SINTERING OF MAGNESIUM METAL MATRIX COMPOSITE BY MICROWAVE ENERGY

A DISSERTATION

submitted in partial fulfillment of the requirements for the award of the degree of

MASTER OF TECHNOLOGY

in

MECHANICAL ENGINEERING

(With Specialization in Production and Industrial System Engineering)

By

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June, 2019

CANDIDATE'S DECLARATION

I hereby declare that the work carried out in this dissertation report entitled "**SINTERING OF MAGNESIUM METAL MATRIX COMPOSITE BY MICROWAVE ENERGY**" is presented on-behalf of partial fulfilment of the requirement for the award of the degree of **Master of Technology** with specialization in Production and Industrial Systems Engineering submitted to the department of Mechanical and Industrial Engineering, Indian Institute of Technology Roorkee, India, under the supervision of **Prof.** A**purbba Kumar Sharma**, Professor, Department of Mechanical and Industrial Engineering, IIT Roorkee, India.

I have not submitted the matter embodied in this report for the award of any other degree or diploma.

Date: June, 2019 **(Sakshi Jaiswal)**

Place: Roorkee Enrol. No.: 16540009

CERTIFICATION

This is to certify that the above statement made by the candidate is correct to the best of my knowledge and belief.

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ABSTRACT

It is difficult to process advanced materials such as composites with conventional processes to attain desired properties. Thus, non-conventional material processing techniques have gained popularity to develop composite materials. In the today's scenario, demand of advanced materials to achieve high specific strength in aerospace industry and automobile sector is increasing, which motivates researchers to develop tailored materials and advanced processing techniques. In recent time, magnesium matrix composite are being researched for enhanced properties due to its lower weight. In the present work, an overview on different processing techniques used for development of magnesium matrix composite processed was studied. The merits and demerits of the different processes have been compared to understand ease of composite processing. A new composite process technique using microwave energy at 2.45 GHz frequency was explored. Microwave sintering was carried out for magnesium based metal matrix composite having reinforcement in amount (5%, 10% & 15% by weight) of SiC powder. Characterization for microstructures was performed using SEM/FESEM, optical microscope, EDS analysis. From optical micrograph, SEM images and EDS analysis, it was found that on increasing the amount of SiC particles in AZ91 alloy more Mg_2Si and SiC phases were appeared in the sintered samples.

Physical property i.e. density was measured using Archimedes principle and micro hardness test was performed to measure microwave sintered samples. Experimental density of the sintered samples were in the range of 85 to 90% of the theoretical density and micro hardness was more in sintered sample which had 5% SiC particle by weight.

Moreover, all the results obtained from all analysis have been discussed. Subsequently, attention was focussed on the challenges in the proposed method and yield of the process. Future scope for the microwave sintering of magnesium based metal matrix composite is also outlined.

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ABBREVIATIONS

P	Power dissipation (W)
D	Depth of penetration of microwaves (m)
E	Electric field through the surface (V m^{-1})
V	Volume (m^3)
f	Frequency of microwaves (GHz)
Z	Distance into the specimen (m)
C_P	Specific heat capacity (J $kg^{-1}K^{-1}$)
k	thermal conductivity of the material (W $m^{-2}K^{-1}$)
Q	heat source (W)
h	coefficient of convective heat transfer (W $m^{-2}K^{-1}$)
ω	angular frequency (cycle / s^{-1})
ε	electric constant in medium (F m^{-1})
ε'	electrical permittivity in medium (F m^{-1})
ε''	dielectric loss factor (F m^{-1})
j	electrical polarizability and imaginary part
δ	skin depth (mm) and dielectric loss tangent
ρ	the density (kg m^{-3}) and resistivity (μΩ-cm)
ε_0	electrical permittivity of vacuum (8.85×10^{-12} Fm^{-1})
μ_r	relative permeability (H m^{-1})
ε_r	relative permittivity (F m^{-1})
σ	the electrical conductivity (S m^{-1})
k_0	wave number in free space
c_0	speed of light in vacuum (m s^{-1})
ε^*	complex electric permittivity (F m^{-1})
γ	collision frequency of electrons (s^{-1})

σ_m	static electrical conductivity of metal (S m^{-1})
σ_0	electrical conductivity of metal at temperature T_0 (S m^{-1})
β	temperature coefficient of metal
ε_{rad}	emissivity of radiative surfaces
σ_i	ionic conductivity of metallic ions in the metal lattice (S m^{-1})
α	attenuation constant
μ_0	absolute permeability (H m^{-1})
μ	magnetic permeability (H m^{-1})
W_1	weight of AZ91 Mg alloy (gm)
W_2	weight of SiC (gm)
ρ_1	density of AZ91 Mg Alloy (gm / cm^{-1})
ρ_2	density of SiC (gm / cm^{-1})

Chapter 1

INTRODUCTION

1.1 COMPOSITE

In the development of advanced material, the composites are most famous among researchers because composite materials are made up of two or more dissimilar materials, which may differ in forms, remain physically distinct, i.e. insoluble in each other and chemically do not react between them. The properties of the new material are different from that of its constituents. Composites have both advantages and limitations, which are discussed as below:

1.1.1 Benefits

- ✓ Composites own an aggregate of outstanding mechanical, chemical, structural, electrical and optical properties.
- ✓ They are light-weighted and better specific strength and specific modulus while compared to traditional materials.
- ✓ Composites can be moulded to any desired form with any favoured specification.
- ✓ They own good anti-chemical and anti-corrosion residences.
- ✓ Assembling and de-assembling of components is simple and quick.
- ✓ Efficient usage of material can be done. The fibres should be oriented in this sort of way to provide greatest strength and stiffness within the preferred direction.
- ✓ Problems like seepage and weathering are nearly minimal in composites.
- ✓ Composites give aesthetic designed look.

1.1.2 Constraints

- ➢ Composites are having low fire and flash points.
- ➢ Composites may additionally expand unwanted biological effects observed in polymers.
- ➢ Polymeric composites are unacceptable for high-temperature applications.
- ➢ Cost of composites continues to be more than many conventional substances.
- ➢ On prolonged left in vicinity of sunlight, the colours of composites typically fade out.

1.1.3 Classification of composites

Composites can be classified as fallows-

1. Agglomerated composites: For e.g. grinding wheel where abrasives are agglomerated in the wheel with the help of the binder.

2. Laminated composites: For e.g. plywood.
3. Reinforced composites.

Reinforced composites consist of a matrix and reinforcement. They can be further classified based on kind of matrix as well as reinforcement. Based on matrix materials used they are classified as:

1. Polymer matrix composites (PMCs)
2. Metal matrix composites (MMCs)
3. Ceramic matrix composites (CMCs)

1.2 METHODS FOR PRODUCTION OF THE MMCs

1.2.1 Stir Casting

It is referred as the vortex procedure. Reinforcement of particles is done for support of casting in molten material. Homogenous distribution of reinforcement is finished through a rotor rotating within the molten metal which develop a vortex and by means of inserting of a gas which carry the reinforcement into molten metal. The finely allotted produced slurry is original with the aid of conventional casting systems, viz. Sand casting, permanent mildew casting, pressure die-casting or squeeze casting. The typical stir casting process is shown in fig 1.1.

Factors that effects the stir casting process are as followings:

a. Gas entrapment
b. Slag inside the molten metal causes porosity and defects in the casted sample
c. Unfavourable chemical reactions at the interface of matrix/reinforcement
d. Due to low wettability of reinforcements with molten material matrix causes debris agglomeration (primarily in nano-reinforcements that might deliver about the formation of nano particle clusters that leads to non-uniform distribution of reinforcements)

Bypassing these problems would be the reason of deterioration of material made by this process. To make it successfully in laboratory and industrial levels, cautious, standardization of system parameters including temperature of molten metal, soften stirring time, stirring pace, melt retention temperature, option of matrix-reinforcements and many others have to be rightly made up our minds on.

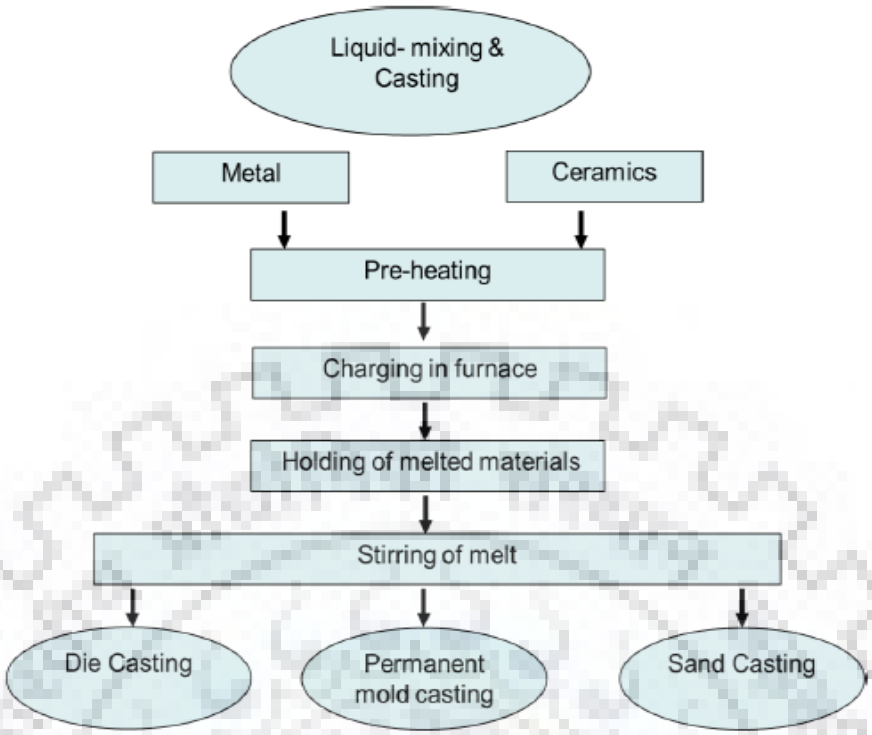


Figure 1.1. Schematic diagram showing stir casting process [1]

1.2.2 Centrifugal Casting

This process is a tremendously cheaper procedure wherein optimal reinforcement placements are performed by way of inducing a centrifugal force straight away all through casting. This ensures an intentional variant in volumetric fraction within the matrix material. A natural example to be used of centrifugal casting is in brake rotors, in which the rotor face is anticipated to be of immoderate put on resistance while in comparison with the hub. Problems due to machining of casted product by general casting are faced due to the excessive hardness, is removed by centrifugal casting method.

1.2.3 Squeeze Casting/Infiltration Process

This procedure includes the invasion of a liquid compound into a ceramic fiber/molecule preform pursued by cementing. The presentation of liquid metal into a preform would be finished both by means of pressure less infiltration and via invasion underneath pressure. In pressure less penetration, ceramic fiber packs are first set within the die. The liquid metal is then poured on to it and permitted to harden. The fixed composites are then sizzling squeezed to achieve 100 % thickness. The underlying invasion happens with no use of outside pressure and the wettability of strands guarantees effective penetration. The weight invasion procedure can be utilized in two distinct ways, in particular through gas penetration and weight

penetration. In gas penetration, vacuum or latent gas air is used to deliver invasion. Focal points of this technique incorporate increment in the wettability due to the expanded surface action of fortification in vacuum condition, end of gas entanglement or porosity and accomplishing close net formed segments. Its fundamental inconveniences are isolation of stages and response between framework/fiber because of the moderate idea of the process. The crush penetration process includes the invasion of liquid metal into an artistic preform utilizing water driven pressure. By this strategy, the downsides of stage isolation and grid/interface response experienced in gas invasion can be killed because of the use of pressure drive.

1.2.4 Spark Plasma Sintering (SPS)

SPS is a comparably new sintering procedure and it permits compaction of powdered metals and ceramics at low temperature in short holding time. SPS is fundamentally the same as traditional hot squeezing, precursors are loaded in die where an uniaxial pressure is applied during sintering. However, a pulsed direct current is passed through the electrically conducting pressure die. Sometimes, pulsed direct current passes through the sample in appropriate cases. This infers the die, likewise acts, as a warming source, and heating takes place from both sides(inside and outside) on the sample. The schematic illustration of this procedure is appeared in Fig 1.2.

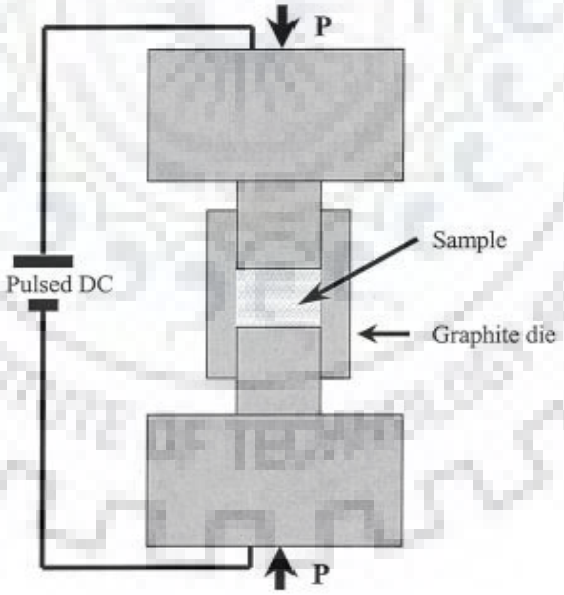


Figure 1.2. Schematic drawing of SPS apparatus [2]

The remarkable highlights of the procedure are the possibilities of utilizing quick warming rates and short holding time to acquire dense samples. Three factors that causes to the quick densification procedure can be noted below:

(i) Application of a mechanical pressure

(ii) Quick heating rates,

(iii)Use of pulsed direct current inferring that the sample are likewise presented to an electrical discipline.

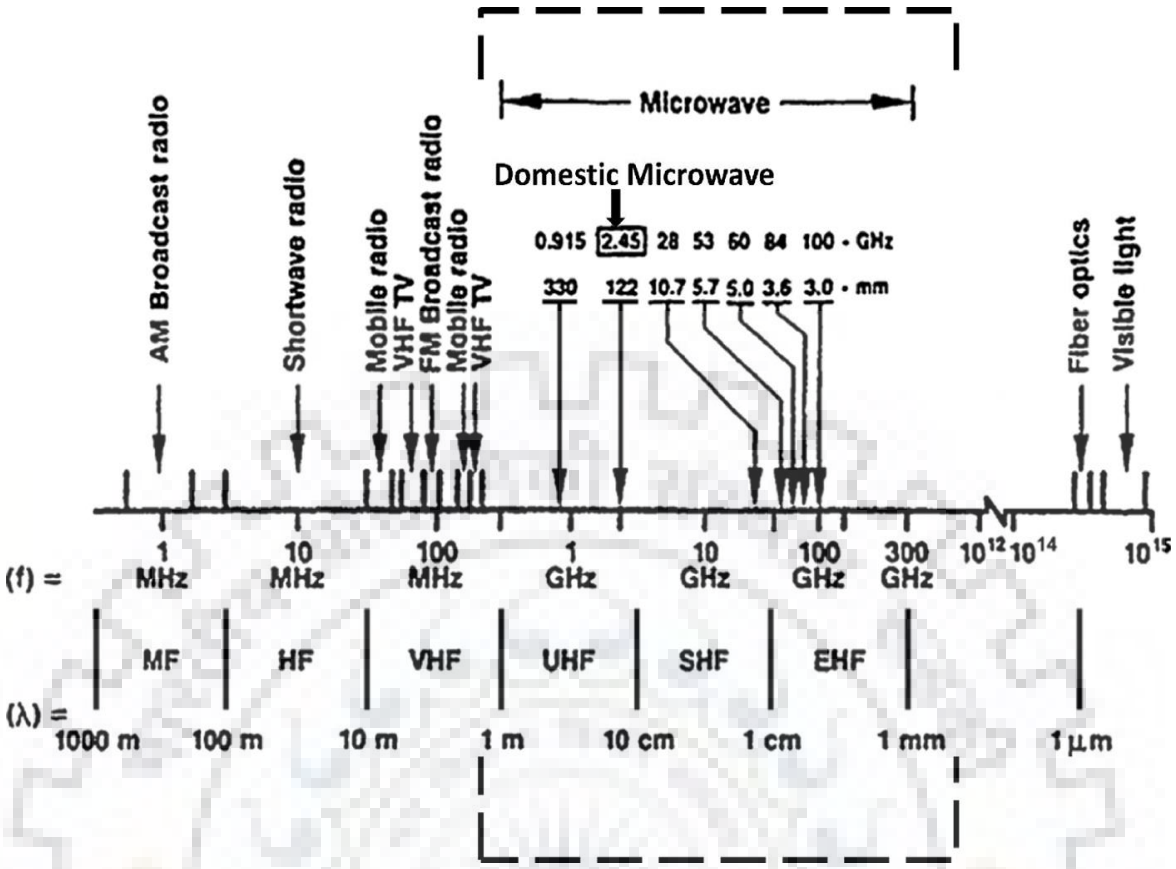
It is probably the most section stated that use of mechanical stress is regularly, worthwhile in getting rid of pores from compact samples and embellishing diffusion. The heat transfer to the compact sample from the die is tremendously efficient in this approach, due to the fact that the die itself (heated from the pulsed direct current) acts as a heating aspect. Accordingly, pulses and then plasma between the powder particles (pattern) develop spark discharges. Hence, this is the reason of calling this approach as SPS.

