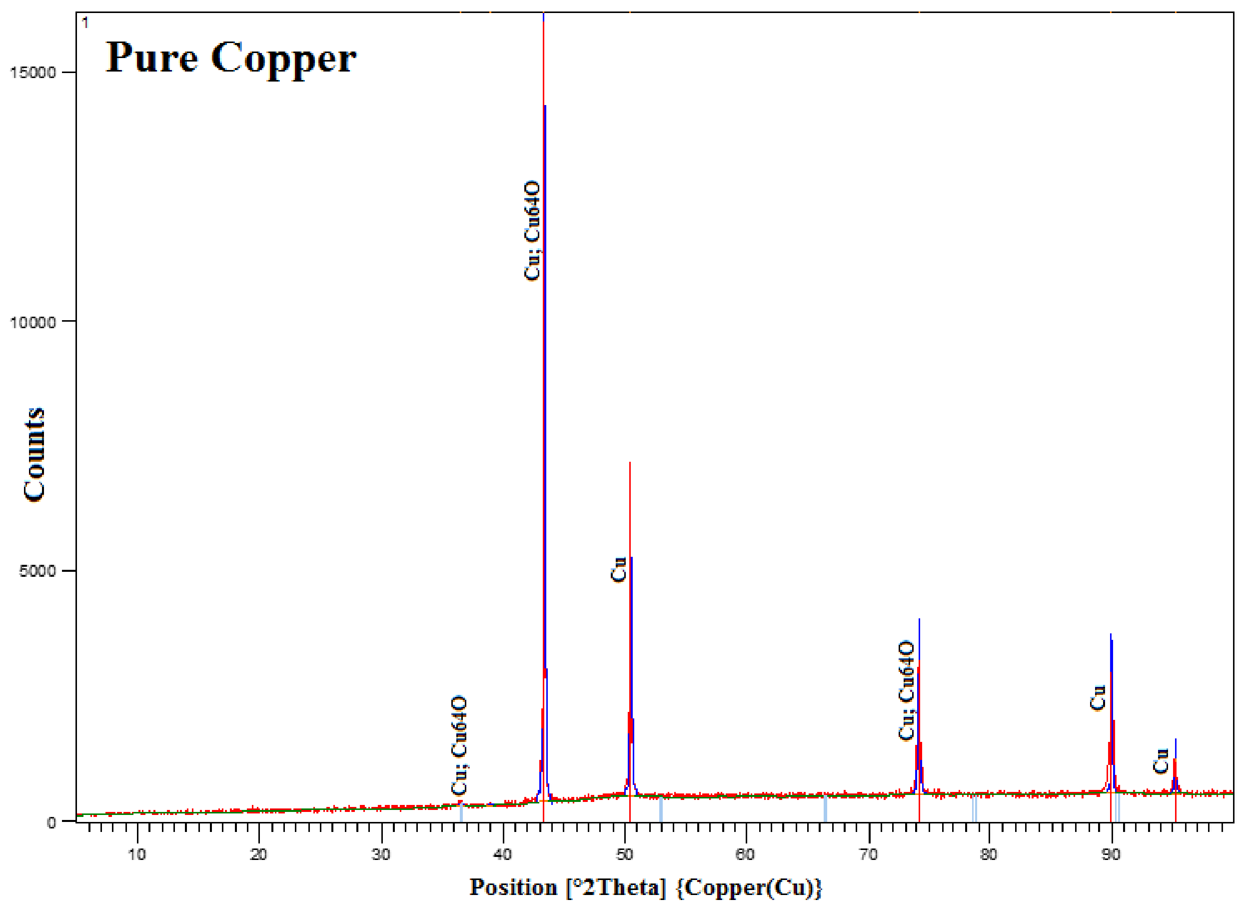


Figure 5.5(a): Typical XRD Spectrum for Pure copper casting

A typical XRD spectrum of the copper-2 wt. % graphite by same methodology is presented in Fig. 5.5 (b). Presence of graphite as one of main carbon constituent with diamond leads to high carbon percentage. Formation of Copper oxides is also identified as carbon reacts with atmospheric oxygen results into the formation of CuO which during solidification (slow cooling rate) escapes subsequently leading to crack free composite and less porosity. The XRD spectrum clearly shows that there is no interaction between the copper and graphite during the fabrication of metal matrix composites, because it has not made any compound with the graphite.

