CHAPTER 2 LITERATURE REVIEW

2.1 Composites

Composites are the materials that contain two or more physically distinct and chemically separable phases as an unified combination. One is the matrix phase and other(s) is the reinforcing phase. Matrix phase is the continuous phase which holds the reinforcing phase in three dimensional network. Composite materials have the ability to combine the properties of reinforcing phase with that of the matrix phase such that the resultant properties are better than the properties of their monolithic counterparts. Their properties can be tailored, depending on end application, through the judicious selection of reinforcement phase, matrix phase, and processing technique. As a result, composite materials have the capability to serve a wide spectrum of applications in aerospace, automotive, biomedical, sports and electronics areas.

Composites have been classified into several categories:-

1. On the basis of matrix
    - 1.1. Metal matrix composites (MMCs)
    - 1.2. Ceramic matrix composites (CMCs)
    - 1.3. Polymer matrix composites (PMCs)
2. On the basis of reinforcing phase
    - 2.1. Fibre (long or short)
    - 2.2. Whiskers
    - 2.3. Particulates
    - 2.4. Laminates or Sheets
    - 2.5. Interconnected reinforcement
    - 2.6. Singular metal core reinforcement

The aspect ratio is used to distinguish between these different forms of reinforcements. The aspect ratio is the ratio of length to diameter (or thickness) of the reinforcement.

2.2 Metal matrix composites

Metal Matrix composites are a class of composite material in which matrix is either a metal or a metallic alloy and reinforcement(s) may be metallic, ceramic, refractory metal, intermetallic, or semiconductor. CMCs are mostly used in high temperature areas. MMCs have higher ductility than CMCs and better environmental stability than PMCs. In comparison with PMCs, MMCs show high transverse strength, high stiffness and shear strength, better high temperature properties, and better fire resistance. MMCs also have an edge over CMCs in terms of high toughness, moisture resistance, high electrical and thermal conductivity, resistance to thermal shock and easy to join, shape and manufacture.

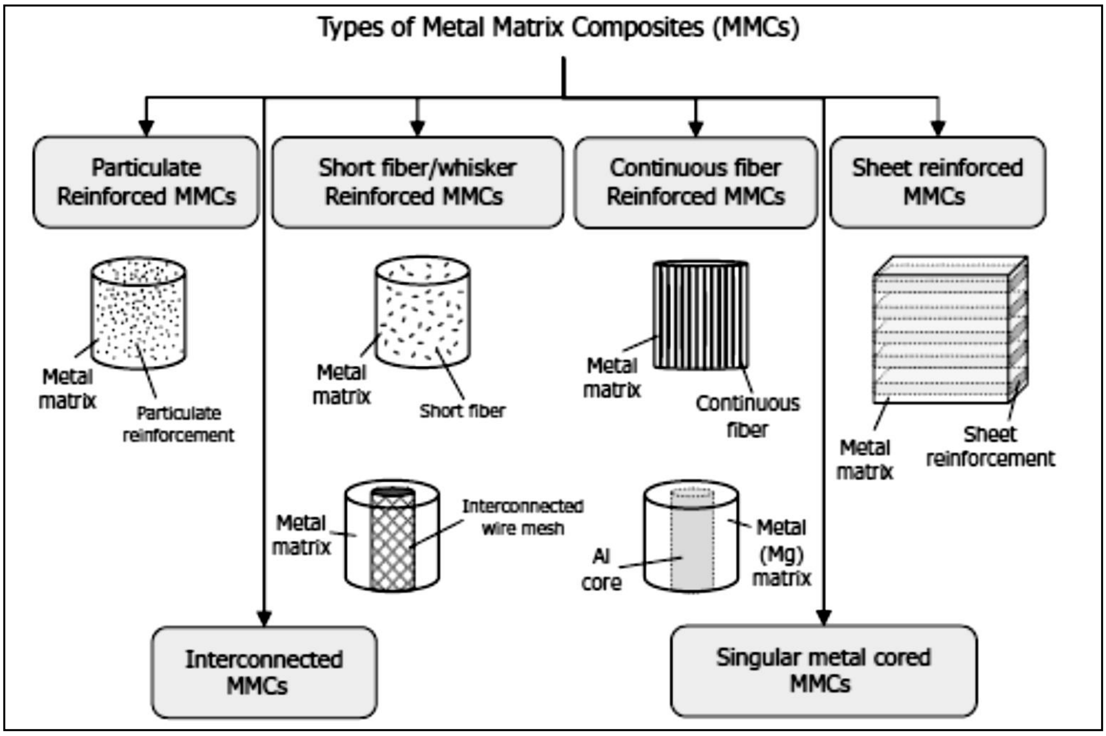

Figure 2.1 Different Types of Metal Matrix Composites [23]

2.2.1 History and applications of metal matrix composites

In 1950s and the first half of 1960s, the first serious attempts to create MMCs were made. The main goal was to significantly increase metallic materials' structural efficiency while maintaining their benefits, such as high chemical inertness, high shear strength, and good property retention at high temperatures. Throughout the 1960s and the beginning of 1970s, significant works on fibre-reinforced MMCs were made due to the development of high-strength monofilaments, first made of boron and then silicon carbide (SiC) [24]. Important applications were established, including 243 structural components on the space shuttle orbiters, despite the fact that these were extremely expensive and had poor reproducibility [25]. This stage of MMCs discovery and development came to an end as a result of major funding reductions brought on by the famous economic recession of the early 1970s.