1.2.5 Microwave Sintering

M Gupta et al. (2007 to 2009) [9] used microwave radiation for volumetric heating of samples, which is kept inside the microwave oven. This process changes the electromagnetic energy in to heat energy. In this process, microwave susceptors are used in microwave to aid the decrease of thermal gradient during sintering. The compacted samples or billets of metal/ reinforced composites are kept in the inside the oven in crucible. There are two crucible is used. In inner crucible, compacted sample is placed and the SiC powder is kept between the gap of inner and outer crucibles, because SiC has characteristic to absorb the microwave radiation promptly and heats up rapidly. This offers incredible warmth that can thus remotely warms the compacted billets. What's more, the compacted billets themselves assimilate microwave and get warmed from inside of. Thereby, uniform distribution of heat is experienced along the whole part of a specimen, thus lowering any core-to-periphery thermal variant. On account that of this purpose, excessive sintering temperatures (~620–650 °C) can be created inside of a brief time frame (12–14 min), which can be close to near the softening purposes of Al and Mg, via the goodness of which improved wettability and reduced porosity may also be complete.

1.3 MICROWAVE MATERIALS PROCESSING

The microwaves are basically electromagnetic radiation and their radiation frequency range lies someplace within the range of 1 and 300 GHz and these microwave frequencies with quite a lot of wavelengths are utilized for a wide assortment of makes use of that are appeared in Fig. 1.3



.Figure 1.3. Electromagnetic radiations spectrum for frequency and wavelength [3]

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

Various methods were used to prepare MMCs with the objective to get the incremental improvements in their mechanical properties and their comparison with other methods of manufacturing the same. Effect of reinforced materials with their varying percentage was studied. The change in reinforcement not just affected properties but it also changed the

microstructure and grain growth of the composites. For the manufacturing of MMCs, the conventional processes such as die-casting, stir casting, friction stir process and common methods to prepare MMC were studied. After summing up, it was concluded that all these properties could be enhanced by fabricating the MMCs through MHH. Metal casting by utilising microwave radiation energy is recent ongoing projects for MWs. The procedure was created and metal cast were accounted for the procedure and cast portrayal are yet to be accounted for.

As of late, the use of microwaves in different applications has expanded numerous folds. The different handling areas where this innovation has been connected effectively is appeared in Fig. 1.4, which incorporates correspondence frameworks, sustenance handling, wood drying, improved chemical responses, vulcanization of elastic, preparing of earthenware production and metallic materials, making of different kind of steels, materials joining and its welding, squander nursing, and recuperation of substitute wellsprings of vitality.

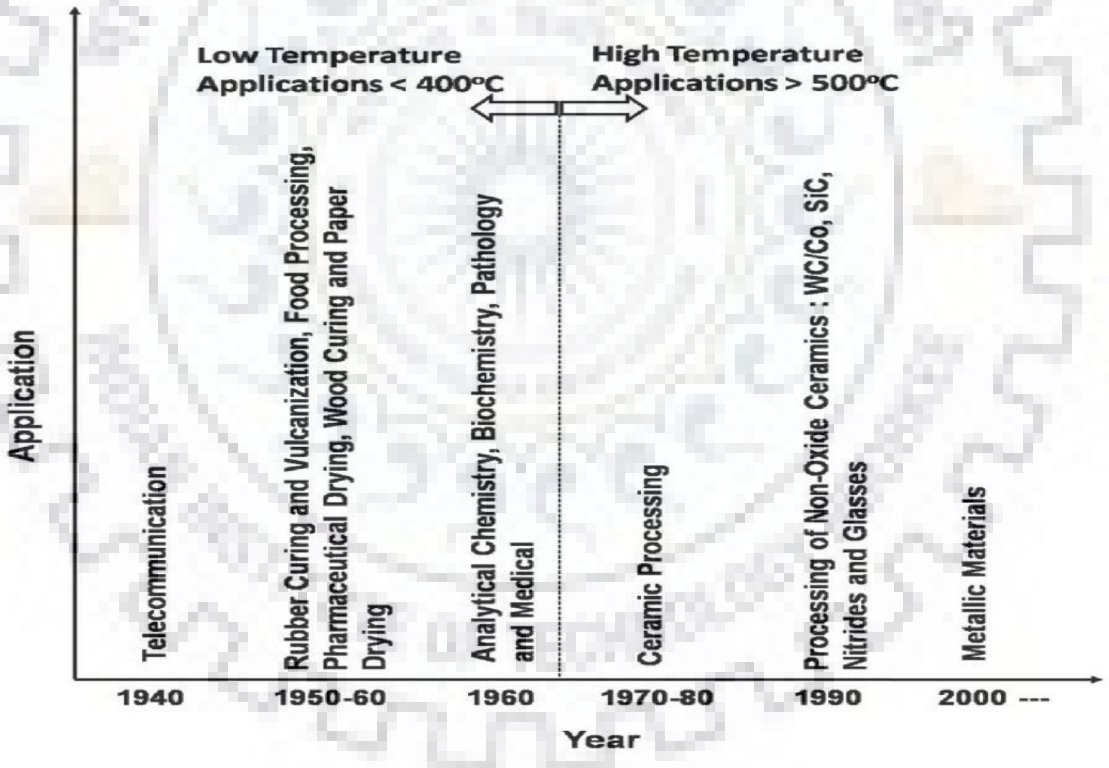


Figure 1.4 Recorded improvements appearing of microwaves in different fields [2]

Presently, it is notable that metal as powder can retain microwaves and can be prepared effectively. Further to investigate the benefits of microwaves, microwave hybrid heating was created which drove numerous scientists to process different sorts of materials for an assortment of uses in the field of material science. The different advancements in the field of

preparing of metals are appeared in Fig. 1.5, which demonstrates year astute ordered advancements utilizing microwaves. Over the most recent 65 years, microwave vitality has been used in an assortment of uses that can be categorized as low temperature (<500 ^{o}C) applications in amalgamation and drying (1950–1970), moderate temperature (between 500–1000 ^{o}C) uses as in sintering applications (1970–1999), and high temperature (>1000 ^{o}C) utilizations in the cutting edge material preparing (1999–onwards). It is obvious from the advancements that, for the most part, metal tests are assuming a significant job in the handling of metal-based materials. The hypothetical and exploratory research works that were accounted for in the zone of microwave preparing of metal-based materials can be separated into four noteworthy gatherings i.e., powder metal, mass powder metal framework, mass metal, and sheet metal based on accessible writing. The hypothetical analyses and test examinations were announced for metal powders as unadulterated metal powder compacts, metal amalgam compacts and metal framework composites. The mass powder metal frameworks were accounted for applications, for example, brazing, joining, and covering/cladding. The mass metal and sheet metal preparing were accounted for by a couple of scientists as melting, casting, drilling (on metal/non-metal utilizing a metallic instrument), and warmth treatment. The hypothetical models and exploratory methodologies were likewise announced for examining compelling utilization of metallic materials amid microwave handling of non-metallic materials in other applications such as nourishment preparing, concoction union, and so on.

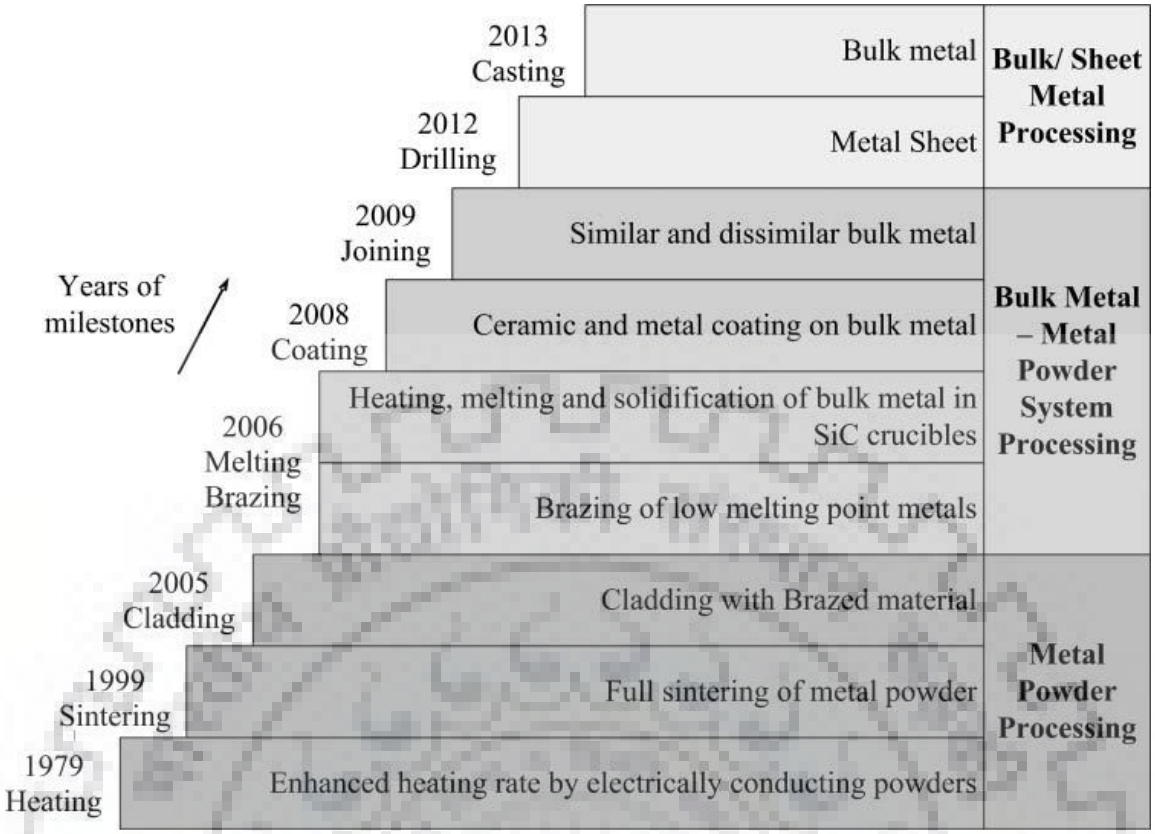


Figure1.5 Sequential improvements in the field of microwave processing of metals [3]

1.4 MICROWAVE MATERIALS PROCESSING

Microwave heating of materials is done effectively through microwave radiation, mainly depends upon the physical properties of that material. Because the mechanical properties of materials helps in interaction of microwave radiation in that materials. Generally, materials are classified in three groups, which is shown in Fig. 1.6. Microwave interactions does not take place in transparent materials and crosses them without any heat transfer. Microwave radiations reflect back on conductors that lead to plasma formation and by this surfacial heating of conductors take place. Absorbers readily absorb the microwave, which retains and changes over these radiations into heat.

Material properties for microwave absorption are the complex. The relative permittivity of space, medium and loss tangent shown by Eqs. (1) and (2) [1, 3].

$$\boldsymbol{\varepsilon = \varepsilon_0 (\varepsilon' - j\varepsilon'') = \varepsilon_0 \varepsilon' (1 - j \tan \delta)} \quad \textbf{(1)}$$

$$\boldsymbol{\tan \delta = \frac{\varepsilon''}{\varepsilon'}} \quad \textbf{(2)}$$

Where

ε=electric constant in medium

ε_0=electrical permittivity in space

ε'=electrical permittivity in medium

ε''=dielectric loss factor

j=electrical polarizability

δ=dielectric loss tangent

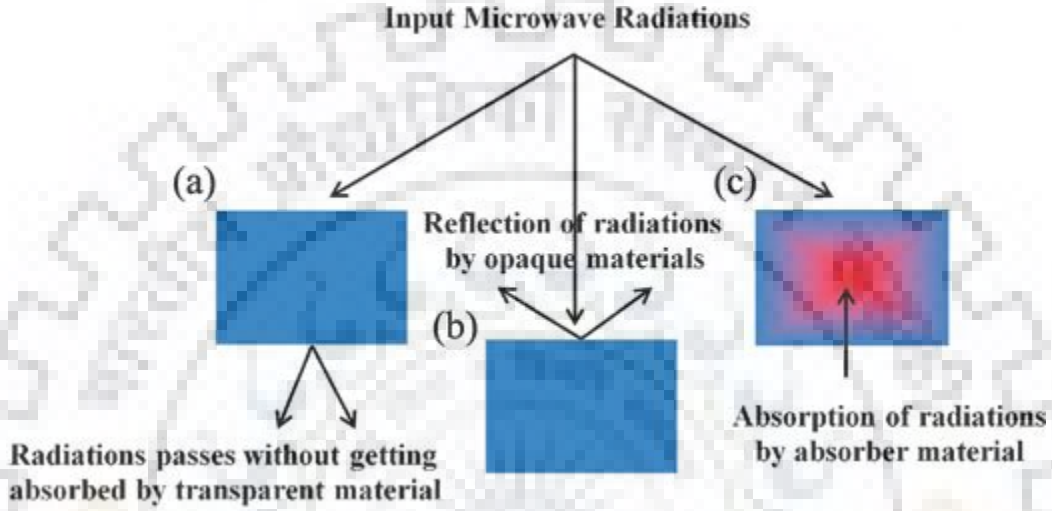


Figure 1.6. Microwaves–materials interactions [3]

The ability of a material to transfer microwave energy into heat is defined by dielectric loss factor, and for measuring, the polarizability of material is dielectric constant. The increase in temperature because of absorption of microwaves is ruled by means of Eq. 3.

$$\frac{\Delta T}{\Delta t} = \frac{2\pi f \varepsilon_0 \varepsilon'' |E|^2}{\rho C_P} \quad \textbf{(3)}$$

The dielectric associations of materials are additionally portrayed by two parameters: power dissipation (P) and depth of penetration (D) of microwaves. The consistency of heating profile relies upon these variables.

The power dissipation is communicated by Eq. (4).

$$P = \frac{1}{2}\omega . \varepsilon_0 . \varepsilon'' . E^2 . V . e^{-2\alpha z} \quad \textbf{(4)}$$

Where, E = electric field through the surface,

V = volume,

ω = 2πf frequency,

z = distance into the specimen and

α = attenuation constant.

The consistency of warming inside the material relies on the depth of penetration on which the episode power is decreased to half of introductory esteem. Depth of penetration is communicated by Eq. (5)

$$\boldsymbol{D = \frac{3\pi_0}{8.686\pi \tan\delta\left(\frac{\varepsilon'}{\varepsilon_0}\right)^{\frac{1}{2}}}} \quad \textbf{(5)}$$

The correlation of the penetration depth in terms of frequency is expressed by Eq. (6)

$$\boldsymbol{D = \frac{C}{2\pi f\sqrt{2\varepsilon'}\left(\sqrt{1+\tan^2\delta}-1\right)^{\frac{1}{2}}}} \quad \textbf{(6)}$$

The higher estimations of t and electrical permittivity decrease the depth of penetration at a specific wavelength of microwave radiation. Surficial warming is due to high frequency and high estimations of dielectric properties so for the volumetric warming moderate dielectric property is required.

At room microwaves radiation of 2.45GHz reflect for mass metallic compounds because of smaller estimations of skin profundity, which results in impression of radiations. The skin depth of materials in connection to microwave handling is characterized as the profundity into the materials from the surface at which the estimation of episode microwave control drops to 1=e (36.8%) times the surface esteem. A significant parameter gives a comprehension of the upper thickness breaking point of materials to be handled through microwave radiation in a proficient way. In any case, it is conceivable to expand the skin depth (δ) of specific material at a specific recurrence by changing the temperature-subordinate parameters, i.e., resistivity and magnetic permeability as shown in Eq. (7).

$$\boldsymbol{\delta = \sqrt{\frac{\rho}{\pi f \mu_r \mu_0}}} \quad \textbf{(7)}$$

Where

δ=skin depth (mm)

ρ=resistivity (μΩ-cm)

f=frequency of microwaves (GHz)

μ=magnetic permeability (H=m)

μ_0=absolute permeability (H=m)

μ_r=relative permeability

Microwave radiations for better coupling with various metallic materials with the idea of hybrid heating was proposed by analysts which is talked about in the next segment.