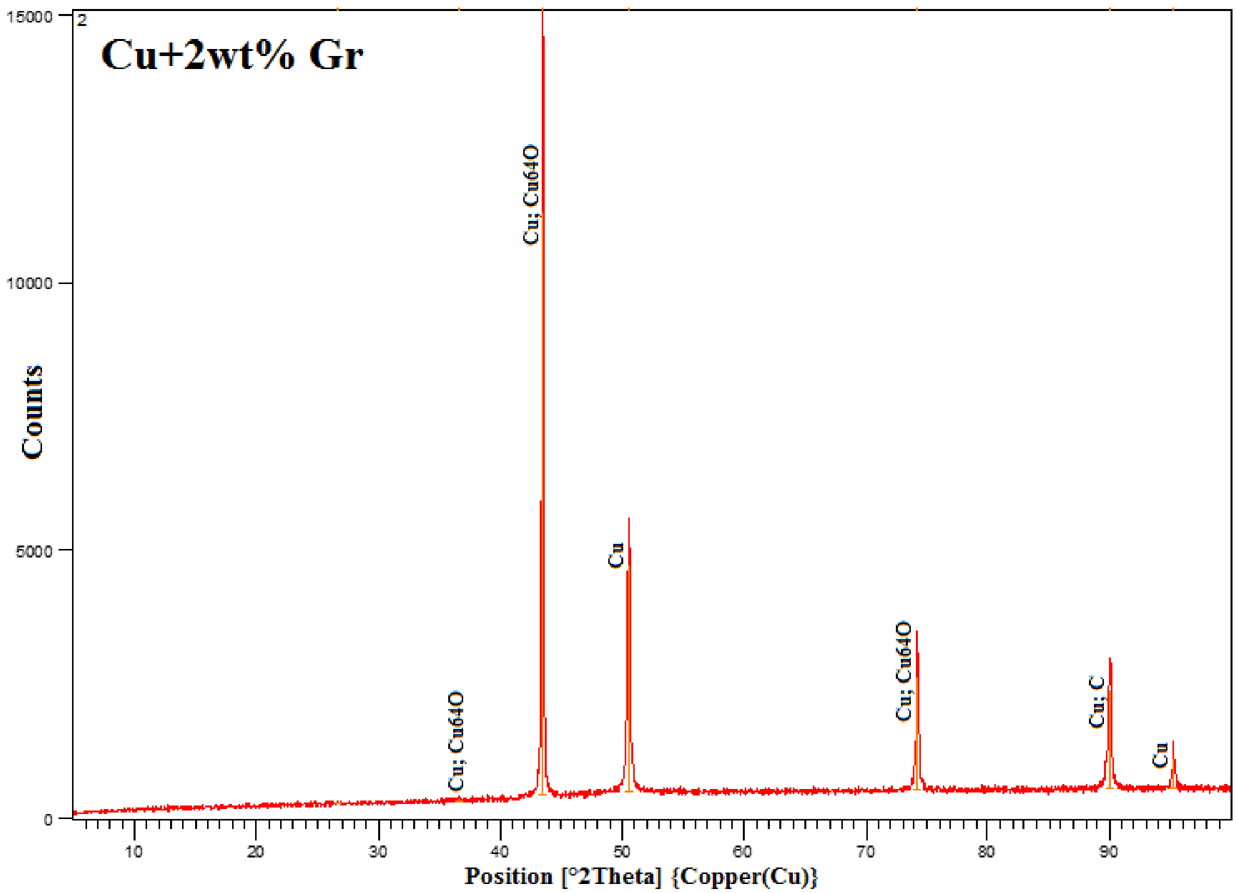


Figure 5.5(b): Typical XRD Spectrum for Cu+2wt% Gr composite

A typical XRD spectrum of the copper-5 wt. % graphite by same methodology is presented in Fig 5.5(c). Presence of graphite as one of main carbon constituent with diamond leads to high carbon. In this XRD spectrum, a peak is formed at an angle of 43°, which shows the interaction of copper particles with the graphite and smaller peaks were also available containing carbon. These uniform peaks of carbon and copper signifies that graphite is uniformly dispersed throughout the matrix. The formation of copper oxides was also seen which is due to the contact of atmospheric gases with the copper at high temperature.