The development of discontinuously reinforced MMCs with SiC whisker reinforcements was restarted in the late 1970s. The idea of particle reinforcements was developed as a result of the high cost of whiskers and the challenges associated with preventing damage to them during consolidation. The new materials offered strength and stiffness but at a significantly cheaper cost and with much simpler production. Through the 1980s, both discontinuous and fibre-reinforced MMCs saw a revival. By 1990s, MMCs were started to be included in automotive, thermal management, aerospace and other applications [26]. For instance, aluminium cylinder liners have been in mass production since 1990 in the Honda Prelude 2.3 litre engine. Discontinuously reinforced titanium (DRTi) MMCs have been used as automotive valves since 1998 in the Toyota Altezza 2.0 litre L-4 engine. A number of electric and hybrid vehicles, such as the Toyota RAV4, Ford Prodigy, and the General Motors Precept, are reported to use MMC brake components [25]. In past two decades, significant research have been done to develop particulate and whisker reinforced composites of aluminium, magnesium, iron, copper, etc.

2.2.2 Particulate reinforced metal matrix composites (PRMMCs)

Particulates or discontinuously reinforced MMCs (PRMMCs) consist of both particulates and short fibres or whiskers. PRMMCs of aluminium and magnesium have a lot of improved properties than their unreinforced metal or alloy [27]. Particulate or discontinuously reinforced MMCs assumed significant interest because of their advantages over other types of MMCs. PRMMCs exhibit isotropic properties. This is achieved when the reinforcements phase is homogeneously distributed in the matrix phase. Over the years many ways have been researched to make sure the distribution of reinforcements is even.

PRMMCs can be easily synthesized using conventional processing routes like casting, powder metallurgy, spray forming, semi solid casting etc.[28]. Each of these techniques can be employed based on the application and required properties of composites. Moreover, PRMMCs can be easily machined using conventional and non-conventional machining processes [29]. PRMMCs have also mitigated many problems of continuously reinforced MMCs like non uniformity in microstructure, fibre damage, large interfacial reactions, fibre – fibre contact, etc.

Ceramics particles when used in low volume fraction can reinforce the strength and toughness of parent metal or its alloys without significant loss of ductility. This makes considerable improvement in properties especially for low density metals like magnesium, aluminium and titanium. In fact, the properties were comparable to most of their engineering alloys. The judicious selection and addition of ceramic particulate reinforcements have improved the strength, damping capacity, wear resistance and high temperature stability of composites. These properties can also be tailored by varying the volume fraction, size and shape of reinforcements.

Many PRMMCs have been developed recently and many have already found applications in aerospace, automotive, sports and electronics fields.

2.3 Selection of matrix material

Matrix is the continuous phase in composites material that surrounds and binds the reinforcing phase and gives three dimensional shape to the composites. It takes up the external load and transfers the load to reinforcing phase. Matrix also protects the reinforcing phase against external effects like mechanical damage, erosion and corrosion caused by the surrounding medium or reinforcement-reinforcement contact. Among all the light weight metals, aluminium and its alloys are the most commonly used metallic matrices but in recent years the trend is slowly shifting towards magnesium and titanium owing to their lightweight properties. Magnesium being the lightest metal to be used in structural application has been underutilized for engineering applications. Pure magnesium has been taken as a choice of matrix material in this study. Table 2.1 enlists some general properties of pure magnesium.

Table 2.1 General Properties of Pure Magnesium

Atomic number	12
Density (at 20 °C)	1.74 g/cm^3
Atomic weight	24.305
Atomic radius	0.160 nm
Crystal Structure	Hexagonal Close-Packed (HCP)
Melting point	650 °C
CTE (20 – 100 °C)	26.1 x 10^{-6} °C^{-1}
Elastic modulus (Tension)	45 GPa
Thermal conductivity (at 27 °C)	156 W m^{-1} K^{-1}
Poisson ratio	0.35

Magnesium is found in abundance, both in earth's crust and sea water. It has a density of 1.738 g/cm^3, which is approximately one-fourth the density of steel and two-thirds of aluminium. Moreover, it has high damping capacity, fairly good electromagnetic shielding and is easily machinable. This makes Mg the perfect choice for weight reduction applications. But its applications are limited due to low strength and ductility, poor creep and wear resistance, high temperature instability and poor corrosion resistance. Incorporating discontinuous ceramic reinforcements have circumvented the negative aspects of Mg to a large extent by imparting strength, ductility and wear properties in the MMCs [13]. Some hard ceramic reinforcements have also improved high temperature stability and corrosion resistance [30].

2.4 Selection of reinforcement

Ceramics particulates are the most commonly used reinforcements for PRMMCs because of their superior mechanical properties and low cost. There are wide variety of ceramic reinforcement available but its selection depends on several factors like elastic modulus, strength, density, melting temperature, thermal stability, and coefficient of thermal expansion, size and shape, compatibility with matrix material, cost and the area of applications. Table 2.2 enlists some commonly used reinforcements for MMCs.

Table 2.2 Commonly used reinforcements for MMCs

Al_2O_3	HfC	Si_3N_4	UO_2
AlN	MgO	SiO_2	VC
BeO	Mo	SnO_2	WC
B_4C	$MoSi_2$	TaC	WSi_2
C	Mo_2C	ThO_2	Y_2O_3
CeO_2	Ni	Ti	ZrB_2
CNT	Si	TiB_2	ZrC
Cu	SiC	TiC	ZrO_2

SiC is the most commonly used reinforcement with magnesium and its alloys. It has been used in all shapes and forms. In the present work, Magnesium Oxide (MgO) has been selected as the reinforcing phase. Magnesium oxide is a hard ceramic having high compressive strength which can potentially improve the hardness, strength and wear resistance of composites. It remains physically and chemically stable even at high temperatures. Thus, it can also be used to impart elevated temperature stability when reinforced with magnesium. It is also cheap and easily available. Table 2.3 enlists some of the properties of MgO.

