CHAPTER 1

INTRODUCTION

Powder metallurgy (P/M) is perceived today as a standout amongst the most important manufacturing methods. The vitality required for processing and forming is less and it is conceivable to introduce coveted properties at different sections of a similar component to satisfy performance necessities. P/M is the field of processing technique in engineering science, in which new forms of composite are fabricated by compacting the various metal powders in different weight or volume ratios. The expanding uses of P/M innovation within the nuclear, defence, automotive industries and additionally in the production of bearing and electrical contacts like pantograph have conveyed the ability of P/M to be an alternative route to traditional metal processing.

1.1 PRELUDE

The significance of P/M as an imperative fabrication method got to be clear as right on time as nineteenth century for electrical applications when tungsten was developed and strengthened with thorium to be used in electric lamp filaments. Examination apropos and investigations of the materials are acclimated to clarify and improve their backdrop in properties for future applications to benefit humankind. Another basic defining moment in the advancement of forefront P/M industry is the development of tungsten carbide tools. Creation of porous bearings and bushes by P/M further prodded its development. In this way, ferrous and nonferrous parts,
composites and also alloys strengthened by dispersion of particles made by P/M came into utilization. Apart from completed parts, P/M is additionally utilized in various helpful applications in advanced innovations.

Imperative illustrations in this class incorporate utilization of metal powder in printing, sustenance industry and catalysts in chemical industries. It is acclimated for the fabrication of completed or semi-completed parts from the metal powders. P/M has additionally settled its predominance by being the only manufacturing technique for the development of several parts such as porous bearings and carbide tools.
Likewise, P/M parts are in effect progressively utilized as part of a wide assortment of commercial enterprises as the innovation joins both exceptionally specialized components with economy and added advantages such as environmental friendliness and energy efficiency.

1.2 STEPS INVOLVED IN POWDER METALLURGY

P/M fabrication turned out to be fascinating in maintaining a strategic distance from the constraints and imperfections of other manufacturing techniques and in delivering materials with predominant properties acquired from the powder attributes. The main steps involved in the fabrication of P/M component are as follows:

1. Mechanical alloying
2. Compaction
3. Sintering
4. Secondary operations
The schematic representations of P/M processing are shown in the form of a flow chart in Figure 1.1. Jianxin Wang et al. (2013) fabricated copper-graphite composites through P/M method.

![Flow Chart](image)

**Figure 1.1 Process flow diagram**

### 1.2.1 Mechanical Alloying

Mechanical Alloying (MA) is a solid state processing method utilized as right on time as 1910, for the creation of thoriated tungsten for electric lamp filaments. It was established as a full scale production technique, only in the late sixties, once Benjamin (1976) of inco alloys utilized this practice for the creation of oxide dispersion strengthened nickel-base alloys from elemental powder mixtures. MA is a novel development of mechanical comminution and is done in attriters or other high-vitality ball processes. An extensive variety of materials incorporating the vast majority of Commercially-accessible compounds, composites and other interesting
materials have been produced utilizing mechanical alloying. The procedure has empowered the fabrication of powders of prevalent quality with high homogeneity and required size range including nanosizes. The schematic view of ball milling is shown in Figure 1.2.

**Figure 1.2 Schematic view of ball milling**

MA involves repeated welding, fracturing and rewelding of powder particles throughout milling, leading to a homogeneous dispersion of the materials. The dispersion in MA can attain such fine levels that inter particle spacing between dispersoids can be well below 0.5 µm, and alloying may even appear homogeneous under X-ray diffraction (XRD). This is seen by the change of particle size with increasing comminution time. Normally, prolonged comminution can cause the particle size to increase, confirming the welding of impacted particles on the surface of the grinding media. During the comminution and alloying processes, a large quantity of heat is generated and this enhances diffusion among the components and promotes recrystallization of the deformed powder.

At present, MA is utilized for the most part in the creation of oxide dispersion strengthened nickel–iron and aluminium-base alloys. Due
to their high-temperature properties, MA super alloys find applications in air-craft and industrial gas turbines, power generation equipment and in addition in heat treatment furnace parts. Mechanically Alloyed aluminium-base alloys with Al₂O₃ and AlC₃ dispersions are presently entering into applications.

In the recent years, several new facts of MA have been perceived, such as MA of liquid immiscible and solid-immiscible systems which are difficult and / or often impossible to process by conventional methods like solidification processing. Such combinations can be acquired in the solid state with a homogeneous distribution of the second phase through MA. Superconducting Nb₃Sn, super corrosion resistant Mg–Fe/Cu/Cr/Ti and hydrogen-storing Mg-Ti/Ce alloys are examples of such alloys. MA has also been successfully used to produce amorphous phases in several elemental as well as multicomponent systems. The development of such amorphous phases has been ascribed to the heavy cold working involved in the process.