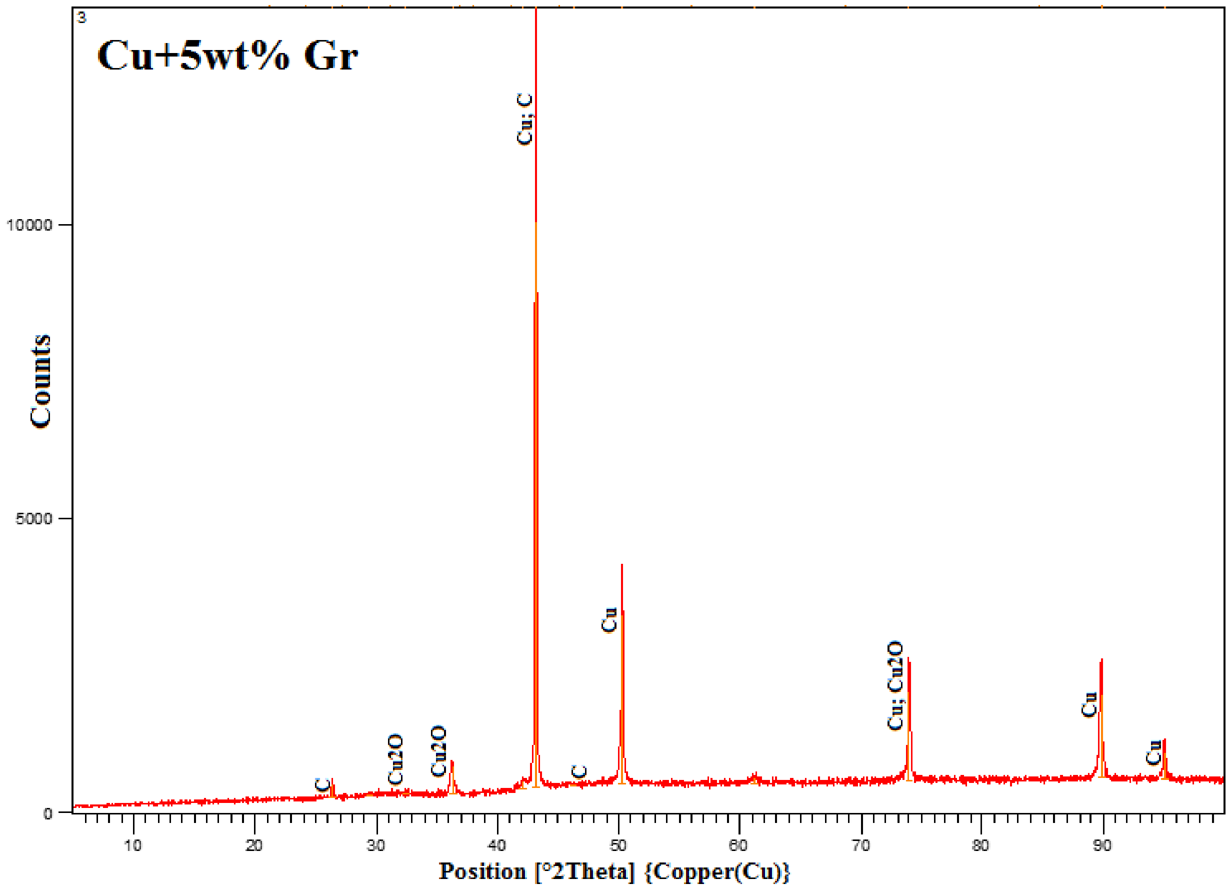


Figure 5.5(c): Typical XRD Spectrum for Cu+5wt% Gr composite

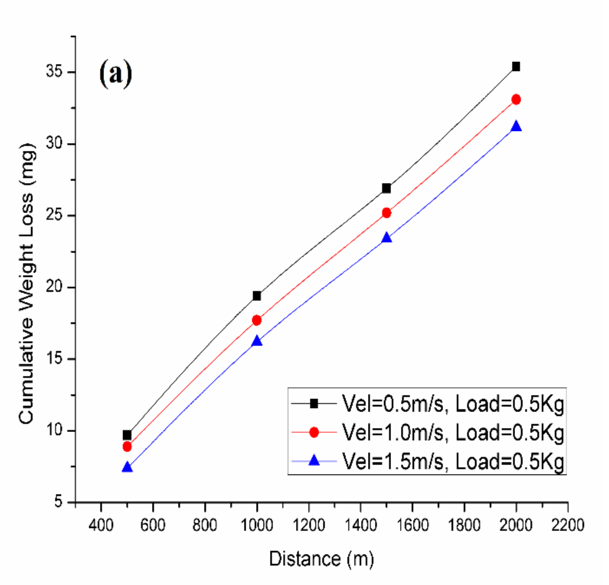
5.5 Wear Study

From the experimental observation and graphs plotted between cumulative weight loss (mg) v/s distance (m), it is observed that the wear resistance increases as per the following trend:

Pure Cu < Cu-2wt. % graphite < Cu-5wt. % graphite

5.5.1 Pure Copper Casting

During the dry sliding wear testing of copper on a rotating disc, wear rate of the casting increased with increasing normal load and decreased with increasing the velocity. As shown in Fig 5.6(a), the maximum weight loss was noticed at the minimum velocity at 0.5 m/s while the weight loss occurred approximately in the linear fashion at velocity1.0 m/s and weight loss decreased with increasing the velocity from 0.5 m/sec to 1.5 m/sec. On the other hand, with increase in normal load, the weight loss increased ultimately.



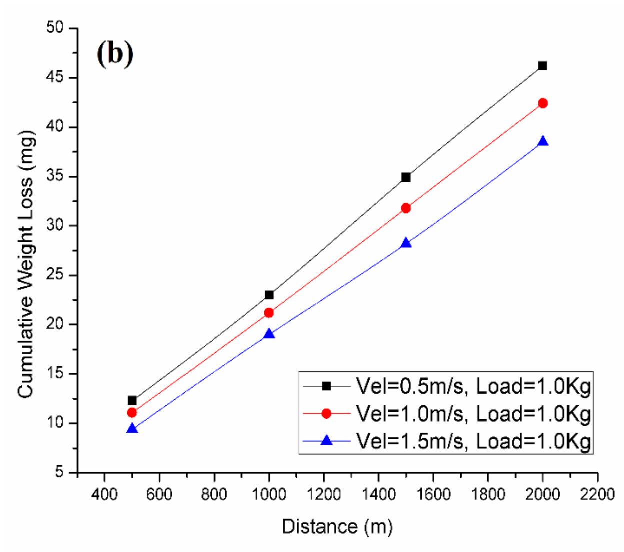


Fig 5.6(a): Weight loss of Pure Copper at 0.5 Kg load   Fig 5.6(b): Weight loss of Pure Cu at 1.0 Kg load

As shown in Fig 5.6(b), the weight loss at velocity 1m/s increases linearly and for velocity 0.5 m/s first increases linearly upto 1000 m sliding distance and then it increases constantly.