Table 2.3 General Properties of Magnesium Oxide

Molar Mass	40.304 g/mol
Density	3.58 g/cm^3
Poisson ratio	0.35 - 0.37
Melting point	2852 °C
CTE (20 - 100 °C)	11.6 x 10^{-6} °C^{-1}
Thermal conductivity (at 27 °C)	40-60 W m^{-1} K^{-1}
Compressive Strength	833 – 1666 MPa

2.5 Magnesium based metal matrix composites

Magnesium alloys and composites have been studied a lot by researchers around the globe. There had been great success in developing Mg alloys and many of them have already found applications in field of aerospace [31], automobile [32-34], sports [23], electronics [35] etc. However, most of the Mg alloys fail to retain properties above 200 °C. There are few Mg alloys which give high temperature stability but the cost is very high. For instance, Mg elektron series of alloys can be used up to 300 °C [23]. The wear resistance of Mg alloys is also low. So, reinforcing Mg or its alloy with ceramic particulates can improve these properties. Despite having high potential to replace many parts in weight reduction applications, use of Mg MMCs are still limited. This is due to high cost of production of composites. There must be a reasonable cost of production versus composite performance relationship for any practical application of MMCs. The full potential of Mg composites can only be realised when an effective low cost technique is developed for its production. Over the years many methods have been invented, modified and combined for the production of Mg MMCs. Some of the major processing techniques are briefly discussed.

2.6 Processing techniques for particulates reinforced magnesium MMCs

A large number of processing techniques have been attempted over the years for Mg MMCs. Each techniques have their own advantages and disadvantages. Based on required material properties and end cost of the product, processing techniques are selected for particular application. The selection of technique is very crucial as material having same composition shows different properties when manufacturing process changes.

Magnesium MMCs have been mainly processed by stir casting, squeeze casting, powder metallurgy, spray forming, in situ methods, infiltration methods etc. These techniques are briefly discussed in the following sub sections.

2.6.1 Stir Casting

Stir casting process as shown in Figure 2.2 generally involves the incorporation of particulates reinforcement in stirred molten metal or alloy and then the molten metal or alloy containing reinforcements can be sand cast, die cast, or permanent mould cast. It is very promising method for Mg MMCs especially for large scale production. High volume fraction of reinforcements up to 30 % can be incorporated in the composite with this method [36]. The cast product can then be extruded for further homogenization of particles and property improvement.

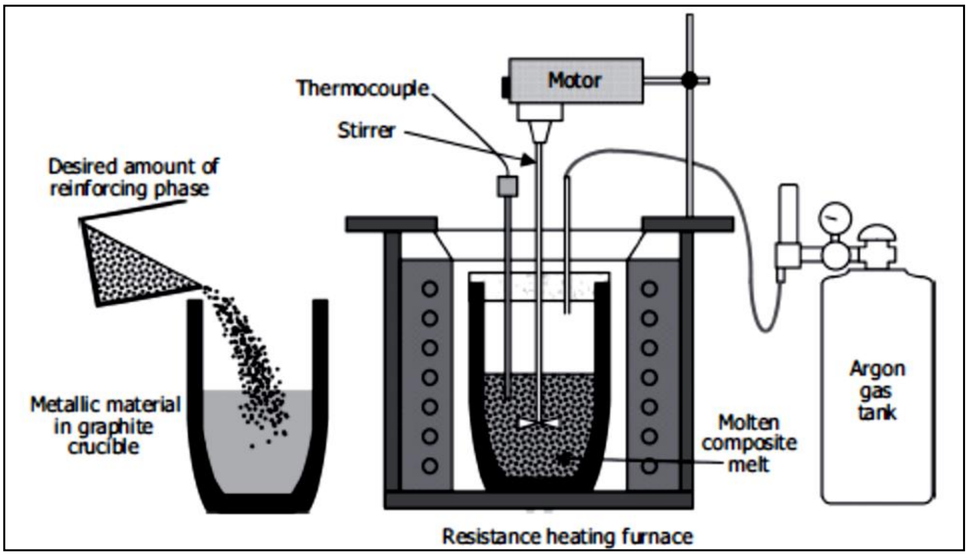

Figure 2.2 Schematic of Stir casting method [37]

Many MMCs like CP – Mg/SiC [36] , AZ31/SiC [38] . WZ73/ SiC [39] etc. were developed using this process.

However, there are several problems associated with this process. The main problem with stir casting is segregation of reinforcements phase due to surfacing or settling of particles during melting and casting stage. This may lead to inhomogeneous distribution of phases. Another problem is that of inclusions and gas entrapment in the cast thereby deteriorating the properties. These problems were resolved to some extent in two step stir casting process [40]. In two step stir casting technique, the material is heated to melt stage and then is cooled between solidus and liquidus temperature i.e. semi solid stage. At this stage pre-heated reinforcements are added. The slurry is again heated to melt stage and stirred which reduces the problem of segregation. This process also breaks the gas layer around the reinforcements thereby reducing gas entrapment problems. This process was employed for AZ91/TiB_2 [40] , AZ91D/SiC [41], etc. composites.

Stirring is the most important stage in stir casting as it determines the homogeneous distribution of phases. Several researchers focused on different stirring methods for better reinforcement distribution like ultrasonic stirring [42] , centrifugal force stirring [43] etc.

2.6.2 Spray deposition process

An atomized stream of molten material droplets is focused onto a substrate during spray forming or spray deposition to create bulk metallic structures as shown by a schematic in Figure 2.3. For the case of MMCs, the reinforcing phase is injected into atomised matrix material which gets dispersed in matrix. The droplets containing matrix and reinforcements are deposited on substrate to get MMCS. Many studies have been conducted where it was found that spray deposition process parameters highly influenced the microstructure and mechanical properties of composites.