1.5 HYBRID HEATING BY MICROWAVE

The effort, which is done for microwave samples (materials) preparations, was principally completed by utilizing local heating by microwave with a recurrence of 2.45 GHz. The immediate warming of samples by microwaves radiations may experience central issue like thermal instabilities that may create thermal runway into the prepared materials. The traditional method of warming prompts poor microstructures of surfaces, while microwave-warming mode causes poor microstructures of centres due to distinction in temperature inclinations in various territories. These slopes can prompt extreme temperature no consistencies and may cause cracks inside the prepared materials. To conquer the issue of thermal instabilities, research specialists have created hybrid heating system because it involve heat exchange marvel which includes both ordinary and heating through microwave radiation. Materials, which are having higher dielectric misfortunes at room temperatures are utilized as an infrared warming source. These sources are names as subsector, which retain the energy of microwave positively, accomplish temperature in high amount, and then use to heat the metallic material, which is in powder form by means of traditional ways. This case is the best example of mixed heat transfer, utilize for material heating which are having less dielectric misfortunes like composite materials. Fig. 1.7 is showing the two way heating of powder samples, which lead to reduce the time of heating and give uniform heating.

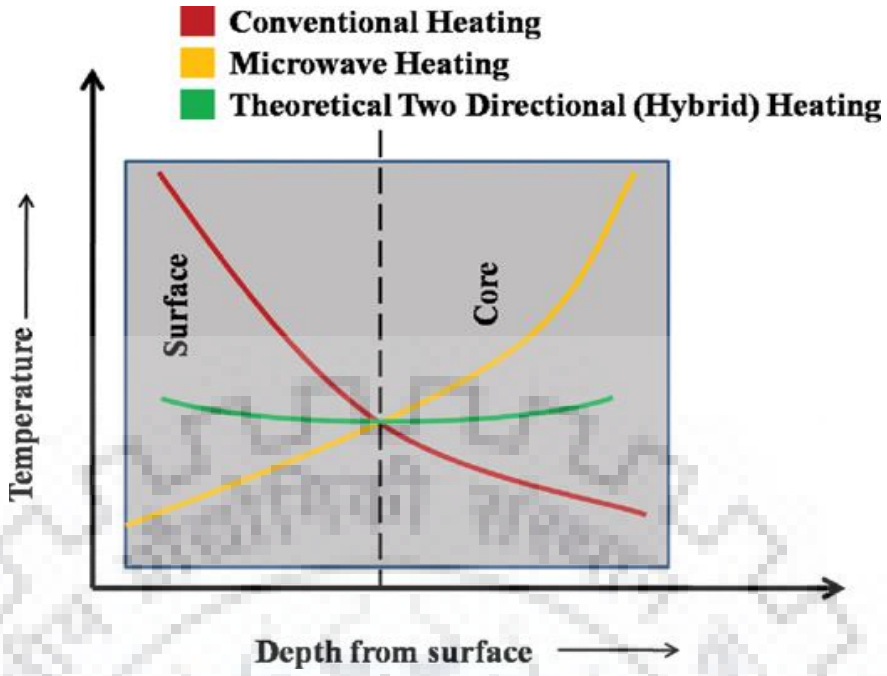


Figure 1.7 Temperature and distance profile for traditional, microwave, and two directional hybrid heating [2]

Preceding the year 1999, the believe was that MMC reflect microwave radiations and the electron mist are created sharp corners because of constrained entrance of microwave radiations in MMC and that create plasma development which leads to sparks generation. The initial effort of researchers revealed that connection of microwave energy with metallic powders for upgrade of warming rate by including a couple of percent amount of metals powders which are having electrically conducting property was amid in preparing of refractory ceramics. Very few researchers through sintering, casting, brazing of chosen metallic materials until 2008, did processing of metallic powder by means of microwave energy. Intensive inquiries about were brought out for warming and sintering of metallic powders with compelling utilization of microwaves energy resulting in improved metallurgical and mechanical properties in compacted samples.

1.6 ADVANTAGES OF MICROWAVE MATERIAL PROCESSING

The points of interest offered by microwave heating phenomena are numerous over traditional heating because of the following reasons:

- The direct ingestion of microwaves inside materials permits volumetric heating which produces upgraded diffusion rates, diminished power utilization, and lower process time. The immediate exchange of energy wipes out losses, which is related to heating of furnace,

furnace walls etc. These give higher rates of heat movement in contrast with traditional techniques and higher temperatures can be accomplished in shorter time as shown in fig 1.8.

- It was analysed that the defects present in the heated samples prepared by using microwave energy was less because of higher heating and diffusion rates in the process. Enhancement of different parameters gives improved microstructure for example temperature rate of heating and temperature angles. The procedure of quick warming in microwave preparing gives a few advantages, for example, high sintered thickness and better microstructures contrasted with moderate heating traditional procedures. These better microstructural advancements, better normal grain measure, higher densification parameters, and lower porosity imperfection result in improved mechanical properties contrasted with traditional processing strategies [1-3]. The prerequisites for microwave sintering incorporate high temperature, better warming rates, uniform temperature slopes, and uniform warm circulation inside the sample. Sintering through ordinary routes does not give uniform heating and temperature slopes, which powers to keep the conservative at high temperatures and for higher handling times. Its moderate warming rates combined with its higher handling time has prompted the distortion and coarsening of compacted samples.

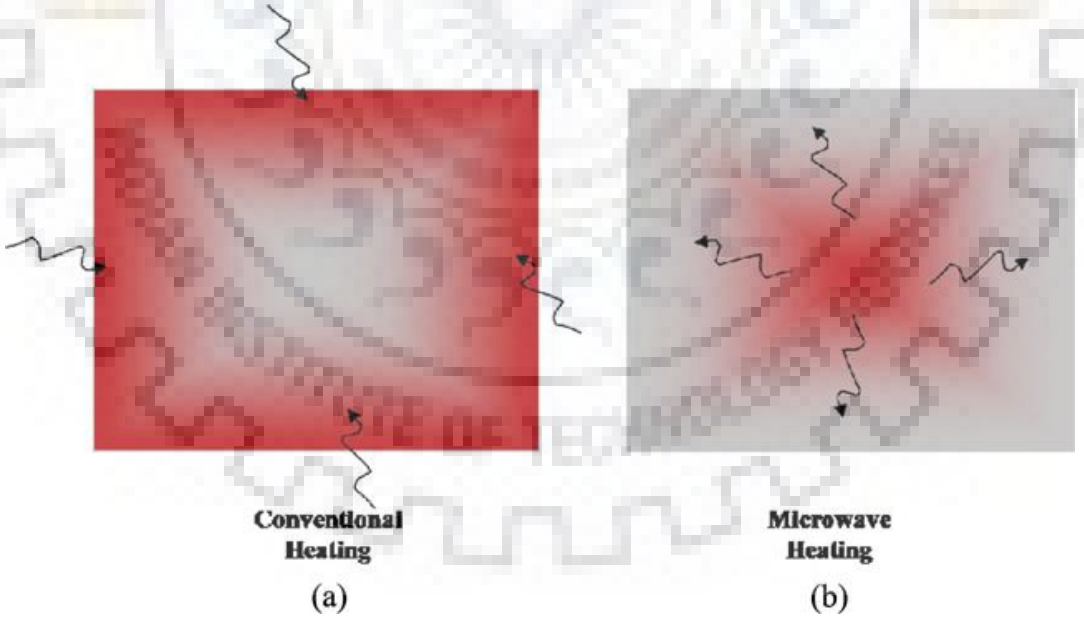


Figure 1.8 Heat generation pphenomenon in (a) traditional heating (b) microwave heating [2]

- The volumetric heating phenomenon promote selective and uniform heating of materials that causes reduce time in processing temperature , lowers down the heat affected zones and ecofriendly as comparison to other traditional methods.

Chapter 2

LITERATURE REVIEW

2.1. WORK BRINGING OUT THE DIFFERENT SIZE OF THE REINFORCEMENT USED

There are various experiments have been performed to find out the ultimate results after creation of metal/matrix composites. Some of result of these kind of experiments are discussed below:

Vaidya et al (1996) [4] showed how the variation of the SiC particle reinforced with Mg alloy AZ91 based composite by volume fraction, effects the High cycle fatigue properties of it. AZ91D Mg alloy metal matrix composites processed by two methods one is squeeze casting and other is extrusion and reinforced by either 15 μm or 52 μm size SiC particles, mixed at both 20% and 25% volume fraction in the composite. SiCp increased the strength and modulus of AZ9lD, with finer reinforcement which provides more strength to the metal matrix composite. Reinforcement with 20 or 25 volume fraction of 15 μm SiCp gives higher fatigue performance with respect to monolithic AZ91D.

W.L.E. Wong et al (2006) [6] reported that use of hybrid length of the reinforcement improves the thermal stability and hardness of the composite when compared the singular reinforced material. SiC particles were reinforced in pure magnesium powder which was mixed in a RETSCH PM-400 mechanical alloying machine at a speed of 200 rpm for 30 minutes and then compacted using uniaxial compaction machine and then sintering process was formed by using microwave. Sintering time was 25 minutes for this experiment. Sintered Mg composite reinforced with SiC particles of two different amount shown better hardness and thermal stability in comparison to monolithic reinforced composites.

K.K. Deng et al (2012) [9] reported about the various results regarding the size of particle on microstructure and mechanical characteristic of SiC/AZ91 magnesium alloy composite. Authors concluded that uniformly distribution of particles depends on the particle size. The larger size of SiC are more uniformly distributed in magnesium matrix in the same condition of volume fraction. Particle distribution and grain refinement is important factors that influence the mechanical properties of MMC, Submicron SiC/AZ91 reinforced MMC exhibits good mechanical characteristic at 2% volume fraction, but at 5% and 10% volume fraction of micron

SiC/AZ91 composite shows better strength. Also related optical micrographs of the experiment has been shown in Fig 2.1.

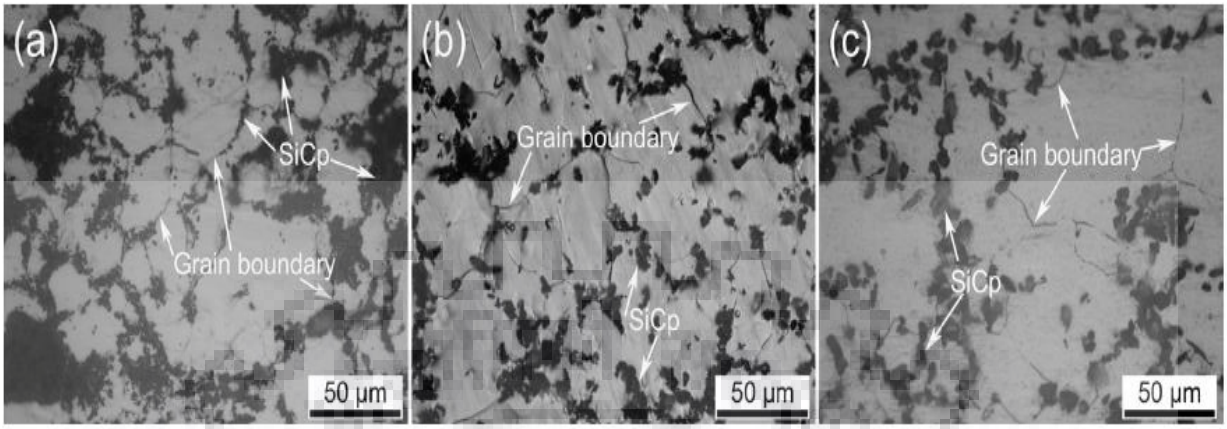


Figure 2.1 Optical micrographs of casted SiCp/AZ91 Mg metal matrix composites in 10 vol.% of SiC particle size of (a) 0.2 µm, (b) 5 µm and (c) 10 µm, respectively [9]

2.2 INTERACTION OF METALS AND MICROWAVES

Mishra and Sharma (2016) [3] represents the wonderful explanation regarding heat interaction between different materials and microwave which is very helpful to understand the exact heat interaction conditions inside the microwave which pictorial illustration is presented in fig 2.2. Mechanism that cause interaction of microwave energy in various materials like metals, non-metals and composites talked about utilizing reasonable illustrations.

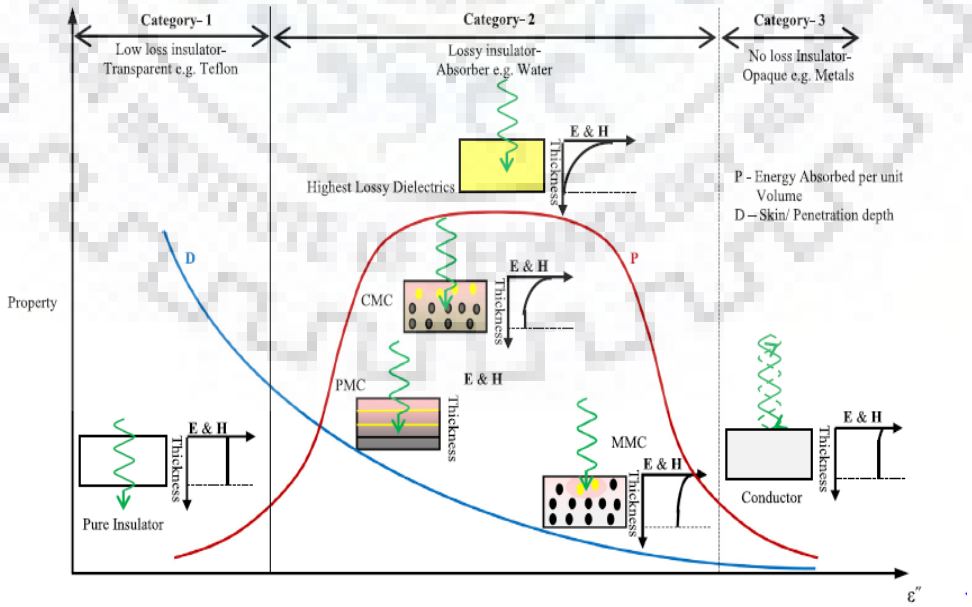


Figure 2.2. Microwave interaction with different types of materials [3]

It has been seen that while microwave energy absorption three losses are played significant role in the heating of metal based materials i.e. magnetic field based loss effects, dipolar loss and conduction loss that led to the electric field effects in microwave heating of non-metals. Difficulties in handling of composites have been distinguished from the accessible literature.

2.3 CHARACTERISTIC OF AZ91 MG ALLOY

Mondet (2016) [9] investigated the influence of $Al_{11}Mg_{12}$ precipitation by sintering temperature and grain size of sintered samples and showed how SPS technique is better than other conventional sintering processes. Fine intergranular $Al_{12}Mg_{17}$ precipitates found in sintered as shown in Fig 2.3. On high sintering temperatures, the populations of precipitate gradually dissolve until their complete dissolution above 380 ^oC. A very few amount of $Al_{12}Mg_{17}$ precipitate found in between 380 ^oC and 500 ^oC. The impact of $MnAl_4$ precipitates were negligible impacted and may be examined that their size and fraction remained unreacted in the sintering process. From the fig 2.4, it was clearly visible that on increasing sintering temperature hardness decreases. The paper also pointed that in SPS process, the lack of ductility, under tensile testing, remains an issue for AZ91 Mg alloys processed by SPS.

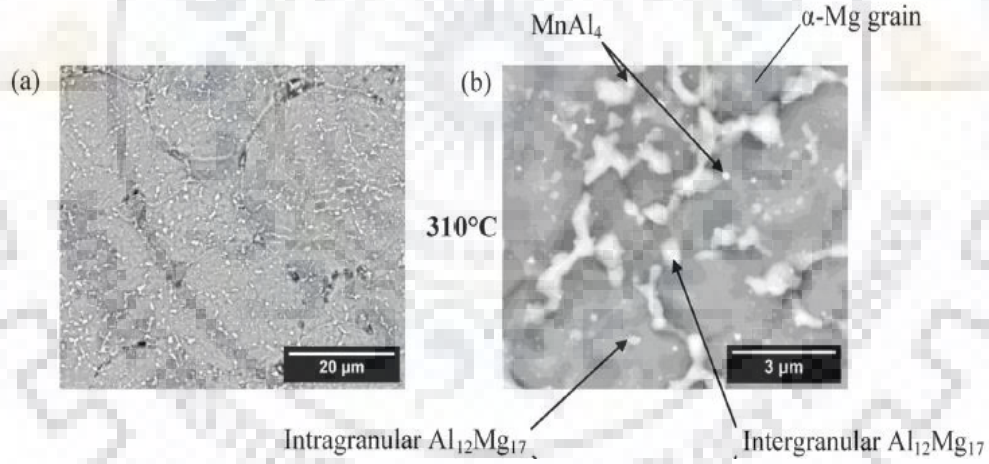


Figure 2.3. SEM images for AZ91 Mg alloy at 310 ^oC [9]

Trojanová (2009) [21] did study on AZ91 Mg alloy reinforced with SiC particles and then additional amount of Si particles were added. It resulted the formation of an ‘‘in situ” composite (Mg–Mg_2Si) which gave a strong bonding between Mg_2Si and the matrix interface as it is shown in SEM images (Fig. 2.5). The effect of reinforcing of SiC and Mg_2Si particles found to be decreased with increasing in temperature. Mechanical properties were also analysed in this study at different temperatures. A ductility improvement was found at 200 ^oC and temperatures over 200 ^oC.