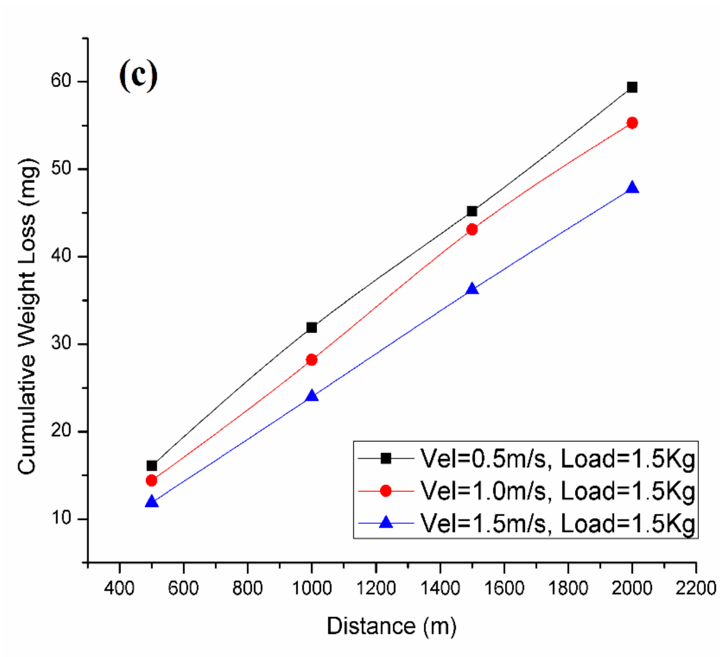


Fig 5.6(c): Weight loss of Pure Copper at 1.5 Kg Load

In case of pure copper castings, weight loss increased in the linear manner at maximum velocity (v=1.5m/sec) and for velocities 0.5 m/sec and 1.0 m/sec it do not followed a specific path. At lower velocities and at maximum load, weight loss changed at each and every sliding distance of 500 m.

5.5.2 Copper+2% Graphite

The weight loss in the composite (Cu+2% Gr) at the minimum load of 0.5kg and at minimum velocity 0.5m/s was noticed to be maximum as shown in Fig 5.7(a) and increased approximately at same rate for velocity 1.5 m/s and at velocity 1.0 m/s, weight loss approached approximately to the weight loss at 0.5m/s.

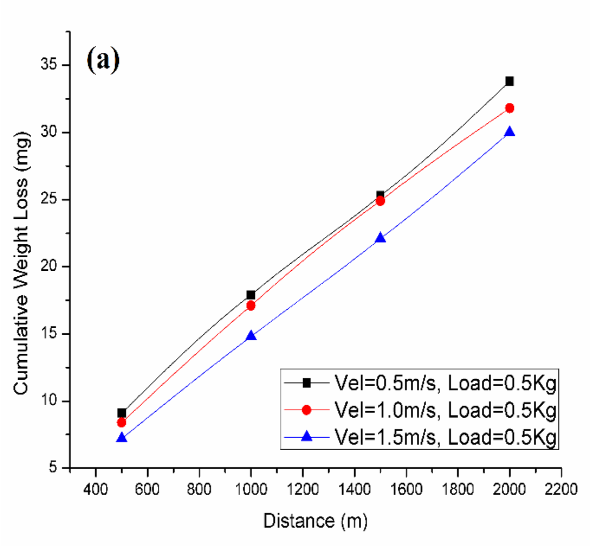


Fig 5.7(a): Weight loss of Cu+2% Gr at 0.5 Kg load

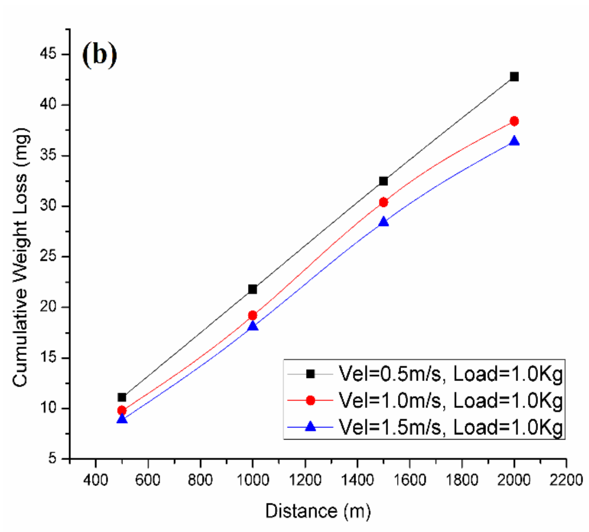


Fig 5.7(b): Weight loss of Cu+2% Gr at 1.0 Kg load

The wear loss at 1kg normal load for the composite changed after every 500 m sliding distance but at the end of 2000m sliding distance the maximum wear loss was observed at minimum velocity of 0.5 m/s as shown in Fig 5.7(b) and linear trend of weight loss was followed at velocity 0.5 m/s.