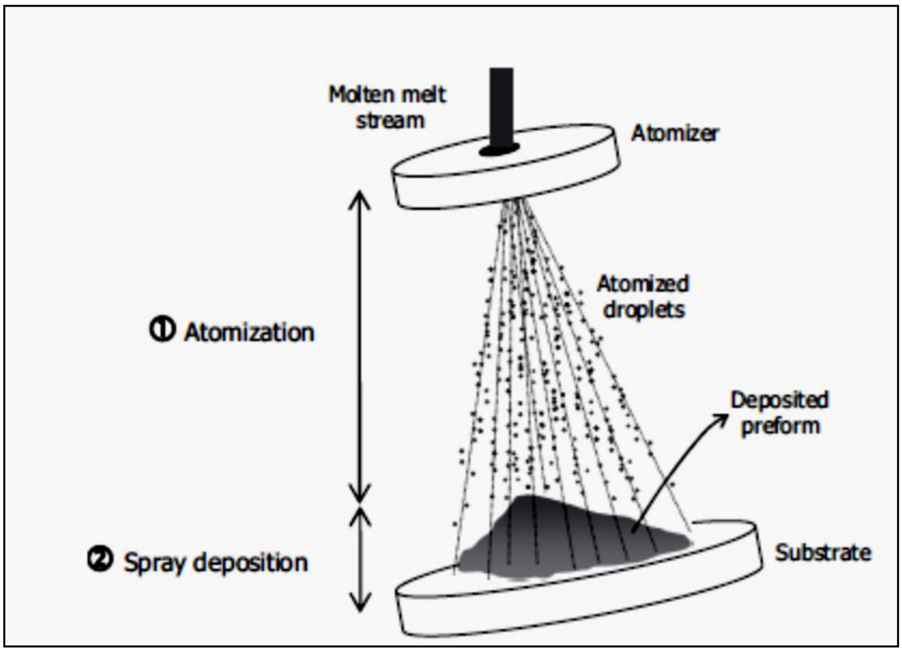

Figure 2.3 Schematic of Spray Deposition Technique for Mg MMCs [23]

Usually spray deposited MMCs showed fine grains, porosity and absence of brittle phases at the SiC/matrix interface due to the rapid solidification rate associated with this process. Several Mg MMCs have been fabricated with this process. For instance, QE22/SiC [44], Mg – 10% Ce /SiC composites [12].

2.6.3 Disintegrated melt deposition (DMD) technique

Disintegrated Melt Deposition **(**DMD) process was developed in 1990s. This process combines together the advantages of stir casting and spray deposition to produce bulk composite material. It involves heating and stirring the melt pool of matrix and reinforcement and then pouring it through orifice where the melt is disintegrated by argon jet and the droplets are deposited on substrate. The schematic of DMD process is shown in Figure 2.4.

Literature shows that DMD process has been utilised to develop composites having different size, shapes and types of reinforcement [45-47].

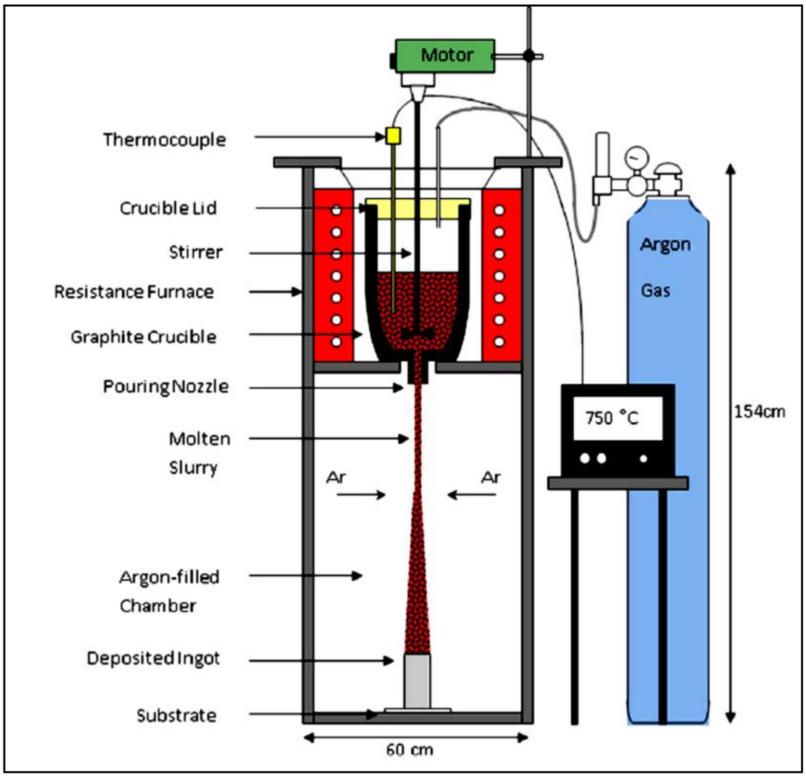

Figure 2.4 Schematic of Disintegrated Melt Deposition Technique [30]

2.6.4 In situ synthesis

In situ synthesis is a process wherein reinforcements are synthesised by reaction between elements or between elements or compounds in the melt pool at many different places. This leads to homogeneous distribution of reinforcements having surface free from contamination. This process works only if the expected reaction has favourable thermodynamic conditions. The reaction system should be carefully screened so that particles size can be controlled. The clean reinforcements formed improves the coherency and wettability with the matrix phase. Mg –TiC [48], Mg – TiB_2 –TiC [49], AZ91– MgO [50] composites have been fabricated using this process.