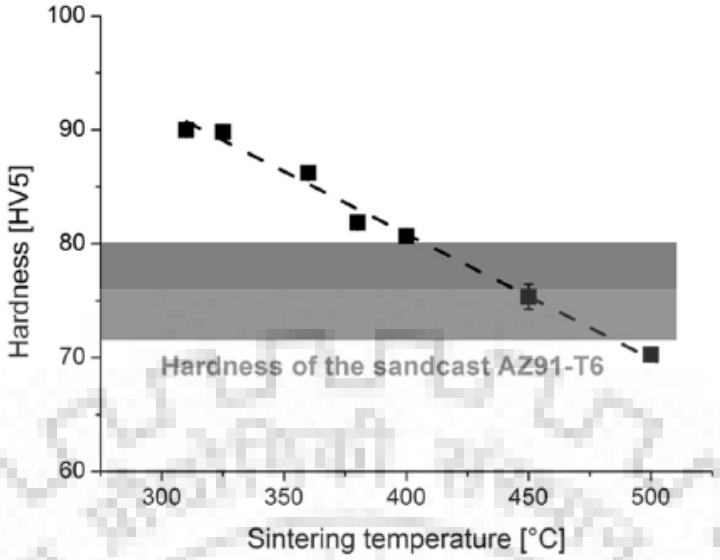


Figure 2.4 Vickers hardness AZ91 mg alloys at different sintering temperature [9]

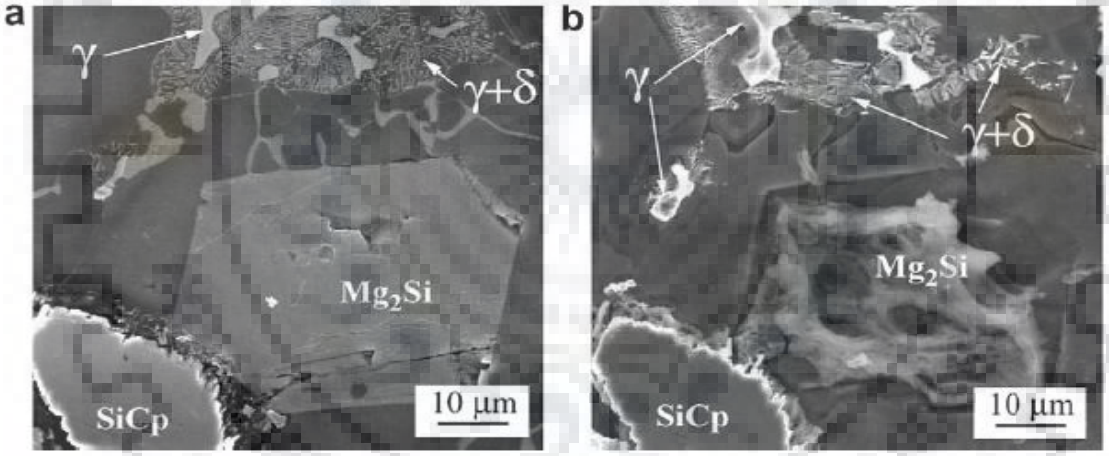


Figure 2.5 SEM images shows SiC particle with primary Mg_2Si coarse dendritic crystal after using glycol etching (a) and deep etching (b) [21]

2.5 MICROWAVE VERSUS CONVENTIONAL SINTERING

Oghbaei and Mirzaee (2010) [22] gave a brief of central parts of microwave hybrid and hybrid sintering and after that its favourable circumstances against the traditional techniques. Some microwave sintering applications are likewise referenced which so far have displayed favourable circumstances of this novel process.

Madhan (2019) [24] this repot comprises the production of Al_2O_3-SiC ceramic composites by using conventional and microwave sintering process. The sample was prepared by mixing of x

wt. % SiC, (x=5, 10, 15 & 20) in Al_2O_3 powder and milled for 180min. Then the samples were compacted at 60MPa for 30sec and sintered by both methods. After sintering hardness, densification, grain size, phase variation and micro structure were examined &comparison were made for the methods.

Wong and Gupta (2007) [23] used the concept of hybrid heating to develop magnesium composites with different amount of Cu particles mixed with it. The process was done by using frequency 2.45 GHz and power 900 W for microwave generation. The concept of using susceptor also applied for hybrid heating of the compacted sample In this it also revealed that microwave processing has capability to make denser product than other conventional ones and gives less porosity in the sintered product. Cu particles blended with the matrix gives more hardness, increase yield strength, elastic modulus, ultimate tensile strength of the metal matrix composites. It was also observed that the tensile properties also increased by processing through microwave energy route

2.6 RESEARCH GAP AND OPPORTUNITIES

Composites are become the lead demand in various applications such as automobile, aerospace, defence and medical science. They have specific properties, which required prominent processing techniques so that final product can fulfil the desired customer‘s needs. However, there are various existing processing techniques are present still material efficient, energy and time efficient process are required to develop. Consequently, microwave material processing is a revolutionary process for the current scenario as it gives the better mechanical, chemical, physical properties than other conventional processes. The operation procedure is not complicated, requires less floor area. From the literature survey, microwave sintering process is suitable for development of new composites with adequate attributes.

It is also observed that MMC can be easily processed by using susceptors in MW i.e. hybrid heating phenomenon. As Mg based alloy are very famous among researchers due to its valuable properties. There is much work has been performed or Mg and Al based alloys in conventional as well non-conventional routes of processing. However, AZ91 Mg alloy reinforced with SiC particles with different weight percentage has not been attempted through microwave sintering. Hence, in this work, development of AZ91/SiC composites using microwave sintering has been proposed and successfully developed with some beneficial results.

2.7 OBJECTIVES AND SCOPE

The objectives of this work are as follows:

I. To develop AZ91/SiC composites through microwave sintering

II. To measure density of AZ91/SiC composite.

III. To characterise the developed AZ91/SiC composite

Chapter 3

MATERIAL SELECTION AND ITS APPLICATION

Material selection for any application is the most important factor as it decides the performance and efficiency of the final product where it used. According to applications and their performance, material should be choose and processed by suitable manufacturing process. In this work, Mg based alloy has been used for development of MMC by adding SiC as reinforcement. The brief explanation why these materials have been selected is given in next paragraph.

3.1 BASE MATERIAL

Magnesium comes under the lightest structural metal. Magnesium alloys are 33% lighter than aluminium, 61% lighter than titanium and 77% lighter than stainless steel making them promising competitors as trade material for these metals. As far as accessibility, magnesium is the sixth richest component in the world's hull containing 2% by mass and third most dissolved mineral in seawater with an accessibility of 1.1 kg/m^3. Its plenitude in planet earth is sixth most accessible in earth outside, third most accessible in seas, and fourth most inexhaustible cation in the human body. Moreover, simplicity of reusing, predominant explicit mechanical properties, high damping capacity, and critical electromagnetic protecting ability are a portion of the characteristics that warrant the across the board utilization of magnesium based materials in both building and biomedical applications. Different points of interest related with magnesium based alloys include:

i. Requirement of energy is low in processes where solidification requires
ii. It can be easily processed by using traditional solidification methods.
iii. Better machinability gives higher tool life generally 5-10 times higher than hard materials.
iv. Ability to be processed using conventional plastic deformation processes at speeds matching with that of conventional structural materials.

Above points, present the interesting facts about Mg based alloys that to be incorporated in designing applications with insignificant infrastructural ventures and varieties, basic prerequisite that influences basic leadership of industrialists to deliver better-required materials. Moreover, decreasing in cost of magnesium and the ability to decrease carbon

impression are factors that are setting off the substitution of aluminium based alloys step by step and continuously over a decade ago. The cost related factors guarantees steadiness of market costs of the item without stressing customer's pocket. Likewise, worldwide are moving to reduce the carbon dioxide outflows by 2 billion tons to maintain the temperature ascend to inside 2°C from the preindustrial levels as proposed in Paris understanding will fuel the expanding utilization of Mg based alloys in not so distant future. It is broadly foreseen that magnesium will supplant aluminium in 2020s in a similar manner as how aluminium supplanted steels in 1980s.

3.2 TYPE OF REINFORCEMENT

Magnesium based composites generally use ceramic reinforcements. They range from carbides, borides, or oxides. From all the ceramic reinforcements, the most broadly explored and utilized reinforcing ceramics with pure magnesium and commercial grade magnesium alloys is the SiC particulates. SiC particles are mostly chemically stable in many molten Mg alloys and SiC particles has generally better wettability with Mg in contrast with the other ceramic type of reinforcements [1, 2].

Chapter 4

EXPERIMENTAL WORK

4.1 MIXING

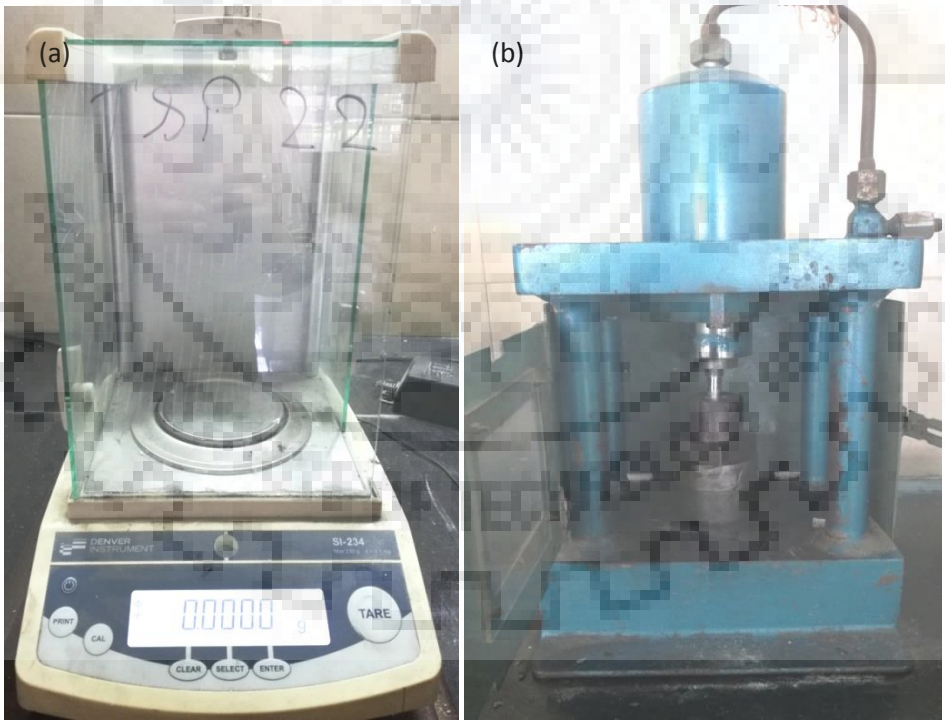
AZ91 Mg alloy (80micron particle size) was uniformly mixed with SiC (80 micron particle size) in 5%, 10% &15% by weight in V blender machine (Fig. 4.1) and mixing time was taken as 4 hours for ensuring the uniform mixing of powders.

Figure 4.1. V- blender machine

4.2 COMPACTION

Before compaction,the measured powder was mixed with Polyvinyl alcohol (PVA) as PVA is binder for the billet preparation.So firstly PVA was melted on hot magnetic (Fig 4.2) plate andthen it was mixed(5% by weight for each sample), properly so that no lumps was formed and then weighted each sample to make h=5mm,d=13mm, pallet in press of maximum capacity 25T at a 9T pressure. Fig 4.3. shows the weighing machine, hydraulic jack during compaction of powdered sample, hydraulic jack during removal of compacted sample and input segment of Hydaulic jack machine. Fig 4.4. shows the compacted samples.

Figure 4.2. Hot magnetic plate machine



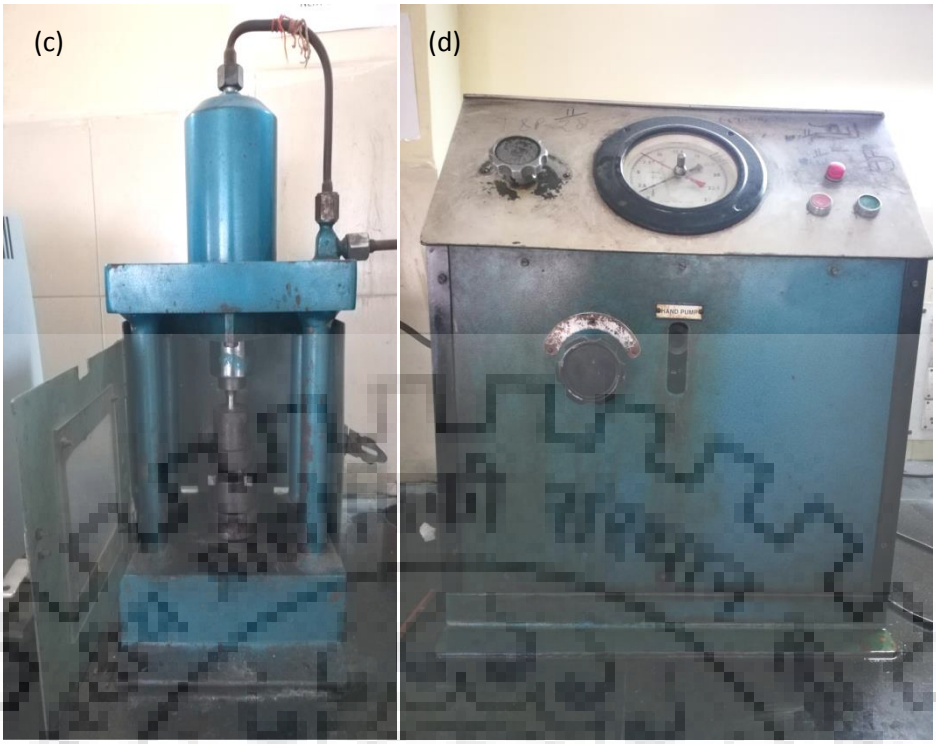


Figure 4.3. (a) Weighing machine, (b) Hydraulic jack during compaction of powdered sample, (c) Hydraulic jack during removal of compacted sample and (d) Input segment of Hydaulic jack machine

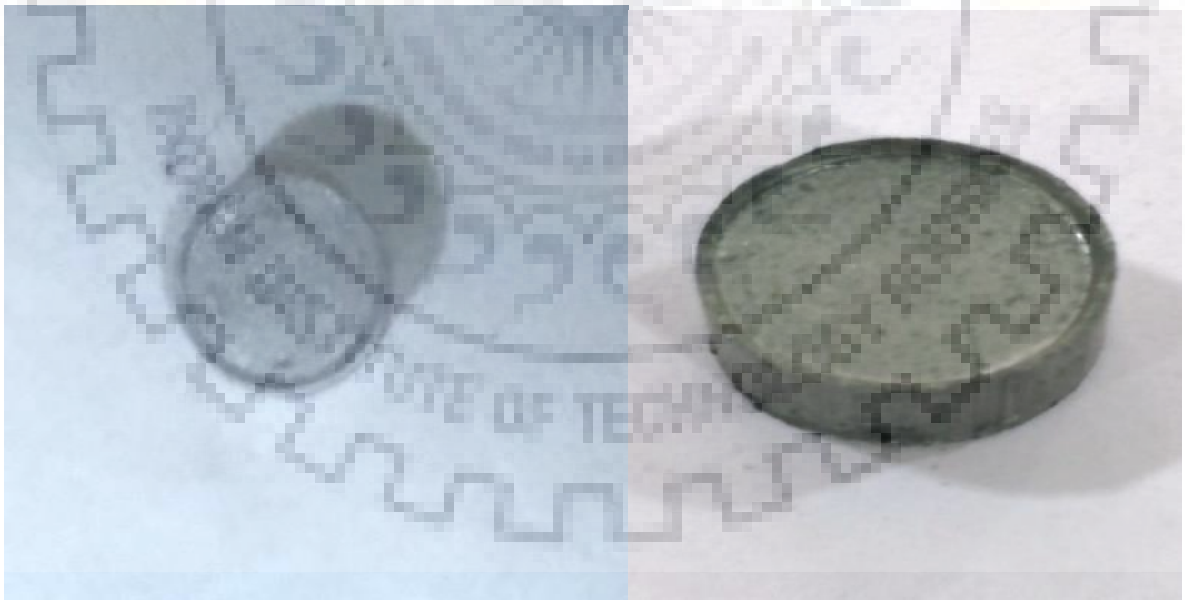


Figure 4.4. Images of compacted samples

4.3 PREPARATION OF MICROWAVE SINTERING SETUP

Peapered sample or billet was kept inside the Alumina crucible(99.7% of alumina from Ants ceramics Mumbai). As shown in the Fig 4.5, there are two crucible and SiC is kept between them to as a Susceptor for the sample. Alumina glass wool is used as an insulator or the process. After this, the whole setup was kept inside the microwave and the base material is refractory brick for it.