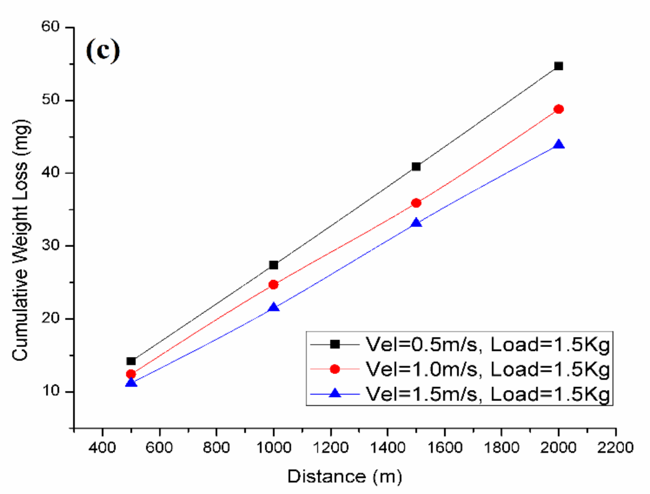


Fig 5.7(c): Weight loss of Cu+2% Gr at 1.5 Kg load

At maximum load of 1.5 kg, due to greater load the wear rate increased but increased approximately with the same manner for all three velocities approximately as shown in Fig 5.7(c).

5.5.3 Copper+5% Graphite

The same trend of weight loss was also formed here as shown in Fig. 5.8 (a). The maximum wear loss occurred at minimum velocity of 0.5 m/sec and same trend was followed for weight loss at this velocity but the amount of wear loss decreased significantly as compared to pure copper cast.

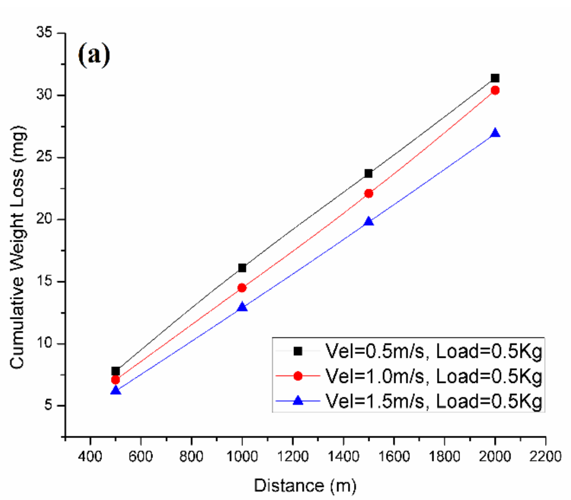


Fig 5.8(a): Weight loss of Cu+5% Gr at 0.5 Kg load

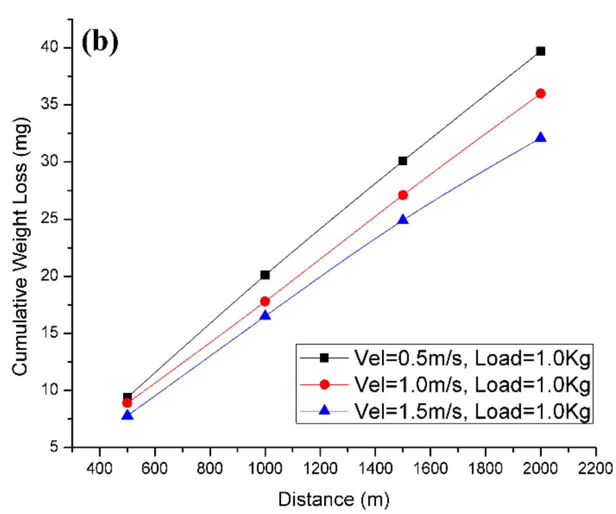


Fig 5.8(b): Weight loss of Cu+5% Gr at 1.0 Kg load

The same trend was followed further in Fig 5.8(b) and (c), but at maximum load and at maximum velocity, weight loss tends to decrease drastically.