2.6.5 Melt infiltration techniques

Melt infiltration refers to the process in which molten metal of low density penetrates the gaps of highly porous ceramic compacts. The gases present in the pores of ceramic compact are slowly replaced by the molten metal. This process is suitable for composites fabrication because of its simplicity, high amount of ceramic content and near net shape fabrication. The filling of pores of preheated ceramic preform takes place either due to capillary action or by external forces (vacuum and pressure).

When infiltration takes place due to capillary forces (develops when liquid metal wets the ceramic particles), the process is called spontaneous or pressure less infiltration. Parts that require high reproducibility can be manufactured by this process. The reinforcing particles in this process are very evenly distributed throughout the matrix as these are not distributed by liquid. Wetting of the solid by the liquid phase is a necessary prerequisite for infiltration to proceed. High ceramic content and near net shape fabrication can be obtained with this process. Several composites system like Mg/TiC [51], AZ91/TiC-TiB_2 [52] have been fabricated with this process.

When the infiltration takes place due to external pressure, the process is called squeeze casting. In this process, a movable mould part (ram) is used to apply pressure on the molten metal forcing it to penetrate a porous preform of the reinforcing phase, placed into the lower fixed mould part. High pressure fills the voids quickly, thereby avoiding the solidification of melt (preform temperature is less than molten metal) before the complete penetration. Minimal reaction between the reinforcements and molten metal takes place due to short contact time at high temperature. The pressure in squeeze casting must be effectively controlled especially for Mg MMCs as high pressure may lead to turbulent flow of molten Mg causing gas entrapment and oxidation. Many Mg MMCs like Mg/SiC_w [53] and ZK51A/SiC_w have been developed by the process.

2.6.6 Powder metallurgy

Powder Metallurgy is solid state fabrication process where matrix and reinforcement powders are mixed, pressed, and sintered in a controlled environment or vacuum. This process is suitable for high volume incorporation of reinforcement phase in composites. The matrix alloy and reinforcements system which are immiscible in liquid state (difficult to fabricate by casting) can be fabricated easily by powder metallurgy. The mixing or blending stage of powders must be done carefully for homogeneous distribution of particles. The compaction of mixed powders are done either by uniaxial pressing or by isostatic pressing. The powders are in direct contact with die in uniaxial pressing unlike isostatic pressing where there is a pressurising medium of gas or liquid between powders and die. The uniaxial pressing can be done by cold compaction and hot compaction approaches. Isostatic pressing is also of two types i.e. cold (room temperature) isostatic pressing (CIP) and hot isostatic pressing (HIP).

Sintering of MMCs are usually performed by different methods. **Conventional resistance sintering** involves heating of compacts from outside (resistive heating element) so that heat gets transferred from outside surface towards inner core.

In **microwave sintering**, the compacts absorb the microwaves and heat is generated at inner core and transferred towards outer surface. A thermal gradient develops during above two sintering processes which results in microstructural and properties variation at core and periphery of compact. This problem is solved to a large extent in **hybrid microwave sintering process** wherein a microwave susceptible material like SiC powders are surrounded around compacts during microwave heating. SiC powders absorbs the microwave and heats the compact from the periphery towards the core. The compacts also absorbs microwaves and gets heated from within. Therefore, there is two directional heating which reduces the thermal gradient. This concentrates the heat and compact is evenly sintered.

Wong et al. studied the energy consumption for sintering of aluminium – magnesium based material using a rapid (hybrid) microwave sintering set up and compared with conventional sintering in the tube furnace. They concluded that there is around 96 % power saving in microwave furnace compared to conventional sintering for aluminium - magnesium based material [22]. This shows that cost of production can be greatly reduced using microwave sintering.

Spark plasma sintering is the pressure sintering technique in which axial pressure is applied with simultaneous heating of the powder sample by means of plasma generation occurring at the gap sites of the particles. Several Mg MMCs have been developed by powder metallurgy route like Mg/TiB_2 [54], Mg/B_4C [55], Mg/CNT [56] etc.

2.7 Studies based on synthesis of Mg/MgO composites

The first Mg/MgO composites was reported to be developed in France in 1962 for nuclear applications [57]. However, serious attempt to fabricate Mg/MgO composite were made in the year 2004 when Deng et al. fabricated in situ MgO and Mg-Zn intermetallic reinforced Mg composites by powder metallurgy route [58]. They studied the reaction mechanism behind the MgO and Mg-Zn intermetallic formation. They predicted a reaction model for the same in which Gibb's free energy of formation of the products and the diffusion constants of O, Zn and Mg atoms were the decisive factors for the formation of MgO and Mg–Zn intermetallic. They also concluded that in situ formation of the MgO and the Mg–Zn occured at ~ 410 °C and ~ 530 °C, respectively.

Then in early 2005 Ma et al. reported the development of MgO and Mg_2Cu in situ reinforced MMCs in Mg – CuO – Cu ternary system through powder metallurgy technique [59]. They concluded that CuO is reduced to Cu and some Mg gets oxidised to form MgO when sintered at 450 °C and when sample was sintered to 550 °C and water quenched, Mg - Mg_2Si nano eutectics were obtained. So, samples comprised of MgO nanowires and Mg – Mg_2Si nano eutectics spread in Mg matrix after the sintering process. These reinforcements were responsible for increase in hardness of composite. In another work the same year Ma et al. developed in situ MgO, Fe and FeO reinforced Mg matrix by same route of reaction sintering process [60]. From DSC and XRD results, they concluded that when Mg-Fe_3O_4 compact was sintered at 450 °C, Fe_3O_4 reduced to from Fe and FeO while some Mg gets oxidised to form MgO. When sintered at 630 °C, all Fe_3O_4 phase were converted to Fe nanoparticles and MgO nanowires. The final product containing Fe nanoparticles and MgO nanowires in Mg matrix improved the hardness. Up till now studies were mainly about understanding the in situ reaction mechanism and formation of phases at different temperatures.