Mechanical alloying has been perceived as one of the important non-equilibrium material processing strategies. As specified before, the process involves repeated welding, fracturing and rewelding of powder particles during milling in a dry, high-energy mill with or without a protective atmosphere. In brief, MA process involves micro-forging, cold welding, and fracturing of particles to gradually produce fine randomly-oriented laminates of the materials. The fine laminate formation is aided by the kneading and impact action of the entrapped powders between colliding balls. In addition, a Process Control Agent (PCA) such as stearic acid, toluene and methanol might be utilized to control excessive cold welding during killing. The procedure can be firmly controlled to accomplish an extensive variety of materials with a decision of microstructures including nano-crystalline, quasi-crystalline and additionally amorphous structures.
In **mechanical alloying**, the raw materials used could also be metal, alloy or ceramic powders. Probably, most components should be ductile enough to act as a binding media for the other components during milling. Typical examples include iron, copper, nickel, and aluminium. Processing of brittle materials is also possible by **mechanical alloying**, due to the fact that the triaxial compressive stress state existing during milling can intent these materials to undergo adequate amounts of plastic deformation. Milling is traditionally applied making use of a ball mill or an attritor. The constituent powder combination is charged into the attritor, normally under a protective atmosphere of argon, through nitrogen and air milling is sometimes employed. During the milling process, the powder particles are subjected to affect when they are caught normally between the milling media or between the walls of the container and the milling media.

### 1.2.2 Compaction

The term ‘compaction’ is used to describe consolidation of powder particles without the applying of heat. The foremost purpose of compaction is to form metal powder compacts of desired shape with sufficient strength to withstand ejection from the tools and subsequent handling as much as the completion of sintering without breakage or damage. Compaction is adopted by means of sintering to make the finished parts. In other cases, such as cemented carbides, superior properties can be achieved through hot pressing of the component powders rather than cold pressing followed by sintering.

There are lots of approaches of compaction and the option is dependent on the application as well as the scale and economy of operation. Most of the methods depend on the application of external pressure on powder contained in a die or a mould. Nevertheless, some other methods like vibratory compaction and slip casting do not use any external pressure.
In many instances, it usually is fantastic to cold press a mixture of the component powders seeing that ease of compacts containing elemental zinc is difficult, while pre-alloyed brass powder is easily cold pressed and sintered. Mostly, pre-alloyed powders are hard and cannot be comfortably cold pressed.

1.2.3 Lubrication

In die compaction, presence of frictional forces limits the degree of densification. A most important consequence of the die wall friction, as acknowledged prior, is a variation of green density in the compact. The frictional forces encountered during compaction may also be minimized to a large extent by the use of lubricants, either admixed or applied to the contact surfaces. These lubricants additionally support in the ejection of the compacts. Lubricants are normally organic compounds such as waxes or metallic stearates or salts and generally have low boiling features. The amount of lubricant added may range from 0.5 to 2.0% by weight of the charge. The lubricant may be mixed with the powders and used or can be directly applied to the die walls. Option of lubricant is predominant as it may affect the flow rate of powders or alter the die fill volume, the microstructure (formation of pores) or dimensions of the compact. Admixed lubricants serve to reduce the inter particle friction and aid better rearrangement and packing. But depending on their volume and density, they may also have an impact on densification significantly. Also in the case of admixed lubricants, it is necessary to remove them before sintering, because they may cause undesirable situation such as distortion of the compact. The use of admixed lubricant also has an adversarial influence on the density. Even a small amount (1wt.%) of lubricant can occupy large volume (5%) and assuming zero porosity, maximum attainable density is only 95%. Lubricants are, consequently, eliminated for the period of a burn-offstage prior to sintering.
Use of die wall lubricants can avoid problems caused by way of admixed lubricants. Graphite and molybdenum disulphide are common die wall lubricants. They are easily removed the fact that they adhere only to the surface of the compact, but take longer production times, because it must be utilized before every operation. This affects the productivity adversely. Some almost always used lubricants are given in Table 1.1.

<table>
<thead>
<tr>
<th>Lubricants</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Paraffin wax</td>
<td>Stearic acid</td>
</tr>
<tr>
<td>Butyl stearate</td>
<td>Sodium stearate</td>
</tr>
<tr>
<td>Aluminium stearate</td>
<td>Oleic acid</td>
</tr>
<tr>
<td>Lithium stearate</td>
<td>Poly glycols</td>
</tr>
<tr>
<td>Magnesium stearate</td>
<td>Talc</td>
</tr>
<tr>
<td>Graphite</td>
<td>Molybdenum disulphide</td>
</tr>
</tbody>
</table>

1.2.4 Sintering

Sintering is among the important steps in P/M processing. It is process of consolidating either a loose aggregate of powder or a green compact of the desired composition beneath controlled conditions of temperature and time. In the early years of sintering research, debate went on about the meaning of the term sintering itself and it was defined as, “the system wherein powders are consolidated into strong and usually dense polycrystalline aggregates by heating”