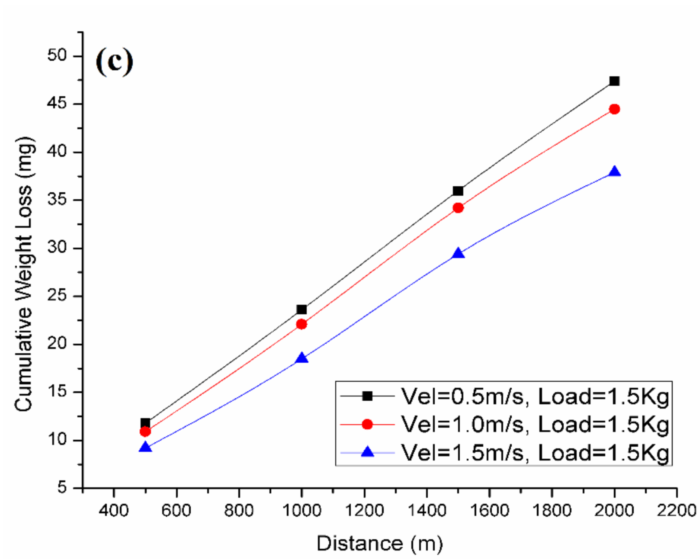


Fig 5.8(c): Weight loss of Cu+5% Gr at 1.5 Kg load

The main reason behind the maximum weight loss at minimum velocity is that at low speed, metal to metal contact was more and then the load acting on it put a great force on the samples during wear study. Due to slow sliding speed and high amount of force tends to produce the localised welding of metal with the rotating disc and this phenomenon of formation of bonds and breaking of bonds remains throughout the process till the steady state is not achieved.

The reason behind the minimum weight loss in case of Cu+5%Gr composite casting than compared to pure copper casting is that graphite adheres to the wear surface due to its sticky nature, and a solid graphite layer is formed as a lubricating film comes into being on the rotating disc (wear surface). Due to the formation of graphite layer on the contact surface, the metal to metal contact was very less and this metal to metal contact was converted into the contact between the graphite layer and copper metal composites. Therefore, the wear properties of Cu–graphite composites are greatly improved in comparison with those of pure copper.

5.6 Analysis of wear rate

The wear rate of the cast samples was calculated from the weight loss and it was observed that at constant load of 0.5 Kg, maximum wear occurred at minimum velocity of 0.5 m/s for pure copper. The wear rate obtained at velocity 0.5 m/s for pure copper was 8.85 mg/s and it was reduced to 7.85 mg/s by adding 5% graphite in copper as shown in table 5.1. As for as wear rate is concerned at velocity 1 m/s, wear rate was decreased from 16.5 mg/s to 15.2 mg/s with the addition of 5% graphite. The huge change in wear rate was noticed for Cu+5% Gr composite at velocity 1.5 m/s, where it was reduced from 23.45 mg/s to 20.17 mg/s.

Table 5.1: Wear rate of casting samples at load 0.5 Kg

Sliding Velocity (m/sec)	Wear Rate ($\times 10^{-3}$ mg/s)											
	Pure Copper				Cu+2% Gr				Cu+5% Gr			
	Sliding Distance (m)				Sliding Distance (m)				Sliding Distance (m)			
	500	1000	1500	2000	500	1000	1500	2000	500	1000	1500	2000
0.5	9.72	9.74	8.96	8.85	9.1	8.95	8.43	8.45	7.8	8.2	7.9	7.85
1.0	17.8	17.7	16.7	16.5	16.8	17.1	16.6	15.9	14.2	14.5	14.73	15.2
1.5	22.2	24.3	23.35	23.45	21.6	22.2	22.14	22.5	18.6	19.3	19.8	20.17

The wear rate also decreased with addition of graphite in copper at higher velocities also. The wear rate also decreased in the similar fashion as described above but the wear rate enhanced here because of the normal load increased here from 0.5 Kg to 1 Kg. The wear rate decreased with increasing amount of graphite and at maximum sliding distance and at maximum velocity, the copper has a wear rate of 28.87 mg/sec and was reduced to 24.07 mg/sec for Cu+5% Gr.

Table 5.2: Wear rate of casting samples at load 1.0 Kg

Sliding Velocity (m/sec)	Wear Rate ($\times 10^{-3}$ mg/s)											
	Pure Copper				Cu+2% Gr				Cu+5% Gr			
	Sliding Distance (m)				Sliding Distance (m)				Sliding Distance (m)			
	500	1000	1500	2000	500	1000	1500	2000	500	1000	1500	2000
0.5	12.3	11.5	11.63	11.55	11.1	10.9	10.83	10.7	9.4	9.6	10.03	9.92
1.0	22.2	21.2	21.24	21.15	19.6	19.2	20.26	19.2	17.8	17.7	18.06	18.0
1.5	28.2	28.5	28.2	28.87	26.7	27.1	28.4	27.3	23.4	24.7	24.95	24.07

The critical condition for the normal load was 1.5 Kg and maximum wear occurred at this load. Generally in electrical appliances, high amount of load comes into contact so composite was also checked at varying load but the same result came into existence. The minimum wear rate was obtained at maximum velocity of 1.5 m/sec and it was observed due to lubrication phenomenon of graphite. At velocity 1.5 m/sec, the wear rate of pure copper was 35.85 mg/sec and was decreased to 28.42 mg/sec.