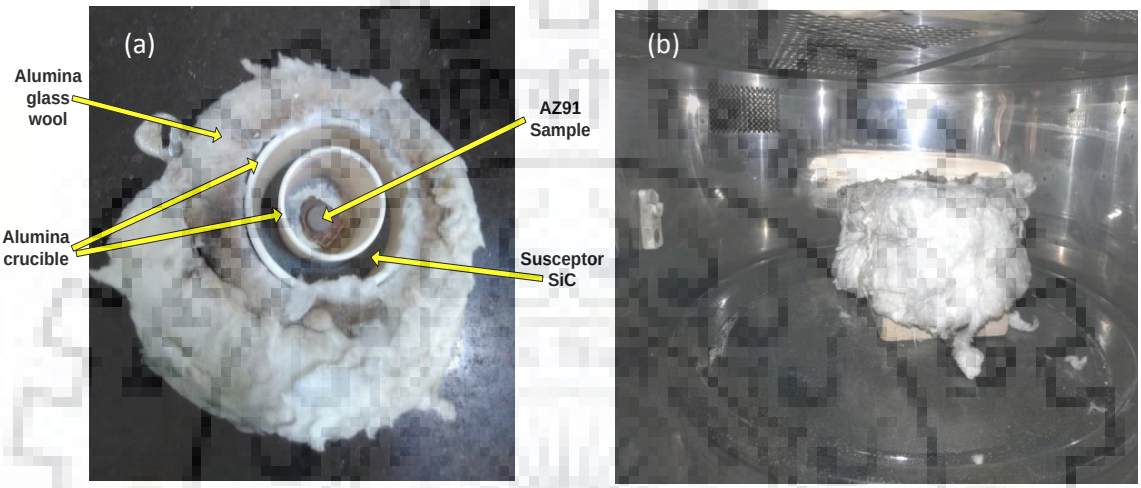


Figure 4.5. (a) Microwave sintering setup, (b) Setup inside the microwave

4.4 MICROWAVE SINTERING

Sintering was done in microwave at 900W for various time periods on domestic microwave which is shown in Fig 4.6.

Figure 4.6. Domestic Microwave applicator

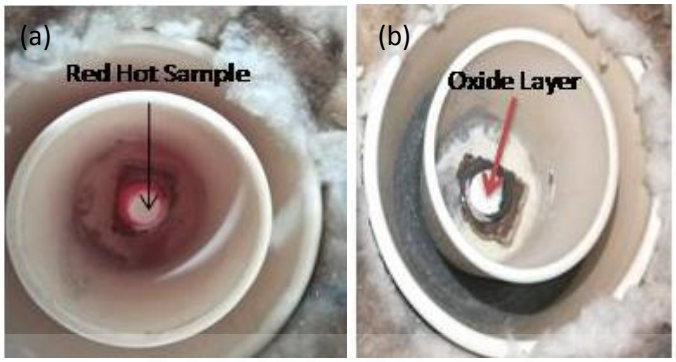


Figure 4.7. (a) Showing the red hot sample after microwave sintering, (b) Oxide layer formation on sintered sample

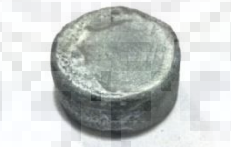
Table 1. Table for comparision of microwave sintering at various temparature

Diameter	Height (before sintering)	Sintering time (min)	Sintered sample
1.3 cm	0.5 cm	13	
1.3 cm	0.5 cm	10	
1.3 cm	0.5 cm	8	
1.3 cm	0.5 cm	5	

Sintered samples at sintering time 8 min was used for futher analysis because at 13min and 10 min compacted samples were burned which was not desired for futher analysis and at 5 min, sintering was not done completely as shown in table 1.

The sintered samples are shown in table 2 with their short names which has been used in results and discussions for convinient purpose.

Table 2. Showing all the 4 different sintered sample's images and their short names

Samples	Image of Sintered Sample	Short name
Pure AZ91 Mg Alloy		Sintered sample 1
5% SiC + AZ91 Mg Alloy		Sintered sample 2
10% SiC + AZ91 Mg Alloy		Sintered sample 3
15% SiC + AZ91 Mg Alloy		Sintered sample 4

4.5 FAILED EXPERIMENTS

Issues faced

- Availability of AZ91 Mg alloy in form of fine sized powder was challenging because it is a costly material and has been made for specialised order in larger quantity (as compared to our requirement)
- Compaction of the powder sample was really a tough job because AZ91 Mg alloy easily react with atmospheric air and form oxides which reduces its binding capability. Mg alloy has good binding capability at 450 MPa pressure. Usually Mg alloy does not

require any binder but due to oxide formation, we used binder PVA for compaction and after mixing PVA in powder sample, compaction was done before it react to the environmental air.

- During compaction of powder, punch and die got distorted due to high pressure applied on it repeatedly. Precise machining was done on the punch and die to make it perfectly align again.
- We use domestic microwave, which does not show any temperature at any time of the heating process. That's why it was difficult to find out perfect time and temperature for the heating process as shown in Fig 4.8. The sintering time was chosen on the basis of hit and trail method.

Figure 4.8. Trials for microwave sintering but not following

4.6 SAMPLE PREPARATION

Grinding and polishing

Coarse grinding was carried out on different sintered samples using the Silicon carbide sand papers of grit size 150, 220, 400, 600, 800, 1000 and 1200 on polishing machine (Fig 4.9).

Water was used as the lubricant during the grinding process. Further fine grinding was carried out using the grit size of 1500 and 2000. During grinding it was observed that cracks on the sample surface are unidirectional, that means all the scratch present on the surface are in one single direction, as any change in the direction of the polishing will lead to randomness of the scratch positioning which will hinder the view of the microstructure of the surface when viewed in the microscope. Also, it was ensured that the sample is parallel to the paper surface.

Once all the marking become unidirectional, the sample was then polished by turning it by 90°. Bearing this the mind, the surface is polished from all the sand paper grades in the same direction from beginning till the end until the surface becomes scratch free i.e. no scratch is left on the surface of the sample.

After fine polishing by sand paper of grade 2000, the samples were next polished on velvet cloth. For this 0.25 µm diamond paste is used together with the diamond lubricant. This method of polishing leads to mirror like finish on the surface on the sample. Care should be taken so that the finished or polished surface should not come in contact from any rough surface or substance as it might lead to scratch formation on the surface which is undesirable.

Figure 4.9. Polishing machine

Chapter 5

RESULTS AND DISCUSSION

5.1 DENSITY

Theoretical Density

As we know the density of AZ91 Mg alloy, and rest samples density is calculated by following formula:

$$\rho_{th} = W_1 * \rho_1 + W_2 * \rho_2$$

Where, ρ_{th}= theoretical density

Density of SiC = 3.21g/cm^3

Weight of sintered sample was measured using Archimedes principle by using weighing machine which is shown in Fig 5.1. Experimental density of sintered samples was calculated by following formula:

$$\rho_s = (\rho_w * W_s) / W_w,$$

Where, W_w = Weight of sintered sample

W_o = Weight of sintered sample within water

W_s = Weight of displaced water

ρ_s = Experimental density

$$W_s = Ws - W_o$$

$$\%Porosity = (1 - \rho_s / \rho_{th}) * 100$$

After all calculations, the result is written in the table 3 such as theoretical density, experimental density and percentage porosity of the sintered samples.

Table 3. Comparision table for theoretical and experimental density and porosity in the sintered samples

Sample	Theoretical Density (g/cm^3)	Experimental Density (g/cm^3)	%Porosity
Sintered sample 1	1.81	1.571	13.2
Sintered sample 2	1.88	1.627	13.4
Sintered sample 3	1.95	1.714	12.1
Sintered sample 4	2.02	1.735	14.1

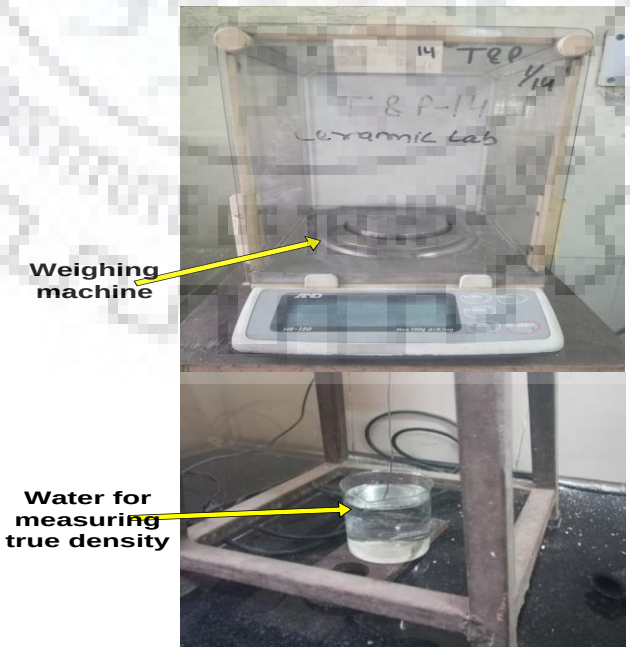


Figure 5.1. Weighing machine which support Archimedes principle

5.2 MICROSTRUCTURE ANALYSIS

Sintered Samples number 1, 2, 3 & 4 were observed under optical micrographs. AZ91 alloy has mainly two phases αMg and $\beta Mg_{17}Al_{12.}$ These phases exist along and adjacent to grain boundary. Following optical micrograph shows the diffusion of SiC particles in sintered samples, taken at 100X. From these it can be inferred that sintered sample 3 & 4 has got better diffusion of SiC particles at the boundary as compared to the other two because of they have mixed with higher amount of SiC particles. Fig (5.2-5.4) below shows the distribution of the SiC particles.

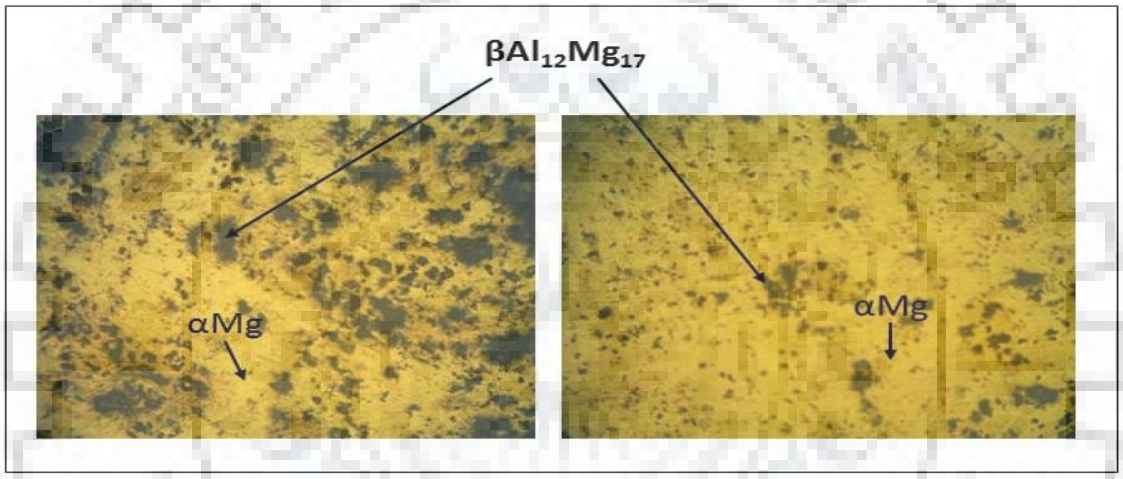


Figure 5.2. Optical micrograph showing the two main phases of sintered sample 1 using 100X magnification

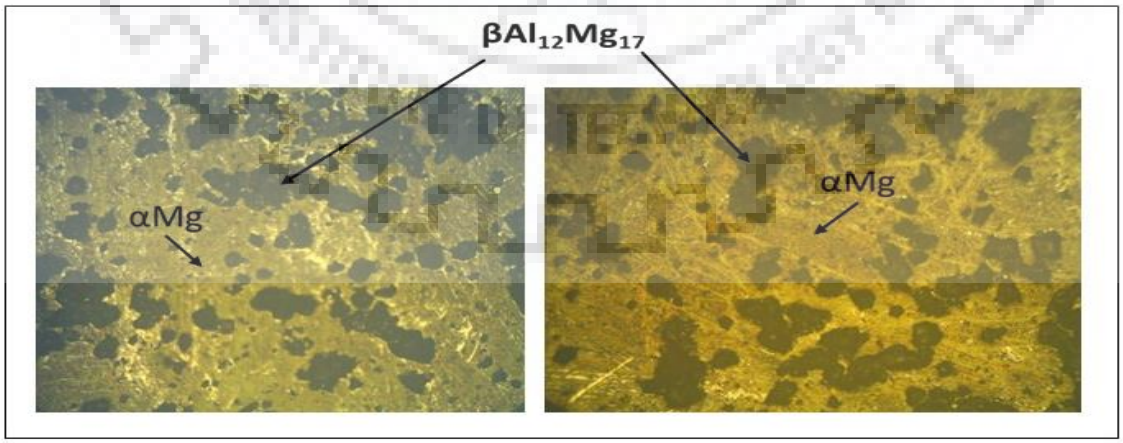


Figure 5.3. Optical micrograph showing the two main phases of sintered sample 2 using 100X magnification

It has been observed that sintered sample 3 and 4 has got agglomerations of the SiC particles (fig 5.4)

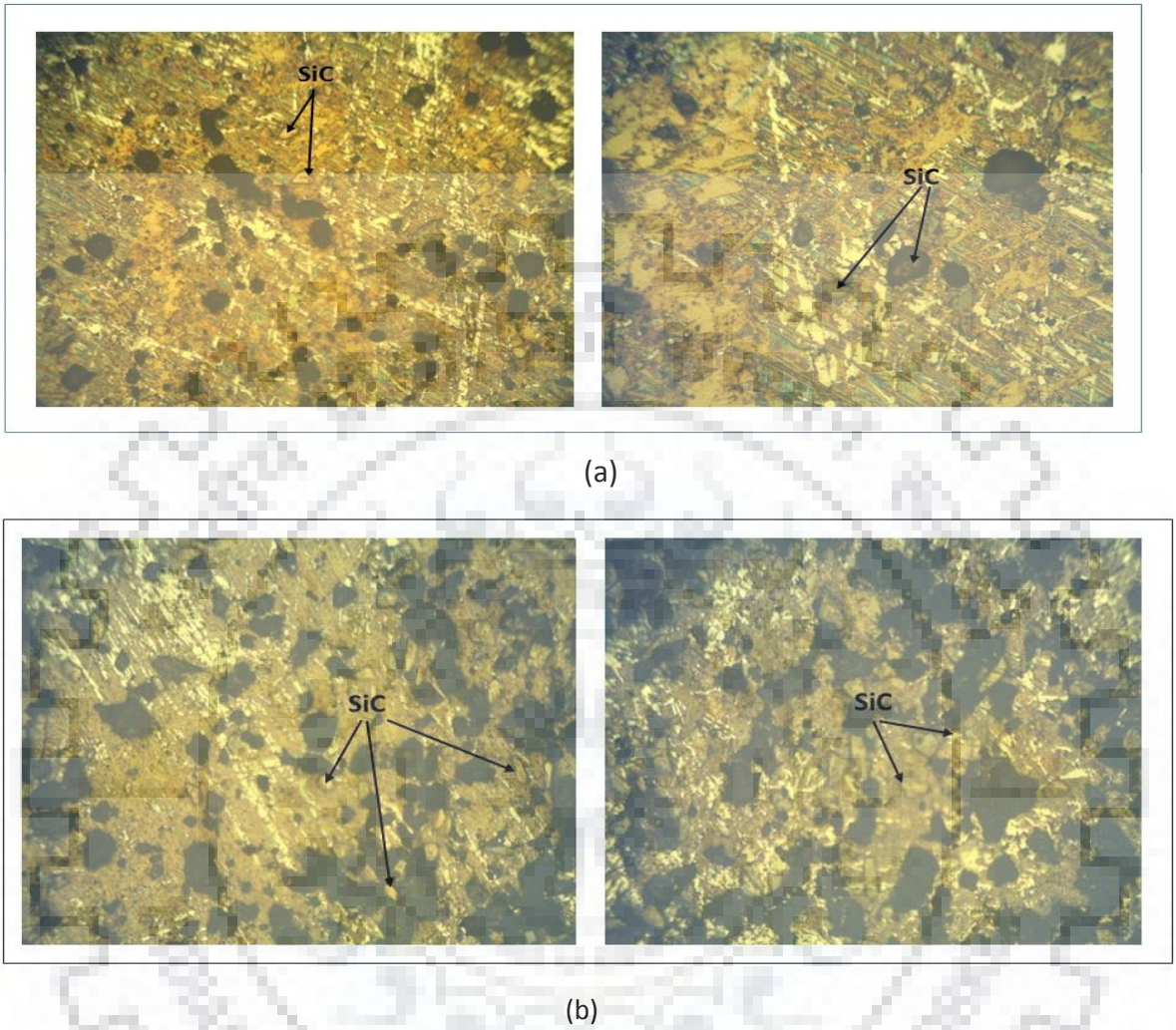


Figure 5.4. (a) & (b) Showing distribution of SiC particles of sintered samples 3 & 4

5.3 SCANNING ELECTRON MICROSCOPE (SEM) OBSERVATIONS

Typical microstructure of the microwave sintered samples are shown in the Fig 5.5, 5.6, 5.7 and 5.8. Microstructure of the sintered samples are having of the δ-phase which is solid solution, of Al in the Mg. It also shows the discontinuous precipitate $Al_{12}Mg_{17}$ i.e. the β-phase [21]. In each sample, precipitate $Al_{12}Mg_{17}$ can be noticed at the grain boundaries along with, fine $MnAl_4$ precipitates [9]. So from the SEM images we can say that the dark areas in the SEM micrograph corresponds to the αMg phase whereas the lighter areas show the $\beta Al_{12}Mg_{17}$.