The International Standards Organization (ISO) defines sintering as, “thermal treatment of a powder or compact at a temperature below the melting point of the major constituent, for the purpose of increasing its strength by bonding together of the particles”. Sintering operation is most
commonly implemented at temperatures ranging from 0.7–0.9 \( T_m \) (\( T_m \) is the melting temperature in kelvin of the metal or alloy). During sintering, the compact is frequently heated in a protective atmosphere such as argon or hydrogen. The individual powder particles, which were either loose or physically bonded together, are metallurgically bonded to yield structural part with the desired properties, several alterations take place during sintering like shrinkage, formation of solid solution and development of the final microstructure. In lots of cases, sintering results in a reduction/elimination of porosity, leading to densification, in different occasions, sintering may not result in any densification and in certain cases sintering can outcome in the progress of the compact (i.e. swelling). The sintered mass after suitable finishing operations such as machining, heat treatment can be put into service. Sintering is the end result of a sequence of powder processing operations, each of which may influence the final product. For the period of sintering, the green compact is consolidated and takes on its ultimate shape and microstructure. Sintering involves a number of processes, which may take place one after other or may just occur at the same time. Accordingly it is very difficult to clearly define and analyze the sintering process, in spite of a large amount of research efforts. There are many conflicting theories and data, and sintering remains to be an active field of research. The modern day trends in sintering can be had from the regular conferences devoted to the subject and from a number of books and studies on distinct aspects of sintering theory published regularly.

Sintering process is influenced by a few variables. In the case of a green compact, which is a multiphase constitution consisting of at least two phases, namely powder particles and porosity, factors such as the size and shape of the particles as well as those of the pores will influence sintering rate, each in its own way. Time is an important variable in P/M processing. This most often ranges from 10 minutes to several hours depending upon the
type of powder, its characteristics, size of the component etc. In the case of powder mixtures, sintering could take situation in the presence of a liquid phase where the sintering temperature is above the melting point of the low melting constituent. Examples are copper/tin alloys as well as tungsten carbide and cobalt-based tool materials. This process is termed as liquid phase sintering.

1.3 COMPOSITES

The term composite is used for describing materials which are semi-homogeneous and have superior mechanical and physical properties than those of their components. The matrix of a composite can be a metal, ceramic or polymer. Furthermore, composites can be grouped on the basis of the reinforcements provided (Schoutens & Tempo 1982).

1.3.1 Types of Composites

a) Fiber composites
b) Particle composites
c) Flake composites
d) Laminar or layered composites
e) Filled composites

1.3.2 Classification of Composite Materials

1.3.2.1 Metal Matrix Composites (MMCs)

The matrix phase for a MMC is a metal often which is ductile. MMCs are manufactured with aims to have high strength to weight ratio, high resistance to abrasion and corrosion, resistance to creep, good
dimensional stability and high temperature operability. MMCs are used in industries like automobile and aerospace. Mostly, aluminium is used as the metal matrix.

1.3.2.2 Ceramic Matrix Composites (CMCs)

This class of composites contains ceramic materials as matrix phase. CMCs are developed primarily to improve the fracture toughness of ceramic materials. This makes the CMCs to be used in extreme environments of high temperature and stress state. The dispersed phase plays a major role in preventing the propagation of cracks. This dispersed phase can be fibers, particles or whiskers.

1.3.2.3 Polymer Matrix Composites (PMCs)

They contain polymer as the matrix phase and fibers such as E-glass, carbon or aramid as the reinforcing phase. The different varieties of PMC mostly used are Glass Fiber-Reinforced Polymer (GFRP) composites, Carbon Fiber-Reinforced Polymer (CFRP) composites and aramid fiber-reinforced polymer composites. The most commonly used polymers as matrix are vinyl esters and polyesters.

1.3.3 Manufacturing Methods of Composites

1.3.3.1 Powder blending and consolidation

The frequently used technique for the preparation of discontinuous reinforced MMCs is the powder blending and consolidation. In this process, powders of the metallic matrix and reinforcement are first blended and fed into a mould of the desired shape. Blending can be carried out dry or in liquid suspension. Pressure is then applied to further compact the powder (cold pressing). The compact is then heated to a temperature, which is below
the melting point but sufficient to develop significant solid state diffusion (sintering). After blending, the mixture can be processed by hot pressing or Hot Isostatic Pressing (HIP) to achieve high density. The consolidated composite is then available for secondary processing. Attaining a homogeneous mixture during blending is a critical factor because the discontinuous reinforcement tends to persist as agglomerates, with interstitial spaces too small for the penetration of the matrix particles.

### 1.3.3.2 Consolidation diffusion bonding

This technique is typically used to manufacture fiber reinforced MMCs from sheets, foils, powder, powder tape or wire of matrix material. The techniques of assembling reinforcement fibers and matrix alloys are determined by the fiber type and fiber array preform method. In the case of monofilaments, such as SiC and B₄C, parallel arrays with controlled fiber-to-fiber spacing are generated using drum winding, weaving with metallic ribbons, or feeding one or more filaments into a continuous process. Tow based fibers, such as alumina or graphite is usually drum wound for a continuous pay out. Matrix materials can be supplied to the composite assembly as separate constituents (e.g., foils, powder mat or tape, wires etc..), or applied directly to the fiber array (e.g., vapour deposition, plasma spray). The composite elements are assembled by layering the fiber array and matrix plies, to realize a predetermined fiber orientation and composite thickness. Composite consolidation is achieved by applying high pressure, in a direction normal to the ply surfaces and a temperature adequate to produce atomic diffusion of the applicable matrix alloy. This method is performed in a vacuum environment.
1.3.3.3 **Vapour deposition**

Prominent among the vapour deposition techniques for the fabrication of MMCs is Electron Beam/Physical Vapour Deposition (EB/PVD). This process comprises of a continuous passage of fiber through a region of high partial vapour pressure of the metal to be deposited, where condensation takes place so as to produce a moderately thick coating on the fiber. The vapour is produced by aiming a high power (10kW) electron beam onto the end of a solid bar feedstock. A unique advantage of this technique is that an extensive choice of alloy compositions can be used. An additional advantage worth note is that, there is a little or no mechanical disturbance of the interfacial region, which may be rather important when the fibers have a diffusion barrier layer. Composite fabrication is typically completed by assembling the coated fibers into a bundle or array and consolidating in a hot press.