Table 5.3: Wear rate of casting samples at load 1.5 Kg

Sliding Velocity (m/sec)	Wear Rate ($\times 10^{-3}$ mg/s)											
	Pure Copper				Cu+2% Gr				Cu+5% Gr			
	Sliding Distance (m)				Sliding Distance (m)				Sliding Distance (m)			
	500	1000	1500	2000	500	1000	1500	2000	500	1000	1500	2000
0.5	16.1	15.95	15.06	14.85	14.2	13.7	13.63	13.68	11.8	11.7	12.0	11.92
1.0	28.8	28.2	28.73	27.65	24.8	24.7	23.93	24.4	21.8	22.1	22.8	22.25
1.5	35.7	36.0	36.2	35.85	33.6	32.2	33.1	32.92	27.6	27.7	29.4	28.42

5.7 Reduction in Wear Rate

The maximum wear rate of the pure copper sample was obtained maximum at velocity 1.5 m/s and at maximum load of 1.5 Kg and it was about 35.85×10^{-3} mg/s. The wear rate with the addition of 2% graphite in copper, was decreased upto 32.92×10^{-3} mg/s and further addition of graphite upto 5% graphite lowered down the wear rate to 28.42×10^{-3} mg/s. The final reduction in wear rate is shown in Table 5.4. As shown in table, the reduction in wear rate is obtained by comparing it with the pure copper cast.

Table 5.4: Total reduction in wear rate (×10-3 mg/s)

Sliding Velocity (m/sec)	Reduction in Wear Rate ($\times10^{-3}$ mg/s)					
	Load=0.5 Kg		Load=1.0 Kg		Load=1.5 Kg	
	Cu+2%Gr	Cu+5%Gr	Cu+2%Gr	Cu+5%Gr	Cu+2%Gr	Cu+5%Gr
0.5	0.4	1.0	0.8	1.63	1.17	2.93
1.0	0.6	1.3	1.95	3.15	3.25	5.4
1.5	0.95	3.28	1.57	4.8	2.93	7.43

5.8 Fractographic Analysis

The cast copper showed maximum wear at minimum velocity and at maximum load and it was obtained about 35.85×10^{-3} mg/s on maximum distance of 2000 m. In the fractographic analysis of pure copper casting sample, it showed wear track because of rotating disc and track impressions were formed on it. Tribolayer also came into contact on the microscopic scale but the defects such as plastic deformation, micro cracks and crater wear also came into play. These defects may be due to rubbing of dry surfaces (copper and rotating disc) with each other and due to rubbing, high temperature may generate which is supposed to be a main reason behind these defects. The fractographic image of pure copper is shown in Fig 5.9(a).

On the other hand, with the addition of graphite, the wear rate was wear rate was controlled upto an extent and it was reduced from 35.85 mg/sec to 32.92 mg/sec. The main reason behind the reduction of wear rate is the formation of tribolayer on the composite sample, which minimise the metal to metal contact because of the lubrication property of the graphite. The wear track was also clearly visible in the fractographic image and the defects such as micro-cracks, crater wear and delamination etc. were also lesser than that of in pure copper as shown in Fig 5.9 (b).