The first insight into mechanical properties were carried out by Wei et al. who studied the creep behaviour of Mg-12Li-Al-MgO composite containing 0, 5, 10, 15 vol. % of MgO synthesised through casting by in situ reaction of B_2O_3 in Mg - 12Li - Al alloy melt [61]. They reported an increase in the creep resistance with increasing content of MgO particle and this improvement in creep performance was mainly attributed to fine MgO precipitates in alloy melt.

Goh et al. in 2005 synthesised Mg nanocomposites reinforced with 0.1, 0.2, 0.3, and 0.4 vol. % of MgO particles by powder metallurgy route [62]. It was reported that Mg nanocomposites were more thermally stable than pure Mg samples. Also, the increasing volume percentage of MgO nanoparticles improved the 0.2 % yield strength (YS). The ultimate tensile strength (UTS) and ductility remained nearly same for all compositions.

After two years Goh et al. again fabricated Mg/MgO nanocomposites by disintegrated melt deposition method followed by hot extrusion [63]. They concluded that the nano MgO particles improved the thermal stability of the composites as well as mechanical properties like hardness, tensile strength, and elastic modulus of nanocomposites compared to pure Mg. They also reported that MgO particles showed good interfacial bonding with Mg Matrix.

Khalajabadi et al. fabricated Mg/HA/MgO nanocomposites using a blend-cold press-sinter powder metallurgy technique to improve the bio-corrosion and mechanical properties of the resulting material [64]. Both hydroxyapatite and MgO vol. fractions were varied. Mg- 27.5HA-5MgO showed high compressive strength. They reported that decreasing hydroxyapatite (HA) content and increasing MgO content decreased the ultimate compressive strength (UCS) but compressive failure strength increased.

Cai et al. in 2018 synthesised in situ Mg/MgO nanocomposites by reactive cryo-milling and subsequent consolidation under high pressure of 6 GPa [17]. High volume fraction up to 40 % was achieved through this process. The MgO particles were mainly settled at grain boundaries and good interfacial bonding of MgO was observed with Mg. The composites showed increase in thermal stability, compressive strength, failure strain and room temperature as well as high-temperature hardness.

Zhang et al. prepared ZK60/0.5 vol. % MgO composites with two reinforcement arrays through conventional powder metallurgy route [65]. They found that the homogeneous nano dispersoids composite shows higher strength but lower plasticity than the composite with fibre-like MgO bands. Both composites showed higher strength and marginally lower ductility than unreinforced alloy.

Yao et al. in 2021 synthesised Mg/MgO composites coating on AZ31B alloy by High Velocity Suspension Flame Spraying (HVSFS) [18]. An increase in hardness and elastic modulus was observed due to MgO addition while bonding strength showed slight improvement.

Zhang et al. in 2021 fabricated Mg-3Zn-0.2Ca composite with 0.6 wt. % MgO through stir casting followed by hot extrusion and then finally subjected to Equal Channel Angular Pressing (ECAP) [66]. The resultant microstructure consists of very refined grains and evenly distributed MgO particles. The ultimate compressive strength increased to 405 MPa and compressive failure strain increased to 34 % with eight passes of ECAP.

Rahmani et al. in the same year fabricated Mg/MgO and Mg/Al_2O_3 composites by Equal channel angular extrusion (ECAE) method [19]. They used large size MgO particles (105 μm) with vol. fraction of 0, 10, 20 & 30 %. Hardness and strength were directly proportional to vol. fraction of MgO.

Wang et al. in the year 2023 developed Honeycomb-like AZ31 matrix composites with 1, 2, and 4 wt.% submicron-MgO using low-energy milling and extrusion [67]. The newly developed composites showed improvement in both strength and ductility simultaneously. The tensile strength of composites containing 1 wt. % MgO improved to 344.1 MPa, and at the same time, its elongation increased to 21.5 %.

Tang et al. reported this year the effect of MgO nanoparticles on the mechanical properties and corrosion behaviour of Mg–Zn–Ca alloy prepared by stir casting followed by heat treatment and hot extrusion [68]. They concluded that MgO nano particles promoted dynamic recrystallization during extrusion and yield strength improved mainly due to grain boundary strengthening and orowan strengthening mechanisms.

2.8 Friction and wear

2.8.1 Friction

The resistance encountered by one body in moving over another is known as friction. Asperities are present on every surface, and the interaction of asperities of two mating surfaces gives rise to high friction. Frictional force is the sum of resistance offered by all the individual asperity contact sites. Coulomb proposed the basic laws of friction. **The first law** states that, friction is directly proportional to the normal load. **The second law** says that the friction force is independent of apparent area of contact between the contacting surfaces. **The third law** says that, the kinetic friction force is independent of sliding velocity, once the motion starts. Some theories were also proposed with time. One of the famous theory was proposed by Bowden and Tabor known as **Adhesion Theory** of friction. It states that when two precisely clean surfaces are pressed against each other at room temperature, strong junctions are formed due to cold welding and adhesion without any interdiffusion or recrystallization of metal atoms taking place at these junctions. A force is needed to break these junctions to move one body over other. Another theory of friction was proposed by Coulomb known as **Asperity Interlocking Theory**, says that when two surfaces are placed in contact with each other, both surfaces touch each other at discrete points known as asperities. Applied load is borne by these asperities and they deform plastically on the application of load. When sliding occurs soft asperities deform under the application of load and leads to frictional resistance. **Molecular attraction theory** proposed by Hardy states that friction is because of molecular attraction. Origin of this theory is the partial irreversibility of bonding force among the atoms. Such molecular attraction works for short distances and thus discriminates between real area of contact and apparent area of contact. It is also known as extension of adhesion theory. Bowden and Leben proposed the **Stick – slip theory**, also considered as the alternative to theory of adhesion, which assumes that one surface rests over the other at junctions. Once the surface starts to slide over another, a rise of temperatures occurs at these junctions, which results in local welding at the junctions. This leads to resistance to motion, i.e. friction. Sliding occurs on account of applied force by tearing apart of these welds.