1.3.3.4 **Stir Casting**

Stir Casting is a liquid state technique of composite materials fabrication, in which a dispersed phase (ceramic particles, short fibers etc.,) is mixed with a molten matrix metal by means of mechanical stirring.

1.3.3.5 **Squeeze casting and squeeze infiltration**

In squeeze casting, a hydraulically triggered ram applies a low controlled pressure to the molten metal, to achieve infiltration of the preform without damaging it. Infiltration may or may not be vacuum assisted. Once infiltration is complete, high pressure is applied to remove the shrinkage porosity, which can occur when the liquid metal contracts as it transforms into the solid state. This ample consolidation, or absence of porosity, endows
the squeeze cast metal matrix composite materials with exceptional mechanical properties.

1.3.3.6 Spray deposition

Numerous methods have been developed under this classification, in which a stream of metal droplets impinges on a substrate in such a way as to construct a composite. If the reinforcement is particulate, it can be fed into the spray. The matrix spray can be applied to an array of fibers. The methods used fall into two discrete classes, depending on whether the droplet stream is produced from the molten bath, or by continuous feeding of cold metal into a zone of rapid heat injection.

1.3.3.7 Slurry casting (Compo casting)

Liquid metal is stirred as solid reinforcement particles are added to the melt to produce slurry. Stirring continues as the melt is cooled till the metal itself becomes semi-solid and traps the reinforcement particles in a uniform dispersion. Further cooling and solidification then takes place without additional stirring. The slurry may be moved directly to a shaped mould prior to complete solidification, or it may be allowed to solidify in a billet or rod shape, so that it can be reheated to the slurry form for further processing by techniques, such as die casting.

1.3.3.8 Reactive processing (In-Situ composites)

There are several different processes that would fall under this category. Directional solidification of eutectics in which one of the phases solidifies in the form of fibers is one such process. The inherent limitations in the nature and volume fraction of the reinforcement and the morphological instabilities associated with thermal gradients have resulted in a decrease in the interest in these types of composites. Exothermic reactions,
such as directed metal oxidation, are one family of processes for the production of in-situ composites. The foremost advantage of this class of composites is that the in-situ reaction products are thermodynamically stable.

1.4 PRECURSORS USED FOR THIS INVESTIGATION

1.4.1 Copper Matrix

Copper is a chemical element with symbol Cu (from Latin: cuprum) and atomic number 29. It is a soft, malleable and ductile metal with very high thermal and electrical conductivity. A freshly exposed surface of pure copper has a reddish-orange colour. It is used as a conductor of heat and electricity. In this experimentation, Copper was used as a matrix material.

<table>
<thead>
<tr>
<th>S. No</th>
<th>Physical properties</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Molecular Formula</td>
<td>Cu</td>
</tr>
<tr>
<td>2.</td>
<td>Atomic number (Z)</td>
<td>29</td>
</tr>
<tr>
<td>3.</td>
<td>Density</td>
<td>$8960 \text{ kg/m}^3$</td>
</tr>
<tr>
<td>4.</td>
<td>Melting point</td>
<td>$1357.77 \text{ K}$</td>
</tr>
<tr>
<td>5.</td>
<td>Heat of fusion</td>
<td>$13.26 \text{ kJ/mol}$</td>
</tr>
<tr>
<td>6.</td>
<td>Heat of vaporization</td>
<td>$300.4 \text{ kJ/mol}$</td>
</tr>
</tbody>
</table>

1.4.2 Reinforcements

1.4.2.1 Carbon Nanotubes (CNTs)

Nanotubes are members of the fullerene structural family. Their name is derived from their long, hollow structure with the walls formed by one-atom-thick sheets of carbon, called graphene. CNTs are valued in industrial applications for their self-lubricating and dry lubricating properties. Multi-walled Carbon Nanotubes (MWCNTs) consist of multiple rolled layers (concentric tubes) of graphene.
1.4.2.2 Nano Boron Carbide

Boron carbide (B₄C) is an extremely hard boron-carbon ceramic material used in tank armour, bullet proof vests and numerous industrial applications. It is one of the hardest materials known, behind cubic boron nitride and diamond. Boron carbide was discovered in the 19ᵗʰ century as a by-product of reactions involving metal borides; however, its chemical formula was unknown. It was not until the 1930s that the chemical composition was estimated as B₄C (Ridgway & Ramond 1934). There remained, however, a controversy as to whether or not the material had this exact 4:1 stoichiometry. The B₁₂ structural unit, the chemical formula of “ideal” boron carbide is often written not as B₄C, but as B₁₂C₃ and the carbon deficiency of boron carbide is described in terms of a combination of the B₁₂C₃ and B₁₂C₂ units (Musiri 2007, Patnaik 2002). Boron carbide is known as a robust material having high hardness, high cross section for absorption of neutrons (i.e. good shielding properties against neutrons), stability to ionizing radiation (Vladislav 2011) and most chemicals. Its fracture toughness (3.5MPa·m¹/²) approach the corresponding values of diamond (5.3MPa·m¹/²) (Zhang et al. 2001).