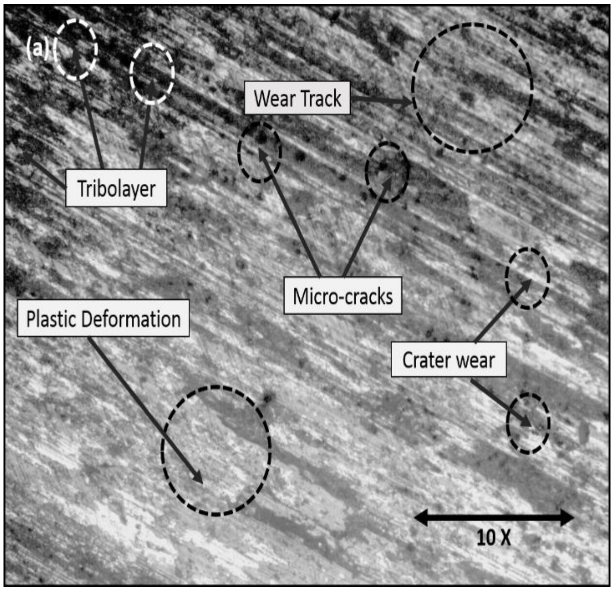


Figure 5.9(a) Fractographic image of Pure Copper casting

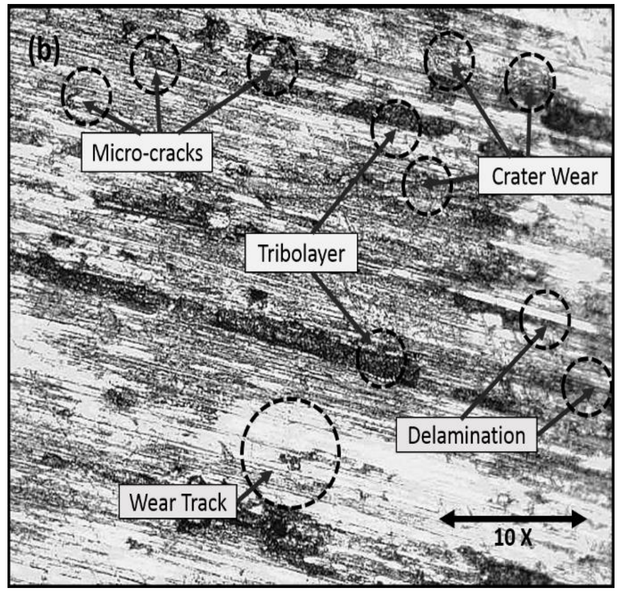


Figure 5.9(b) Fractographic image of Cu+2% Gr casting

The same phenomenon for reduced wear rate was observed for Cu+5% Gr composite. Due to large amount of graphite contact, more tribolayer formation lead to reduced metal to metal contact. Large amount of graphite provide the lubrication and due to this lubrication effect, the temperature of the composite during tribology testing was controlled. Less amount of temperature generation on the mating surfaces, it lead to generation of less defects as shown in Fig 5.9 (c).

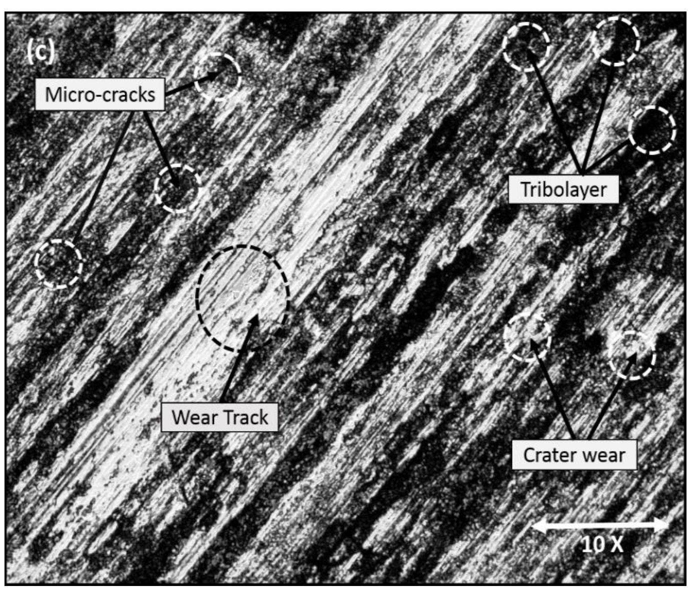


Figure 5.9(c) Fractographic image of Cu+5% Gr casting

Chapter- 6

Conclusion and Future Scope

6.1 Conclusion

The processing of materials through microwave energy has emerged as a novel material processing technique, which has extended its domain from sintering of ceramics to metallic materials. Further, high temperature applications using microwaves were carried out such as joining of bulk metals and claddings on the bulk metallic substrate. The present work was to fabricate the metal matrix composite through microwave hybrid heating and using copper as a matrix and graphite as a reinforcing agent. Following major conclusions are drawn from the work:

1. The development of metal matrix composite was successfully attempted by using different compositions of the reinforcement through microwaves in domestic multimode microwave oven.

2. The characterizations of the manufactured composites were carried out and it was observed that microwave processed composites have lower visible defects and even though a poor bonding between copper and graphite, a dense product was obtained.

3. SEM analysis revealed the good bonding between copper matrix and graphite reinforcement and graphite was uniformly distributed over the matrix in both the reinforcement composites.

4. Higher hardness was obtained in microwave processed pure copper casting due to the absorption of carbon from graphite sheet as well as graphite cavity. On the other hand side, the composites prepared have also higher hardness than pure copper.

5. Fractographic analysis of the worn out samples carried out after dry sliding wear behavior of the composite showed that graphite acted as a solid lubricant layer between the metals (rotating disc and Cu-Gr composites) and reduced wear.

6.2 Scope for Future Work

A lot of scope is still left for enhancing the research in this particular area of microwave material processing. Following work can be carried out in future:

- Effect of pitch coke can be studied in improving the interfacial bonding characteristics between copper and graphite.
- To study the effect of load, coefficient of friction, wear volume, wears mechanisms, etc. on MMC developed through microwave energy.
- Electrical conductivity of Cu-graphite composites can also be measured and effect of reinforcement can be studied on conductivity.
- To study the mechanical characterizations in terms of tensile testing of the composites.
- Porosity level and surface roughness of microwave processed MMC’s can be studied.
- Cu-graphite composites fabricated through microwave hybrid heating can also be fabricated through microwave sintering & other advanced techniques for manufacturing MMC’s and a comparative study can also be carried out.

Visible Outputs

1. Filed one Indian Patent:
   Satnam Singh, Dheeraj Gupta, Vivek Jain, **Rohit Kumar.** “A method for Metal Ceramic Composite Casting through Microwave Energy”. Indian patent, Application No. TEMP/E-1/20263/2015-DEL.
2. Submitted one paper in SCI Journal:
   Rohit Kumar, Satnam Singh, Dheeraj Gupta and Vivek Jain. “Dry sliding wear behavior of Microwave Processed Cu-Gr Composites”. Tribology Transaction (submitted).

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