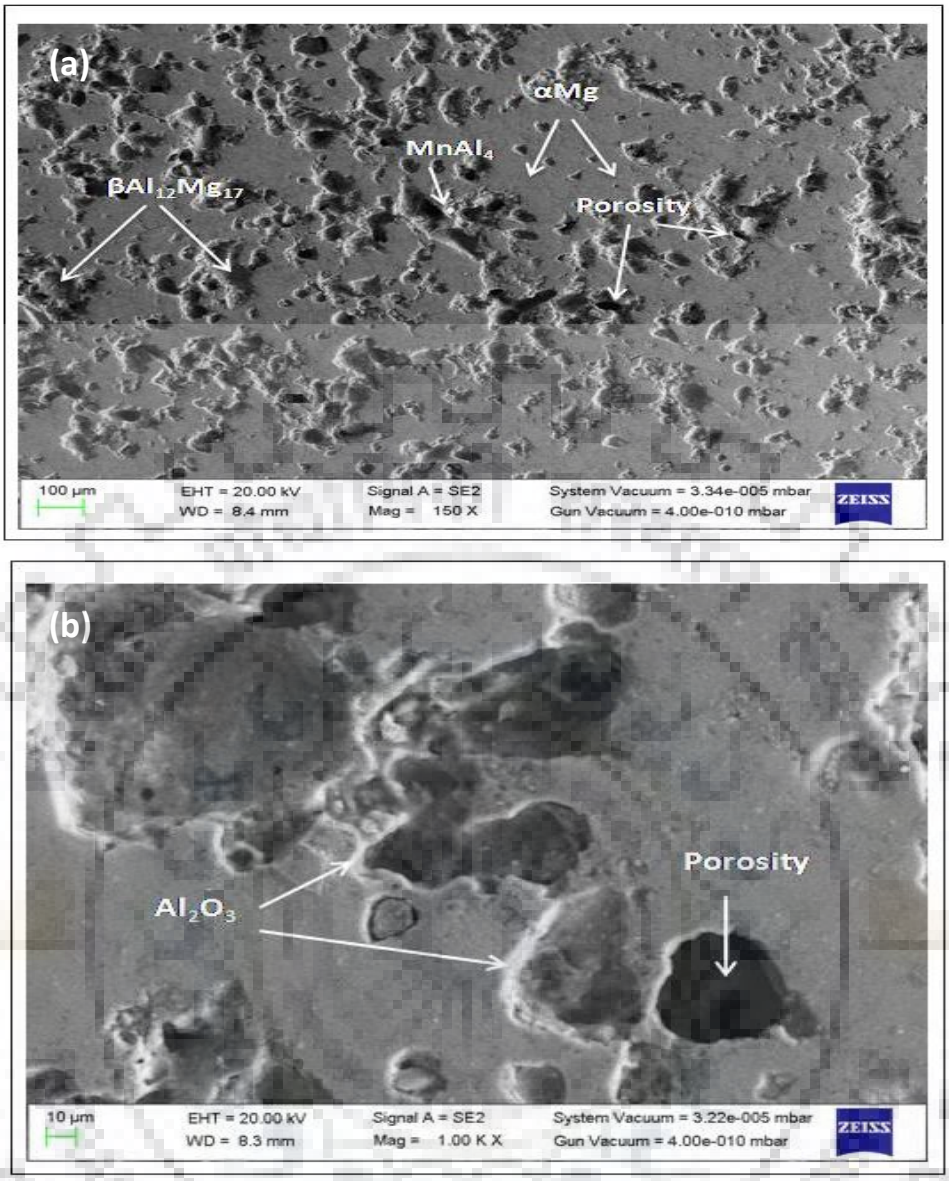


Figure 5.5. SEM images for sintered sample 1: (a) represents the different phases (b) showing oxide formation (Al_2O_3)

For better compaction of powder samples PVA was used. During sintering, PVA evaporate at 230^o C as it is its meting point temperature. So due to evaporation of PVA it causes porosity on that positions. And the porosity is clearly seen in each sample.

The addition of SiC powder in AZ91 Mg alloy leads to ascent in the Mg_2Si phase, which is formed as eutectic particles in the form of Chinese script type, or present as a primary Mg_2Si coarse dendritic particles. The Mg_2Si phase is located generally in the δ-phase grains interior, and also islands of the entrapped δ-phase are often present in some Mg_2Si coarse dendritic crystals [21].

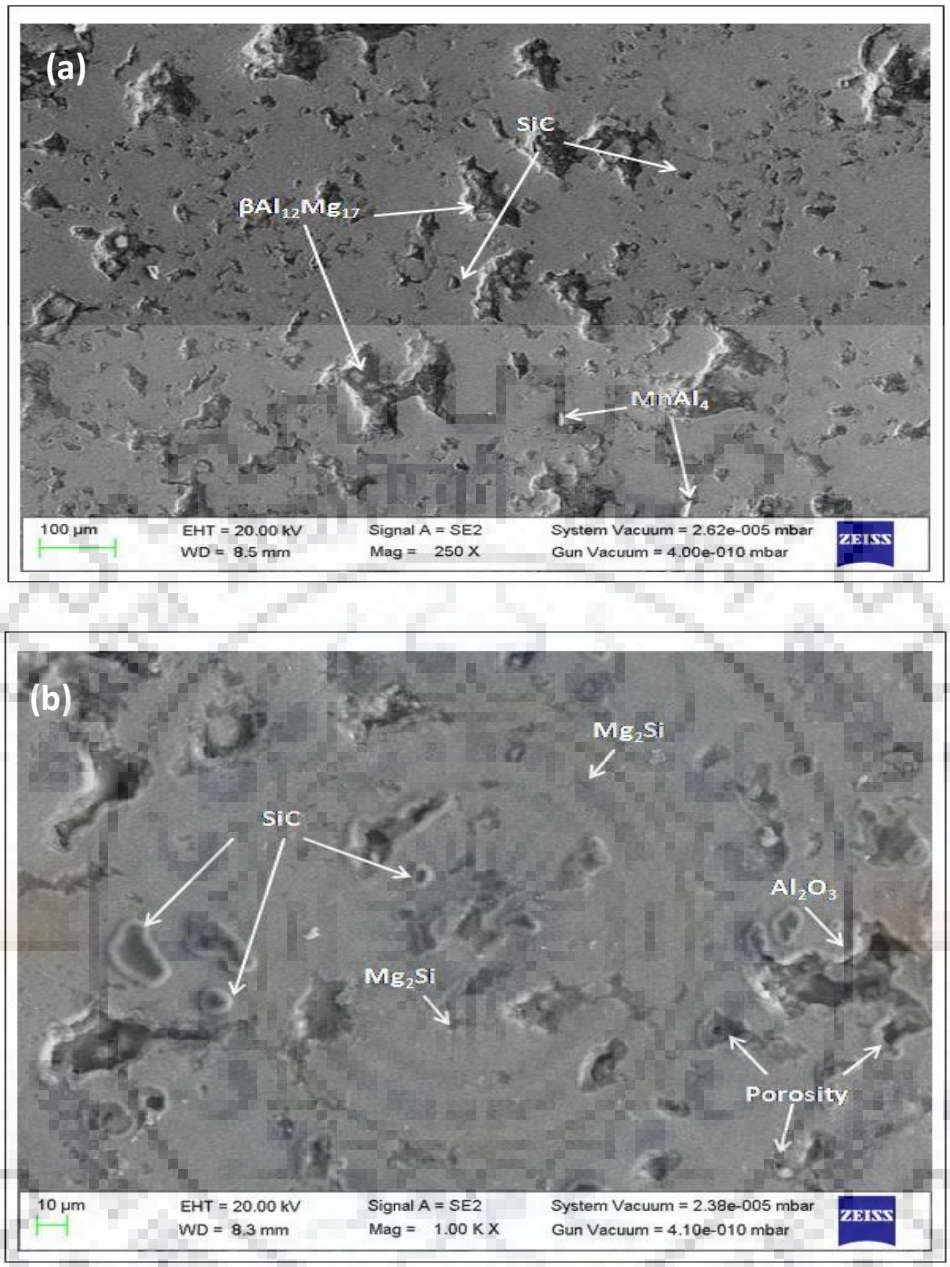


Figure 5.6. SEM images for sintered sample 2: (a) represents the different phases (b) showing the other different phases on 10µm area

SEM analysis was done to analyse, the chemical composition of the AZ91 Mg alloy/SiC interface. The excess of SiC can lead to the formation of Mg_2Si phase. The segregation of Zn has not been perceived [21].

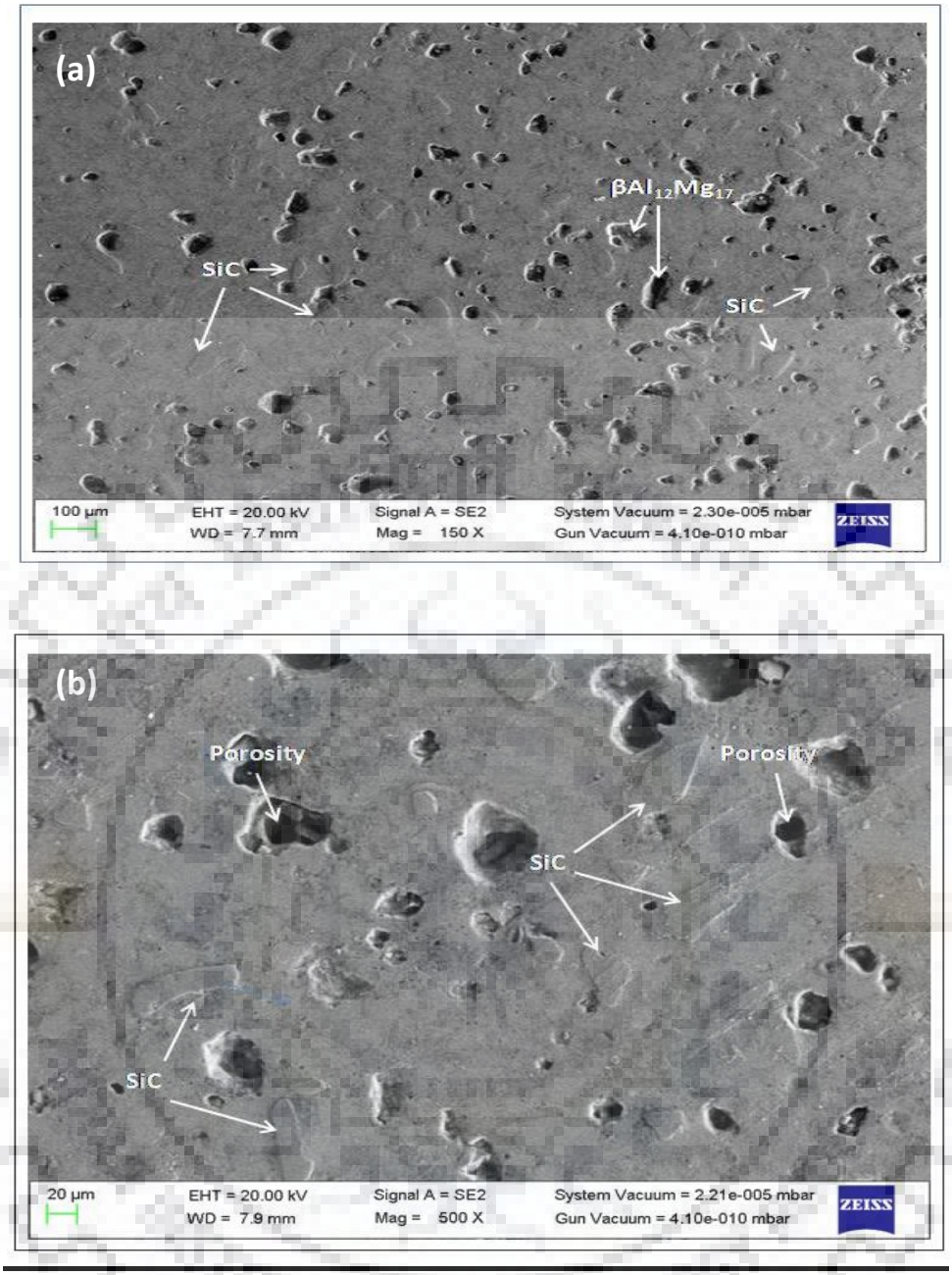


Figure 5.7. SEM images for sintered sample 3: (a) & (b) represents the different phases on 100µm & 20µm area

Sintered sample 3 is not showing the clear evidence of the presence of Mg_2Si from Fig 5.7 but the diffusion of SiC particles can be observed whereas sintered sample 4 depicts the presence of all in Fig 5.8.

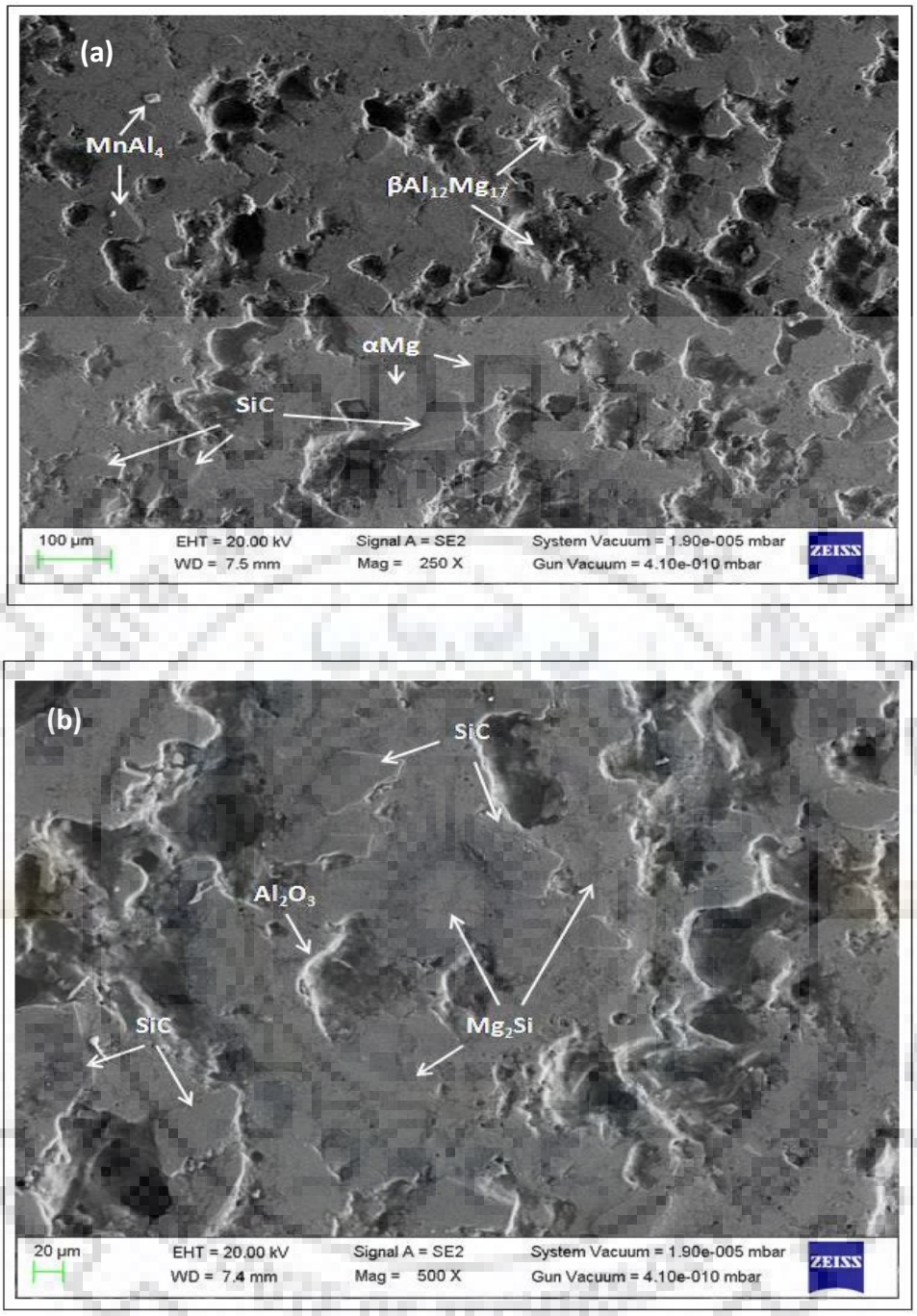


Figure 5.8. SEM images for sintered sample 4: (a) & (b) represents the different phases on 100µm & 20µm area

5.4 ENERGY DISPERSIVEX-RAY ANALYSIS (EDX)

EDX analysis was done on the sintered samples to ascertain the composition of the different phases present in the microstructure of the microwave sintered samples. The selected locations (spectrum) for elemental analysis and their corresponding results are presented in Fig 5.9, 5.10, 5.11 and 5.12. The X-ray elemental composition in Fig 5.9 (a) reveals that at spectrum 2

position presence of Mg & Al with contributions of approximately 38.17% and 36.38% respectively which indicate the presence of $\beta Al_{12}Mg_{17}$as indicated in SEM image Fig 5.9 (b). The X-ray composition study at spectrum 3(Fig 5.9. c) indicates the presence of C, O, Mg& Al elements (Fig 5.9. d).