1.5 TRIBOLOGY

Tribology was first reported by Jost, England, in 1966. The word tribology derived from the Greek word tribos which means rubbing (Williams 1994). It is defined as the science and technology of interacting surfaces which might be in sliding and/or rolling motion. It has three major parts (a) Friction (b) Wear and (c) Lubrication. It requires knowledge from various subjects such as Physics, Chemistry, Mathematics, Thermodynamics, Heat Transfer, Material Science etc., to understand the tribological behaviors. Many failures of metals take place because of
tribological problem when compared to mechanical problems similar to fracture, plastic deformation and fatigue.

1.5.1 Friction

It is defined as resistance encountered when one body moves (either sliding or rolling) tangentially with respect to another (Bhushan 1999 & Singer 1992). This resistance force acting opposed to its motion is called Friction force as shown in Figure 1.3. The ratio of Friction force \( F \) to Normal load \( N \) is termed as coefficient of friction \( \mu \). The friction is not a property of material; it is a system response. More commonly, when two clean surfaces (without any oxide films and adsorbates) are contacted, it displays very high friction (Bhushan1999). It varies from 0.001 (for light loaded rolling bearing) to greater than 10 (clean metal sliding against itself in vacuum environment).

!(Source: Bhushan1999)

Figure 1.3 Friction force required to cause relative motion by (a) sliding (b) rolling
Generally, there are two types of friction: (a) Dry friction and (b) Fluid friction. In dry friction, contact surfaces are free from lubricant. If the contact surfaces are partly or fully separated by the lubricant, then it is called fluid friction. The friction is largely controlled by (1) kinematics of the surfaces in contact (i.e., the direction and the magnitude of the relative motion between the surfaces in contact), (2) externally applied load and displacements, (3) environmental conditions, (4) surface texture, and (5) material properties (Suh 1986).

1.5.2 Laws of Sliding Friction

The sliding contact friction is always higher than the rolling contact friction. This led to the conclusion that the static coefficient of friction ($\mu_s$) is greater than the kinetic coefficient of friction ($\mu_k$). The empirical laws of friction are given below:

**First law**: The friction does not depend on the normal load.

**Second law**: The frictional force is independent of the obvious area of contact between the contact surfaces.

**Third law**: The kinetic friction force is independent of sliding velocity once the motion starts.

These three legal guidelines don't couple in all occasions and for all metal pairs. For illustration, in copper sliding adjacent to copper in air, the coefficient of friction is low at low loads and it is greater at high loads. At low load, copper oxidizes in air and produces low shear strength oxide film which separates the metal contact surfaces. Nonetheless, at high loads, the oxide film breaks down and metal to metal contact occurs which is responsible for high friction (Whitehead 1950). On the other hand,
composite in air exhibits low friction at high loads. The reason for this high friction is because of increased surface roughness and a large quantity of wear debris (Bhushan 1999). Conversely, the coefficient of friction will not be constant for all metals. The high strength materials have high shear strain which results in low real area of contact and exhibits low friction (Bhushan 1999). The copper composite exhibits increase in friction proportional to the sliding speed which is against the third law of friction (Rabinowicz 1971). Hence, the friction laws will obey limited number of pairs under explicit conditions such as temperature, humidity, contact pressure and sliding speed. The coefficient of friction of many pairs depends on normal load, sliding speed and working environment.

1.5.3 Important Factors Affecting Sliding Friction

1.5.3.1 Surface defects

A portion of the normal surface abnormalities are shown in Figure 1.4. The surface deposit occurs because of synthetic responses with the environment. The surface scratches may happen due to improper machining and improper treatment of surfaces. The cleavage steps occur due to cryogenic temperatures. The etch pit is a type of surface imperfection which happens as a result of chemical etching. During chemical etching process, the high energy sites react more rapidly than bulk material and create pits. All grain boundaries are high energy sites on a solid surface and go about as an anomaly, particularly the region at which two adjacent grains meet (Buckley 1981).
Figure 1.4 A few models of surface irregularities of metal surfaces

1.5.3.2 Surface roughness

One of the early hypotheses state that friction is related to surface roughness. The roughness theory explains that the frictional force is equivalent to the force required to climb up the asperity of slope. However, it is demonstrated that asperities undergo deformation because of the sliding activity instead of just sliding over each other (Suh 1986). The impact of surface roughness on friction is the largest at light loads (Archard 1957). From the experimental perceptions, it was found that friction coefficient increases and decreases with surface roughness for hard materials and soft materials respectively. The most widely accepted friction theory states that “when two surfaces are brought into contact under external load, energy is required to move relatively quite considering the work to be performed against mechanical interlocking of asperities or to break micro-welds. The contact intersection get broken amid the relative movement or ploughing of
softer material by asperities of harder material, prompting physical changes (micro and macro-scale deformation) of contacting surfaces” (Bowden & Tabor 1950). Figure 1.5 displays a schematic view of the real area of contact between two surfaces.