2.8.2 Wear and their types

The gradual loss of material from solid bodies which are in relative motion is known as wear. Mechanical properties of rubbing surfaces, chemical interaction and testing conditions affect the wear process. The wear of materials has been classified on the basis of the operative mechanisms and major types of wear are (i) abrasive (ii) adhesive, (iii) erosive, (iv) fatigue, and (v) corrosive wear.

Abrasive wear occurs when loss of material is caused by protuberance of hard material that are forced against soft material. It depends upon type of contact and contact environment. If there are two rubbing parts involved in friction process, then it is called two body abrasion. In this mode of abrasive wear, asperities of hard body cause the material loss from the soft body while sliding. If the material loss occurs due to entrapment of hard particles between two mating surfaces, it is called three body abrasion.

Adhesive wear occurs due to formations of micro junctions caused by welding between opposing asperities of two rubbing surfaces. In other words, adhesive wear occurs in the form of material transfer when two bodies either slide or pressed against each other. Applied load on the body is borne by asperities. Due to less apparent area, load on asperities is high enough to deform them and adhere to each other forming the micro joints. The motion of bodies' results in rupturing of the joints and rupture takes place from non-deformed region. Material transfer takes place in this manner from counter - body.

When the material loss takes place from a solid surface by the impingement of fluid or solid particles, such type of damage is known as **erosive wear**. In this type of wear, repeated deformation and fracture occurs due to mechanical action of particles on solid surface. Particles with some velocity impinge on the surface and cause pits, subsurface deformation etc.

The repeated loading and unloading cycles to which the materials are exposed may induce the formation of sub- surface or surface cracks, which eventually after a critical number of cycles, will result in the breakup of the surface with the formation of large fragments. This is called **fatigue wear**. Prior to this critical point (which may be hundreds, thousands, or even millions of cycles), negligible wear takes place, which is marked contrast to the wear caused by an adhesive or abrasive mechanism.

Fretting Wear occurs when low amplitude oscillatory motion in the tangential direction (ranging from a few tens of nanometers to few tens of microns) takes place between contacting surfaces, which are nominally at rest. This is common occurrence, since most machinery is subjected to vibrations, both in transit and in operation. It is common in shrink fits, bolted parts, splines, bearing housing on rotating shafts or axles, etc.

Wear due to corrosion or oxidation during sliding in the presence of corroding medium is known as **corrosion wear**. It can occur during dry sliding or in the presence of some gas. Harsh environmental conditions like high temperature, sea water and high acidic or basic medium can cause this type of wear.

2.8.3 Dry sliding wear of Mg composites

Literature suggests that dry sliding wear behaviour of magnesium composites have been studied by researchers by incorporating hard ceramics like SiC, BN, ZnO, etc. Notable contributions made by different people are discussed here.

Saravanan and Surappa in 2000 studied the dry sliding wear of Mg/SiC composites at 5, 10 and 50 N load and sliding velocity of 0.5 m/s for fixed distance of 1.5 km [36]. They found that the wear rate of the composite were generally less by two orders of magnitude compared to unreinforced Mg at all loads.

A detailed study was done by Lim et al. in 2003 [69]. They studied the dry sliding wear of Mg-Al/SiC MMCs fabricated by powder metallurgy. The tests were carried out at 10 N and 30 N load over a range of sliding speeds from 0.2 - 5 m/s. They reported that at lower load, wear occurred due to oxidation and improvement in wear resistance was observed due to load bearing capacity and stable oxide film. But as load was increased to 30 N, wear due to delamination and abrasion occurred deteriorating the wear resistance. As sliding velocity was increased, adhesion wear became dominant and at even higher velocity thermal softening and melting occurred due to high frictional heat.

Kumar et al. in 2004 studied the role of tribo - chemistry on fretting wear of Mg/SiC MMCs under low load of 2-10 N [70]. They observed that reduction in friction and wear of composites was observed only at higher reinforcement content (26.3 wt. %). The wear rate initially increased (4–8 N) and then subsequently decreased at the highest load of 10 N due to formation of soft, viscous tribo-products. At lower SiC content (9.8 wt. %), the properties of base Mg matrix dominated. In pure Mg, MgO reacts and forms hydrous magnesium oxide, $Mg(OH)_2$ whereas hydrous magnesium silicate forms in composites which reduces the COF values.

Selvam et al. in 2014 carried out dry sliding wear study of Mg/ZnO nanocomposites under three different loads (5 N, 7.5 N and 10 N) and at three sliding velocities (0.6 m/s, 0.9 m/s and 1.2 m/s) [71]. The wear rate was higher under higher loads at all three sliding velocities. The coefficient of friction (COF) at 7.5 N load was greater than those for 5 N and 10 N load regardless of sliding velocities. However, COF decreased as the sliding distance increased. Wear rate significantly enhanced due to addition of ZnO nanoparticles in Mg matrix.

Nguyen et al. in 2014 studied the wear characteristics of AZ31B and AZ31B/Al_2O_3 nanocomposites under sliding speeds of 1, 3, 5, 7, and 10 m/s for 10 N load and 1, 3, and 5 m/s for 30 N normal load [72]. They observed that the wear rates of the composites gradually reduced over the sliding speed range for both the normal loads.

The COF of both the alloys and composites are in the range of 0.25–0.45. The main wear mechanisms observed were abrasion, oxidation, adhesion, and thermal softening and melting for all specimens, in addition to delamination for the composite materials.