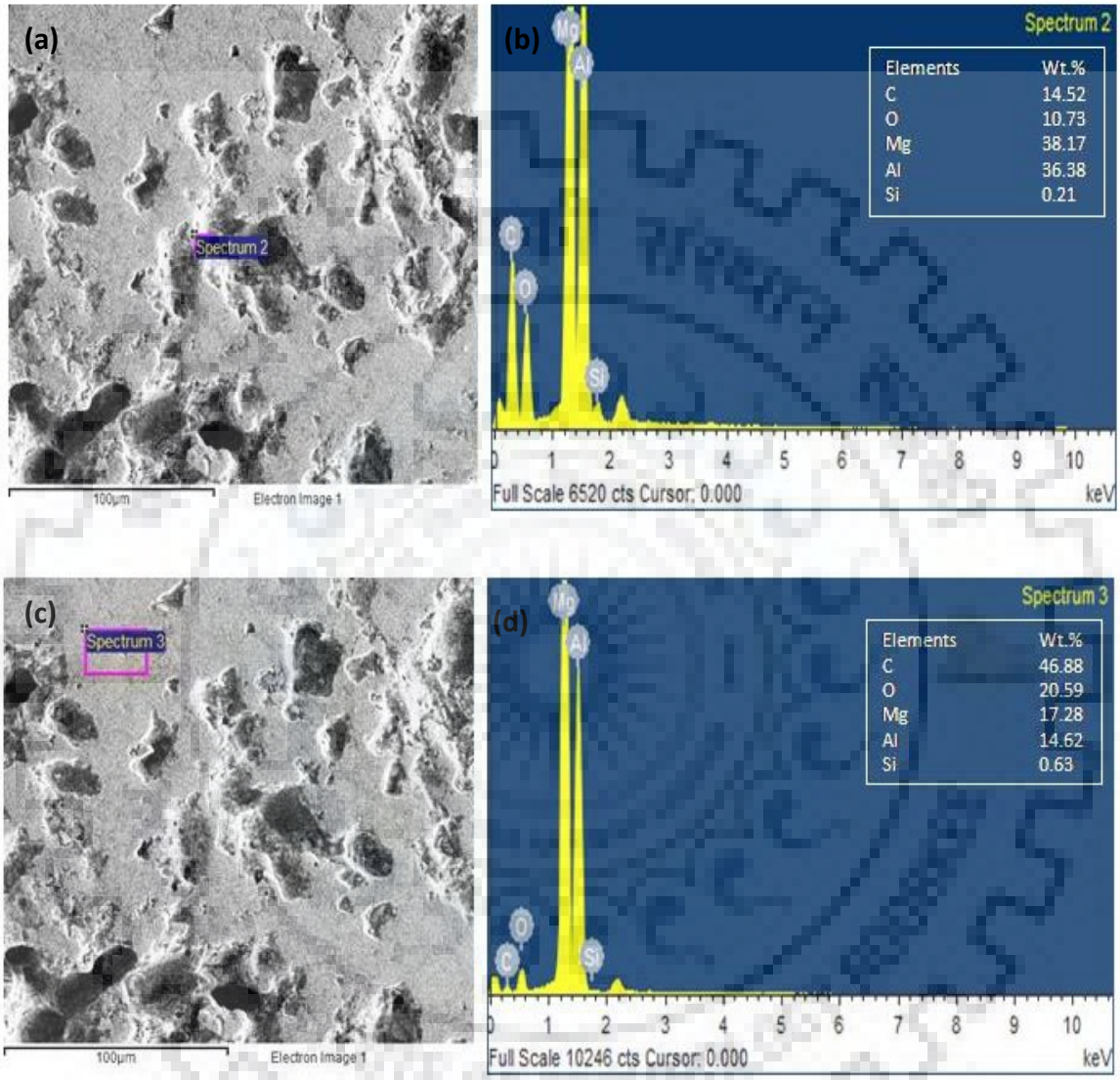


Figure 5.9. Typical EDS analysis of the sintered sample 1: (a)& (c) SEM image representing locations of EDS analysis; EDS spectra at (b) spectrum 2(d) spectrum 3

The X-ray composition study at spectrum 1(Fig 5.10. a) for sintered sample 2 indicates the presence of C, O, Mg& Al elements (Fig 5.10. b). It shows the formation of $\beta Al_{12}Mg_{17}$ and Mg_2Si phases.

In Fig 5.10. (d), it depicts the formation of SiC phase.

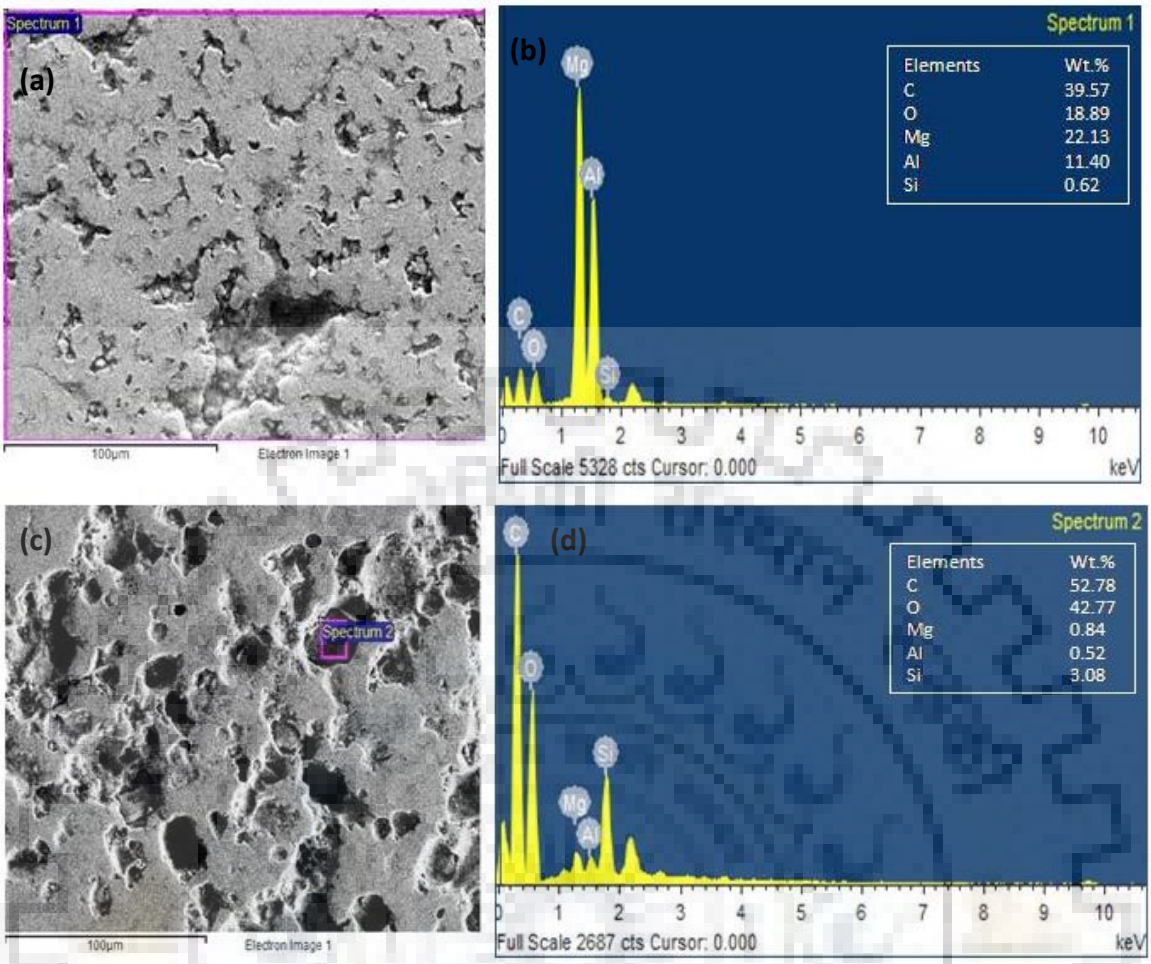
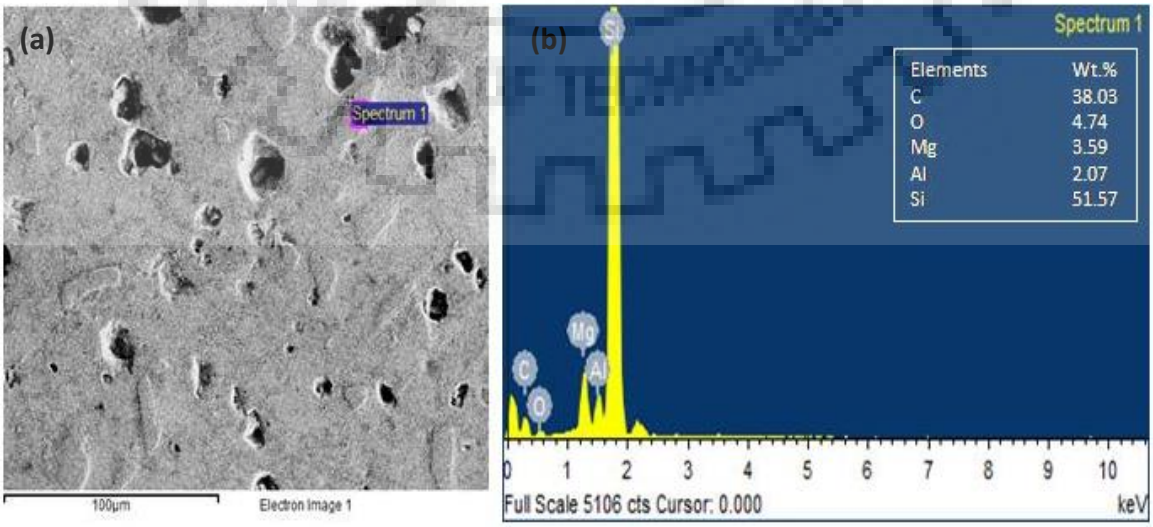


Figure 5.10. Typical EDS analysis of the sintered sample 2: (a) & (c) SEM image representing locations of EDS analysis; EDS spectra at (b) spectrum 1(d) spectrum 2

The X-ray composition study for sintered sample 3, both figures show the formation of SiC phase as it is also indicated in SEM images (Fig 5.11. b & d).



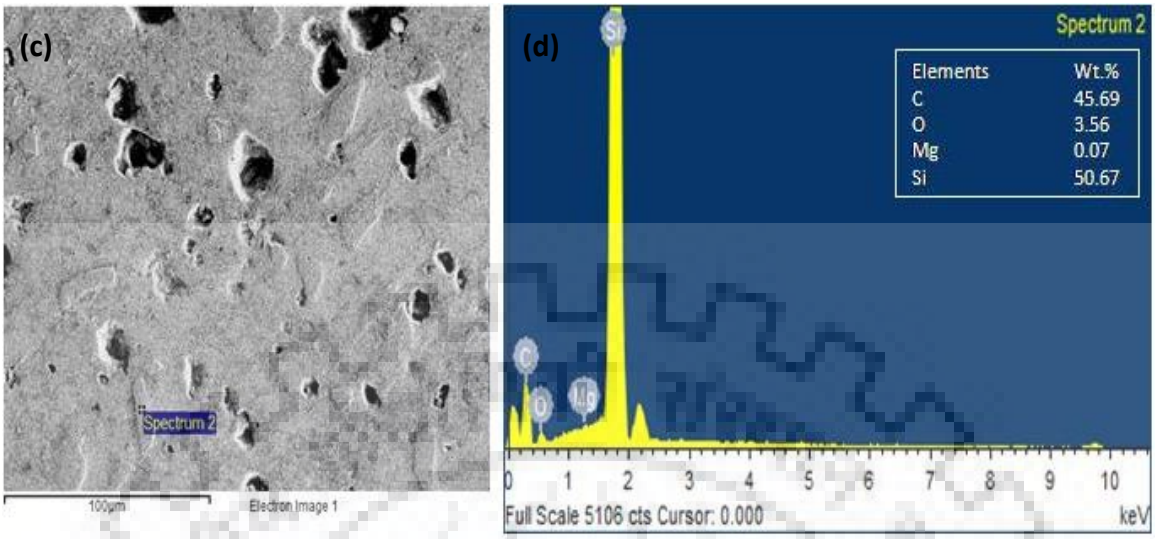
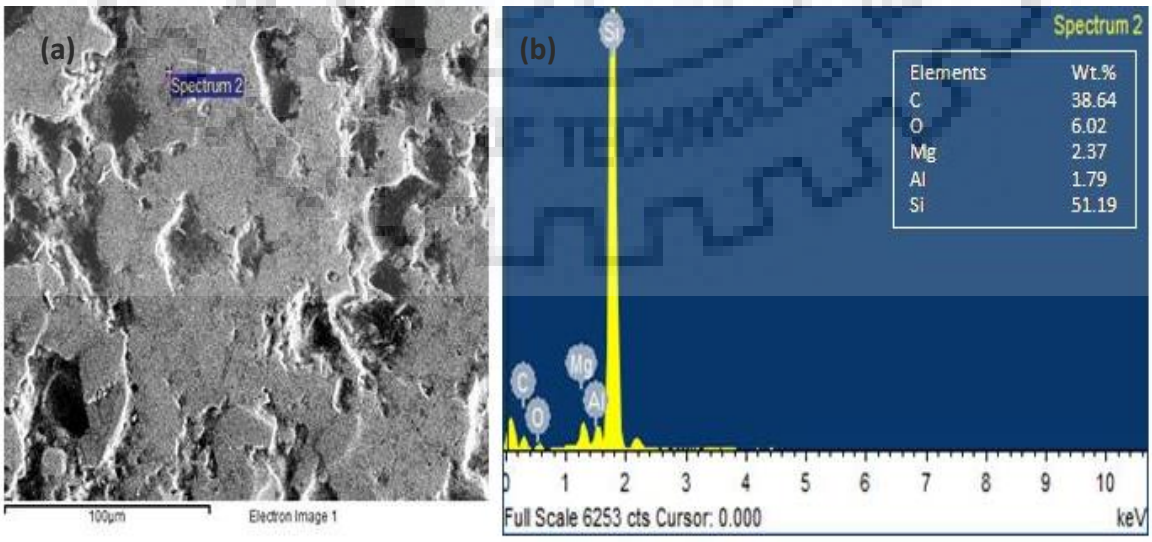


Figure 5.11. Typical EDS analysis of the sintered sample 3: (a) & (c) SEM image representing locations of EDS analysis; EDS spectra at (b) spectrum 1(d) spectrum 2

The X-ray composition study for sintered sample 4 for Fig 5.12 (a) show the presence of C and Si with contribution of approximately 38.66% and 51.19% respectively which indicates the formation of SiC phase as it is also indicated in SEM image (Fig 5.8).

In Fig.5.12 (b), it shows the presence of C, O, Mg, Al and Si in significate amounts which indicates the formation of Al_2O_3 (oxide formation) $\beta Al_{12}Mg_{17}$ and Mg_2Si phases.