Figure 1.5  A schematic view of real area of contact between two bodies

Bowden & Tabor (1950) performed an electrical conductance experiments and assessed the real area of contact between stationary surfaces. Experiments confirmed that the real area of contact was very small and it varied with the function of pressure. The real area of contact might be less than one thousandth of the obvious area of contact. The real area of contact is not enormously influenced by the size, shape and degree of roughness of the surface. It depends more commonly on the pressure. However, experiments with moving surfaces showed that the real area of contact was not consistent but was fluctuating rapidly during sliding. The character of those junctions relied on the relative physical properties of the two metals.
1.5.3.3 Hardness

It is a good known incontrovertible fact that severe damage occurs on the surface regions (i.e., at asperity contacts) when a soft metal is sliding over a harder surface. It is tentatively demonstrated that there is no relationship between bulk hardness and friction. However, earlier report (Reid & Schey 1987) suggested that surface hardness would be relevant to coefficient of friction and is not a matter of lower for harder materials. The detailed experimental study has been performed on the impact of hardness on the frictional behavior of pure metals. Hard metals have lower frictional resistance as compared to the softer metals due to their stronger inter-atomic bonds (Mokhtar 1982). Besides, Mokhtar (1982) argued that the atomic bonds in harder metals are strong and as a result of the resistance, adhesion is increased which provides low frictional characteristics. It has been tentatively demonstrated that low friction coefficients are gotten with metals having high hardness, high elastic modulus and high resistance to plastic flow. The low friction for the hard surfaced metals is ascribed to the absence of plastic stream and decrease in the ability of adhesion. The friction force relies on the real area of contact and the shear strength of the contact asperities. When two hard surfaces are in sliding contact, its real area of contact is small and thus higher the shear strength at the contact asperities (Bowdon & Tabor 1950). Henceforth, the friction is low. As indicated by the experimental confirmation, the friction coefficient diminishes with expansion in elastic modulus. Any increase in elastic modulus results in decrease in the real area of contact and thus, reduction in the adhesion and friction. Likewise the shear modulus additionally influences frictional behavior of pure metals. The friction coefficient decreases with an increase in shear modulus. Metals that have low shear modulus show a bigger zone of metal transfer than those with high shear modulus.
1.5.3.4 Grain size

As the grain size decreases, the mechanical and chemical properties of metal become enhanced. As a result, nano-crystalline materials have best industrial significance and widespread use in advanced innovation technology. These materials are utilized as bulk as well as coatings to engineering substrates. However, the hardness is the predominant aspect in sliding friction which is increased inversely proportional to grain size.

Bregliozi et al. (2003) carried out an experimental study on the influence of atmospheric humidity and grain size on friction and wear of copper based composite using a ball-on-disk tribometer. The finely grained steel, (with a higher hardness) confirmed a relatively lower weight loss and low frictional coefficient than copper. An investigation of tribological characteristics of a thin nano-crystallized layer on a nano copper reported that the friction coefficient decreased and the wear resistance increased with the nano-crystallized surface layer (Wang 2007). The change in friction and wear properties were ascribed to the harder nano-crystallized surface layer, which reduced the degree of ploughing, micro-cutting and the degree of plastic removal. On the basis of earlier reports, it can be concluded that friction coefficient has a direct relation with decrease in grain size.

1.5.3.5 Surface energy

In dry and clean situation, adhesion and friction strongly depend upon surface energy of the materials. The metals which have excessive surface energies form adsorbed layer on its surface through reacting with gaseous and liquid particles in air (Rabinowicz 1999). The presence of adsorbed layer reduces surface energy of metals and likewise diminishes the friction coefficient. However, the distinctive results were observed in vacuum environment for the identical set of experimental conditions.
Moreover to, it has been experimentally proved that high surface energy metal exhibits low friction (Mokhtar et al. 1980). Low values of the coefficient of friction are related to low values of surface energy to hardness ratio.

### 1.5.3.6 Sliding speed and load

The quantum of energy at the interface increases with increase in load or sliding velocity. At high sliding speed, the contact surface reaches there crystallization temperature and the surface will be annealed. Bill & Wisander (1977) claimed that in the event of annealing with longer time, the crystallites could undergo grain growth at the surface. The effect of normal load on friction coefficient for copper sliding against copper in air was experimentally studied by Whitehead (1950). Copper shows lower friction at low loads therefore of oxide film formation. This oxide film successfully isolates the two metal surfaces. However, at high loads, the oxide film breaks down and shows higher friction. However, Blau (1992) found that stainless steel at very high loads in air exhibited low coefficient of friction. The large number of wear debris at the interface was once liable for lower friction coefficient. According to the literature, it is obviously understood that friction coefficient of metallic pairs increases with an increase in load at low loads because of oxide film breakdown and begins to drop at high loads because of interfacial alterations brought about by means of wear particles. The experimental results for the influence of sliding speed on frictional coefficient exhibit very low friction coefficient at high sliding speeds. A thin molten film is created at the contact area considering the very high flash temperature. This thin molten film acts as a lubricant between sliding surfaces, which results in very low friction coefficient obtained at higher sliding speeds. Mainly, the friction coefficient decreases with increase in sliding velocity. Formation of oxide layers on metal surfaces at high
temperature, which is triggered via sliding speed, outcome in low friction. Moreover, softening of the metal surface results in elevated ploughing in the softer material and displays high friction. Both these reasons make it not easy to expect the effect of sliding velocity on friction.