Kaviti et al. in 2018 studied dry sliding wear behaviour of Mg/BN (0, 0.5, 1.5, 2.5 wt. %) nanocomposites at 5 N, 7 N and 10 N and sliding speeds of 0.6, 0.9 and 1.2 m/s [73]. With increase in load and sliding speed, wear rate increased. For Mg-0.5 wt. % BN nanocomposite the change in rate of wear and loss of volume were very less compared with other compositions of Mg/BN nanocomposites. Higher friction coefficient value occurred at a normal load of 7 N compared with other working loads.

Al-Maamari et al. in 2019 studied the dry sliding wear of Mg/Graphite (3, 5, 7 and 10 wt. %) at 10, 20 and 30 N normal load and sliding speeds of 0.5, 0.8 and 1 m/s [74]. The Mg–Gr composite possessed lower wear rate and lower friction coefficient as compared to pure Mg and these were minimum at 5 wt. % graphite composites. However, above 5 % graphite addition, both the wear loss and friction coefficient of Mg-Gr composites increased due to the formation of porosity caused by graphite particles.

Recently Shen et al. in 2022 studied dry sliding wear behaviour of AZ31/SiC (1 vol. %) nanocomposite with the different sliding velocities of 0.1, 0.3 and 0.5 m/s at normal loads of 10, 20, and 30 N [75]. The wear resistance of composites were more prominent with respect to initial alloy under all test condition. However, wear rate decreased constantly with increasing sliding velocity. Abrasive wear and oxidative wear were mainly dominant at lower speed. At higher sliding speeds, delamination was more dominant wear mechanism.

Many works have been successfully done to improve the wear characteristics of Mg or its alloys by reinforcing with ceramic particulates. Tribological studies of Mg/MgO composites have not been done till now. However, Salman et al. studied the effect of MgO and TiO_2 on wear behaviour in aluminium matrix [76]. The tests were done for 2, 4, 6, 8 and 10 N loads. They found wear rate increased with increase in applied load for all specimens. The Al/nano MgO showed high wear resistance than Al/nano TiO_2 at all loads.

2.9 Research gaps

- Mg/MgO composites have great potential to be a good engineering material and they have been tried by different processing techniques but their use is limited because of high production cost.
- There are very limited works on the use of powder metallurgy technique for the fabrication of Mg/MgO composites.
- Detailed investigations have not been carried out on the effect of MgO reinforcements on different physical and mechanical properties of Mg MMCs fabricated by powder metallurgy.
- Although hard MgO particles have been used with aluminium to improve the wear properties of Al MMCs but there are no studies with magnesium. Investigations have not been done on the wear behavior of MgO reinforced metal matrix composites under different loading and sliding velocity conditions.

2.10 Formulation of problem

A critical review of literature presented above suggest that MgO has great potential to enhance the mechanical properties of pure Mg. Wang et al. reported that MgO has large possibility to act as a potent nucleant for heterogeneous nucleation of α - Mg grains [77]. Many researchers synthesised MgO reinforced pure Mg or its alloys by different processing techniques. Each technique has its advantages and disadvantages. The common factor associated with all the processes is the high cost of fabrication. This restricts the use of composites in different applications despite having required properties for the same. So, it becomes imperative to explore a low cost fabrication technique which does not compromise the end properties. It is also observed that Mg/MgO MMCs have not been fabricated by powder metallurgy route especially microwave sintering which saves more than 90 % power than conventional sintering thereby reducing overall cost. Literature also suggests that only few works have been done to understand compressive behaviour of Mg/MgO MMCs. Further the hard MgO particles are anticipated to improve the wear behaviour when reinforced with pure Mg or its alloys. There are several works on wear behaviour of Al or its alloys reinforced with MgO particles [78-80]. But there are almost no studies on evaluation of tribological potential of MgO particulate in pure Mg or its alloy.

In the view of above, the present study is being conducted to explore the low-cost sintering method for fabrication of Mg/MgO MMCs through powder metallurgy. Therefore, Mg/MgO composites have been fabricated by two sintering methods. The first is the rapid (hybrid) microwave sintering and the second is a novel sintering approach which uses aluminium foil to protect samples during sintering. The study also unravels the potential of MgO particulates to impart mechanical and tribological enhancement to pure Mg. So, MgO reinforced Mg MMCs having varying vol. fraction of MgO may be synthesised by the above processes. Pure Mg without any reinforcement was also synthesised by same processes for comparison purpose. The synthesised pure Mg and Mg/MgO composites were characterised for physical and mechanical properties. The distribution of phases in composites may also be studied by diffraction and microscopy techniques to get a better understanding of properties. The present study also envisages to explore the tribological performance of MMCs in dry sliding condition. The effect of reinforcement content on coefficient of friction and wear rate may be studied with changes in load and sliding speed. The study also intends to establish the prevailing mechanism of wear under the conditions used in the investigation.

2.11 Objectives of present work

In the light of the above, the present study has been carried out with an aim to fulfil the following objectives.

1. To fabricate Mg/MgO MMCs through powder metallurgy route assisted by rapid microwave sintering method.
2. To study different physical and mechanical properties of microwave sintered Mg/MgO composites.
3. To fabricate Mg/MgO MMCs through powder metallurgy utilising a novel sintering method.
4. To study the distribution of phases and its effect on properties through diffraction and microscopy techniques.
5. To study the effect of varying weight fraction of MgO particulates on physical and mechanical properties like density, hardness, and compressive behaviour of composites.
6. To study the dry sliding wear behaviour of composites fabricated by novel sintering method under varying load of 5, 10, and 15 N and at each sliding velocity of 0.6, 0.9 and 1.2 m/s.