1.5.3.7 Environment

The contact surface environment assumes an essential part in friction coefficient. The frictional force between steel surfaces is much higher in argon atmosphere when compared to the ambient conditions (Endo & Goto 1978). The frictional investigations were carried out on copper and found that the friction coefficient in vacuum is much more (10 times) higher than the values obtained in air (Tsuya 1975). When two iron solid surfaces were heated in a vacuum procedure, the surfaces were clean sufficiently to form strong adhesive bonding between two surfaces and underwent seizure. The contacts asperities were adhered to in a single touch even at no load conditions. This kind of adhesive bonding can happen for metal contact with metal as well as for metals with non-metals such as diamond. However, the admission of oxygen or other surface dynamic gases reduces friction force. Tsuya (1975) reported that when surface oxidation was nil, broad welding and metal exchange occurred thus a result of high friction. In ambient condition, most metals oxidize and form oxide films, typically between 1 and 10 nm thick within a few minutes of exposure of clean surfaces. This oxide film goes about as low shear quality film and minimizes the metal to metal contact at asperities level and therefore prompts low friction. Conversely in vacuum condition, there is less chance of oxidation and direct contact existing from metal to metal surfaces. For this situation, depending upon the metallurgical compatibility, the level of adhesion governs the frictional behavior of the metals. The factors impacting friction on various types of pure metals (silver, platinum, copper, magnesium, iron, titanium,
aluminum) have been studied and suggested that the friction and wear are depended on the oxidation activity of the metals, atmospheric oxygen and also relative shear strength of the metal–oxide interface (Hiratsuka et al. 1992).

1.5.3.8 Temperature

The surface temperature of the metals in sliding contact will be particularly very high (Bowdon and Ridler 1956). They performed sliding friction experiments as an influence of sliding speed and load, and obtained interesting results. They observed that both sliding speed and load increased the surface (Flash) temperature. The Flash temperature used to be so much better than the bulk metal temperature. The surface and subsurface temperature is normally measured by inserted thermocouple technique. This flash surface temperature is adequate to cause recrystallization on the surface. It causes the physical, chemical and metallurgical changes on the contact surface resulting in friction changes. At elevated temperatures, the contact surface softens which results in superior flow, ductility, a larger real area of contact and better adhesion (Bhushan1999).

1.6 FULL FACTORIAL DESIGN (FFD)

Design of Experiments (DoE) is the well-known statistical technique applied in this method to determine the influence of process parameters by conducting the total number of experiments. Utilizing orthogonal arrays, a small number of experiments with the effects of a large number of variables were studied. Dry sliding wear behavior of copper hybrid nanocomposites with various parameters such as a material factor (intrinsic) and wear factors (extrinsic) has been selected for the optimization of friction coefficient (CoF) and specific wear rate (SWR) using design of experiment (Basavarajappa et al. 2007). The thrust actions related to the
development of composites with wear resistance that has an effect on the
scope of the present work was identified (wt.% of reinforcements, load and
sliding distance) and subsequent, research approach had been designed. For
developing and exploring the optimized design, an attractive tool (Full
factorial design) follows, a procedural approach, was selected in this study.

1.7  TAGUCHI DESIGN

The Taguchi design principle is an attractive tool follows a
procedural approach useful for planning and analyzing the optimized design.
If the inputs (parameters) selected for the experimental analysis are larger, a
number of experiments to be carried out must be higher. To overcome this
problem, a method which uses a special design of Orthogonal Arrays (OA)
to evaluate the entire key parameter with reduced number of experiments
was selected.

1.8  GREY RELATIONAL ANALYSIS (GRA)

Using this technique, the single performance grade can be greatly
achieved by optimizing multiple responses. Often, this method is helpful in
realizing the process optimization due to the systematic application of the
optimization tools and designing the experimental methods. This is one of
the current studies implemented to support the Taguchi method for ranking
the multiple quality performance characteristics. In this technique,
normalization is in the range between zero and one. This Grey relational
grade helps in computing the integrated Grey relational coefficients for each
of its corresponding performance. To normalize the original data in
sequence, the primary requirement is the data pre-processing to determine
the performance. This is an empirical relationship to find the grade for the
output responses such as wear and friction coefficient in determining the
optimized process parameters.
In this method, Grey relational grade is also important to study the strong dependence of wear rate and friction coefficient on the parameters. Basically, response of a system is analyzed on three different scales; (1) lower the better, (2) higher the better and (3) nominal the best. Depending on the type of response it is necessary to choose a corresponding formula to find out the normalized data values. In the current study, the response should attain a minimum to get a good combination of factors. If the response is higher, “higher the better” will be the performance of the multiple quality characteristics. In this work, “lower the better” has been considered because both the responses of the hybrid composite must be a minimum value. The overall performance of the composite depends on the processing parameters like applied load and the sliding distance.

1.9 SCOPE AND APPLICATION OF THE THESIS

Metal matrix composites are exciting areas of research in which reinforcements of hard ceramic particles are added to improve wear resistance and mechanical performance. Copper is considered as an ideal matrix for composites due to its properties of high electrical and thermal conductivity and is widely used in industrial applications. However, the relatively low hardness and strength and poor wear resistance of copper are crucial factors that limit its extensive application. These distinct shortcomings could be avoided by incorporating carbon and nano-ceramic particles into the copper matrix, i.e., oxides, carbides and borides used as tribological components, for use of this material in brushes for motors and contact strips for pantographs for railway current collectors. Selection of the correct reinforcement for enhanced physical properties of composites has attracted scientific and technological interest in carbon nanotubes, which when paired with a metal matrix, offer significant advantages over most existing materials. Recently, many researchers have shown that carbon
nanotubes (i.e., a three-dimensional carbon material) have higher electrical and thermal conductivities and can be combined with polymer, ceramic and metal matrices as a strengthening phase using various methods to obtain composites with superior properties. In addition, ceramic particles are preferentially considered as a secondary strengthening phase in the metal matrix to hybridize the composite, which enhances the mechanical properties as well as the wear resistance.

Previously, work has been carried out by fabricated composites in several routes, i.e., spraying, hot pressing, hot extrusion and spark plasma sintering, to obtain excellent mechanical properties. Strengthening of copper composites without the agglomeration of CNTs is the most important challenge. Ravindran et al. (2012) found that the particle size and proportion of reinforcement to matrix medium have significant influences on the mechanical properties. Composites reinforced with the addition of nano-ceramic particles exhibit better specific strength due to their excellent hardness. Specimens prepared via powder metallurgy are always porous, which decreases the strength of the sintered samples. However, hard nano-reinforcements can be employed to improve the mechanical properties and fill the porous regions of composite specimens prepared via powder metallurgy methods. Ravindran et al. (2013) reported that the wear behaviour of aluminium composites, improved by increasing the nano-ceramic addition for materials was prepared via the powder metallurgy method. A number of research groups have investigated the use of P/M for the production of metal matrix nanocomposites incorporated with CNTs.

However, a copper matrix reinforced with multi-walled carbon nanotubes and boron carbide for improvement of wear, mechanical properties and machining behaviour prepared using powder metallurgy has not yet been reported. The main objective of this work is to evaluate the
combined effect of the higher hardness material (nano B₄C: 0-2%) and superconductive material (MWCNTs: 2%) as reinforcements in the copper matrix for the improvement of various mechanical and electrical properties for pantograph application. In the current work, an attempt is carried out to determine the wear rate, electrical resistivity, mechanical properties, material removal rate and tool wear rate of composites prepared using the powder metallurgy method. Addition of B₄C particles increases both mechanical strength and wear resistance of copper matrix. But the consequent increases in hardness make the machining difficult and also reduce the electrical conductivity. On the other hand, addition of MWCNT facilitates easy machining and results in increase in electrical conductivity of copper composites compared to pure copper. These are the reasons for choosing hybrid reinforcements.

1.10 OVERVIEW OF THE THESIS

This chapter is organized into seven chapters. A brief chapter wise outline of the thesis is given below:

Chapter 1 explains an introductory survey of powder metallurgy method, tribology and fabrication of composites by various techniques. The significance of Cu-MWCNT-nano B₄C composite and its application are briefly discussed.

Chapter 2 gives a literature survey of powder metallurgy, tribological behavior of copper based composite and electrical resistivity measurement. The literature regarding the machining behaviour of the composites and Taguchi analysis on wear behaviour of Copper hybrid nano composites is also reported.

Chapter 3 reports a complete detailed scheme of investigation using a flow chart. This chapter reports the method of preparation of sample
specimen, experimental technique used for the preparation and testing of Cu and Cu-W composite preforms in detail.

Chapter 4 gives the importance of full factorial design and also predicts the percentage of contribution of each factor on the results using ANOVA.

Chapter 5 explains the prediction behavior of Copper hybrid nano composites using Taguchi followed with Grey relational analysis. It also identifies the most influencing combination of parameter on the response by rank order.

Chapter 6 deals with the experimental results of outcomes derived from wear test and machining. Tribological behavior, worn surface and debris particles study are carefully discussed in detail. Also material removal rate and tool wear rate during machining is also discussed.

Chapter 7 is the concluding chapter in which the major contributions of the research study are summarised. Guidelines for future work are also included in this chapter. A bibliography of the literature relevant to the research study is listed at the end of the thesis.

1.11 SUMMARY

In the current chapter, the various techniques for the fabrication of composites were studied and optimal method for the fabrication of copper hybrid nano composite was decided as the powder metallurgy route and it is explained elaborately. Optimization techniques like full factorial design and taguchi analysis for the prediction outstanding results and factors affecting wear behaviour were also studied.