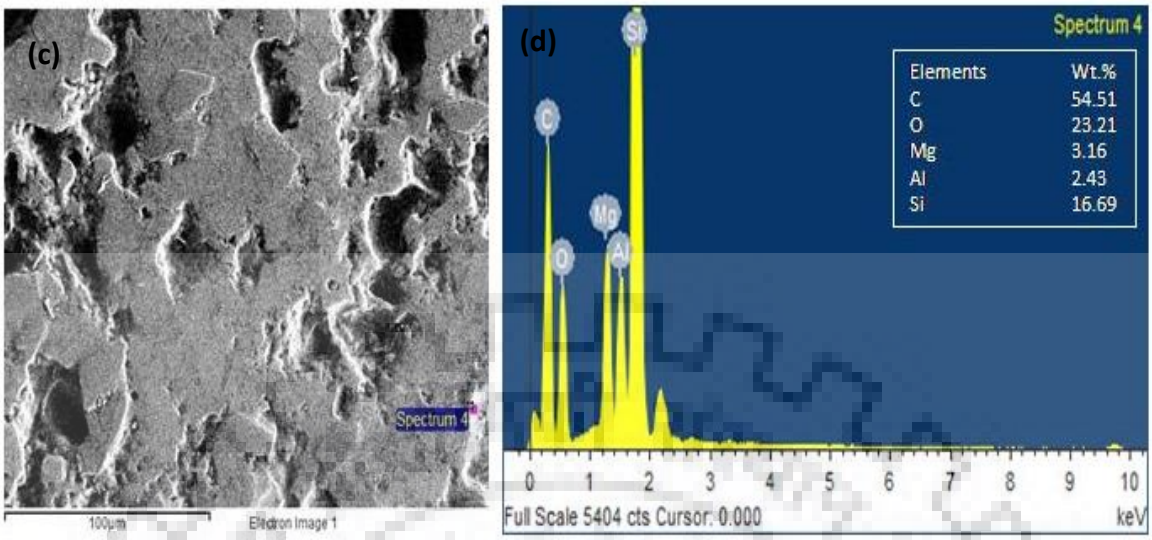


Figure 5.12. Typical EDS analysis of the sintered sample 4: (a)& (c) SEM image representing locations of EDS analysis; EDS spectra at (b) spectrum 2(d) spectrum 4

5.5 MICRO HARDNESS STUDY

Micro hardness analysis was done on the sintered samples. Load applied was 20 gm with dwell time of 10 seconds. Mean micro hardness for the sintered samples 1, 2, 3 & 4 were found to be 106.3 HV, 132.6 HV, and 115.7 HV & 89 HV respectively. From this result and above graph (Fig 5.13) we obverse that hardness increases with the addition of SiC but for sintered sample 4, it is lower than that of sintered sample 1 which is pure AZ91 Mg alloy. Micro hardness impressions in sintered sample 1, sintered sample 2, sintered sample 3 and sintered sample 4 are shown in Fig 5.13

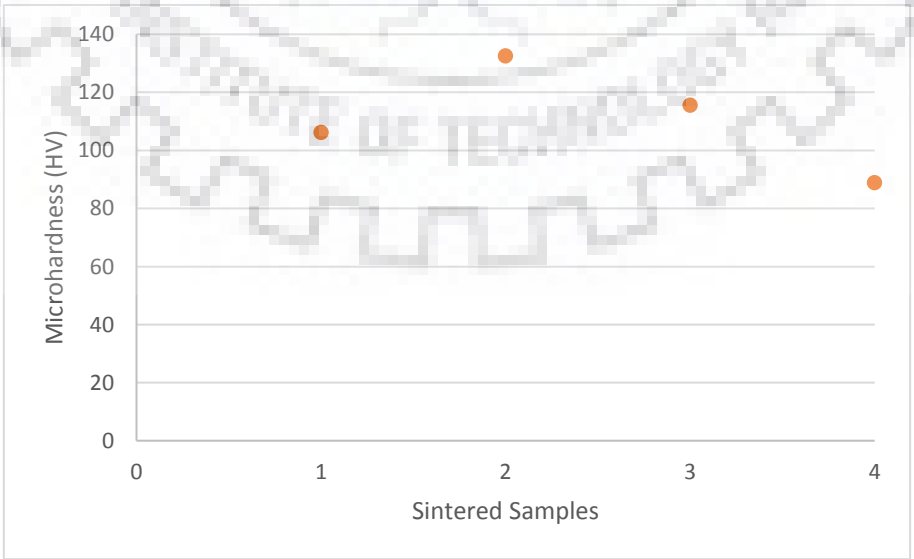


Figure 5.13. Micro hardness for sintered sample

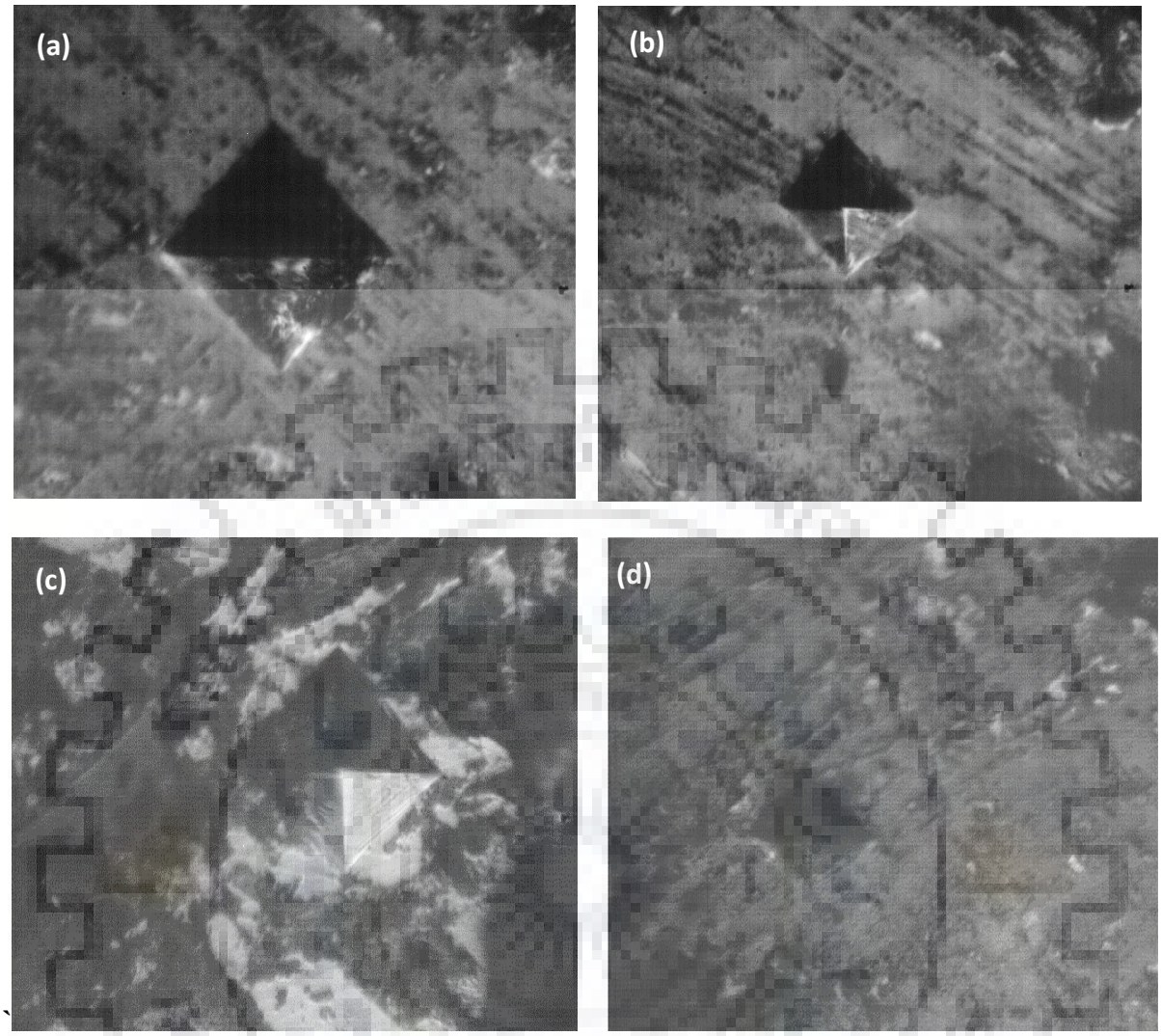


Figure 5.14. Micro hardness impression in (a) sintered sample 1, (b) sintered sample 2, (c) sintered sample 3, (d) sintered sample 4

Chapter 6

CONCLUSION

Increasing demand of high yield strength materials, Mg and its alloys fascinates researchers. As of now it finds major application in automobile and aerospace industry. There are several traditional modes to sinter the Mg, its alloys and composites. In this work, an eco-friendly way of sintering was explored for Mg alloy by means of microwave heating. The main conclusions drawn from this experimental study are the following:

i. Mg alloy easily reacts with the atmospheric air which leads to oxide layer formation and it reduces the binding strength of powder Mg alloy and strength of sintered solid bulk.
ii. Polyvinyl alcohol (PVA) was mixed in the mixture of AZ91 Mg alloy powder and SiC powder which causes porosity in microwave sintered samples.
iii. Domestic microwave does not give any idea of temperature at any particular time so it was difficult to analyse the temperature at which the compacted samples were sintered in microwave.
iv. Experimental density of the sintered samples are in the range of 85 to 90% of the theoretical density.
v. From optical micrograph, SEM images and EDS analysis, it is observed that on increasing the amount of SiC particles in AZ91 alloy more Mg_2Si and SiC phases are formed in the sintered samples. Formation of Mg–Mg_2Si results in strong bonding between Mg_2Si and the matrix interface.
vi. Micro hardness results shows that for sintered sample 2(5% weight % of SiC in AZ91 Mg alloy) the hardness is more than the rest of the sintered sample, So on increasing the amount of SiC particles in AZ91 Mg alloy hardness increases, but after a certain percentage it reduces as sintered sample 3(10% weight % of SiC in AZ91 Mg alloy) but it has more hardness than sintered sample 1(Pure AZ91 Mg alloy) but Sintered sample1 micro hardness is more than that of sintered sample 4(15% weight % of SiC in AZ91 Mg alloy).

Chapter 7

FUTURE WORK

- Try to compact the powder without using any binder may be attempted which is expected to provide better density and less porosity.
- Flash sintering may be attempted which will give the better idea of sintering time and its corresponding temperature.
- Fracture properties may be analysed for the sintered samples.

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MTech Dissertation Final

by Sakshi Jaiswal

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Chapter 1

INTRODUCTION

1.1 COMPOSITE

In the development of advanced material, the composites are most famous among researchers because composite materials are made up of two or more dissimilar materials, which may differ in forms, remain physically distinct, i.e. insoluble in each other and chemically do not react between them. The properties of the new material are different from that of its constituents. Composites have both advantages and limitations, which are discussed as below:

1.1.1 Benefits

- ✓ Composites own an aggregate of outstanding mechanical, chemical, structural, electrical and optical properties.
- ✓ They are light-weighted and better specific strength and specific modulus while compared to traditional materials.
- ✓ Composites can be moulded to any desired form with any favoured specification.
- ✓ They own good anti-chemical and anti-corrosion residences.
- ✓ Assembling and de-assembling of components is simple and quick.
- ✓ Efficient usage of material can be done. The fibres should be oriented in this sort of way to provide greatest strength and stiffness within the preferred direction.
- ✓ Problems like seepage and weathering are nearly minimal in composites.
- ✓ Composites give aesthetic designed look.

1.1.2 Constraints

- ➢ Composites are having low fire and flash points.
- ➢ Composites may additionally expand unwanted biological effects observed in polymers.
- ➢ Polymeric composites are unacceptable for high-temperature applications.
- ➢ Cost of composites continues to be more than many conventional substances.
- ➢ On prolonged left in vicinity of sunlight, the colours of composites typically fade out.

1.1.3 Classification of composites

Composites can be classified as fallows-

1. Agglomerated composites: For e.g. grinding wheel where abrasives are agglomerated in the wheel with the help of the binder.

2. Laminated composites: For e.g. plywood.
3. Reinforced composites.

Reinforced composites consist of a matrix and reinforcement. They can be further classified based on kind of matrix as well as reinforcement. Based on matrix materials used they are classified as:

1. Polymer matrix composites (PMCs)
2. Metal matrix composites (MMCs)
3. Ceramic matrix composites (CMCs)

1.2 METHODS FOR PRODUCTION OF THE MMCs

1.2.1 Stir Casting

It is referred as the vortex procedure. Reinforcement of particles is done for support of casting in molten material. Homogenous distribution of reinforcement is finished through a rotor rotating within the molten metal which develop a vortex and by means of inserting of a gas which carry the reinforcement into molten metal. The finely allotted produced slurry is original with the aid of conventional casting systems, viz. Sand casting, permanent mildew casting, pressure die-casting or squeeze casting. The typical stir casting process is shown in fig 1.1.

Factors that effects the stir casting process are as followings:

a. Gas entrapment
b. Slag inside the molten metal causes porosity and defects in the casted sample
c. Unfavourable chemical reactions at the interface of matrix/reinforcement
d. Due to low wettability of reinforcements with molten material matrix causes debris agglomeration (primarily in nano-reinforcements that might deliver about the formation of nano particle clusters that leads to non-uniform distribution of reinforcements)

Bypassing these problems would be the reason of deterioration of material made by this process. To make it successfully in laboratory and industrial levels, cautious, standardization of system parameters including temperature of molten metal, soften stirring time, stirring pace, melt retention temperature, option of matrix-reinforcements and many others have to be rightly made up our minds on.

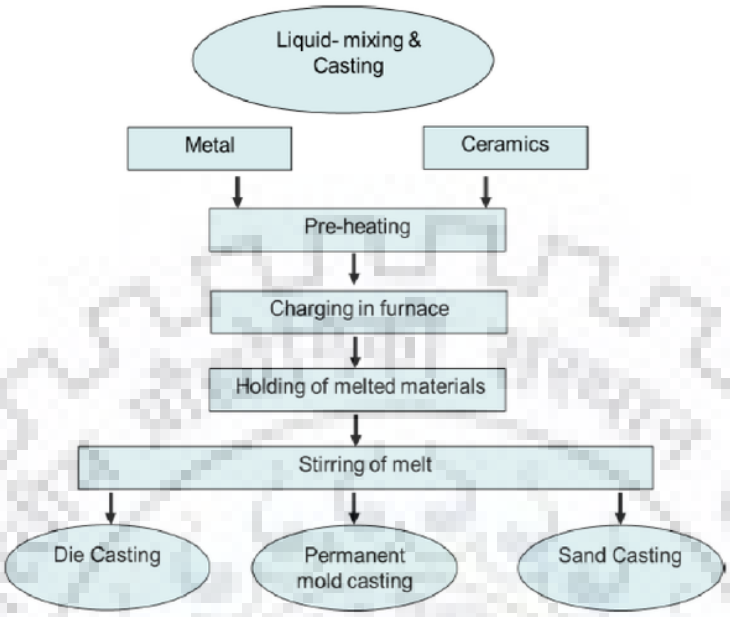


Figure 1.1. Schematic diagram showing stir casting process [3]

1.2.2 Centrifugal Casting

This process is a tremendously cheaper procedure wherein optimal reinforcement placements are performed by way of inducing a centrifugal force straight away all through casting. This ensures an intentional variant in volumetric fraction within the matrix material. A natural example to be used of centrifugal casting is in brake rotors, in which the rotor face is anticipated to be of immoderate put on resistance while in comparison with the hub. Problems due to machining of casted product by general casting are faced due to the excessive hardness, is removed by centrifugal casting method.

1.2.3 Squeeze Casting/Infiltration Process

This procedure includes the invasion of a liquid compound into a ceramic fiber/molecule preform pursued by cementing. The presentation of liquid metal into a preform would be finished both by means of pressure less infiltration and via invasion underneath pressure. In pressure less penetration, ceramic fiber packs are first set within the die. The liquid metal is then poured on to it and permitted to harden. The fixed composites are then sizzling squeezed to achieve 100 % thickness. The underlying invasion happens with no use of outside pressure and the wettability of strands guarantees effective penetration. The weight invasion procedure can be utilized in two distinct ways, in particular through gas penetration and weight

penetration. In gas penetration, vacuum or latent gas air is used to deliver invasion. Focal points of this technique incorporate increment in the wettability due to the expanded surface action of fortification in vacuum condition, end of gas entanglement or porosity and accomplishing close net formed segments. Its fundamental inconveniences are isolation of stages and response between framework/fiber because of the moderate idea of the process. The crush penetration process includes the invasion of liquid metal into an artistic preform utilizing water driven pressure. By this strategy, the downsides of stage isolation and grid/interface response experienced in gas invasion can be killed because of the use of pressure drive.

1.2.4 Spark Plasma Sintering (SPS)

SPS is a comparably new sintering procedure and it permits compaction of powdered metals and ceramics at low temperature in short holding time. SPS is fundamentally the same as traditional hot squeezing, precursors are loaded in die where an uniaxial pressure is applied during sintering. However, a pulsed direct current is passed through the electrically conducting pressure die. Sometimes, pulsed direct current passes through the sample in appropriate cases. This infers the die, likewise acts, as a warming source, and heating takes place from both sides(inside and outside) on the sample. The schematic illustration of this procedure is appeared in Fig 1.2.

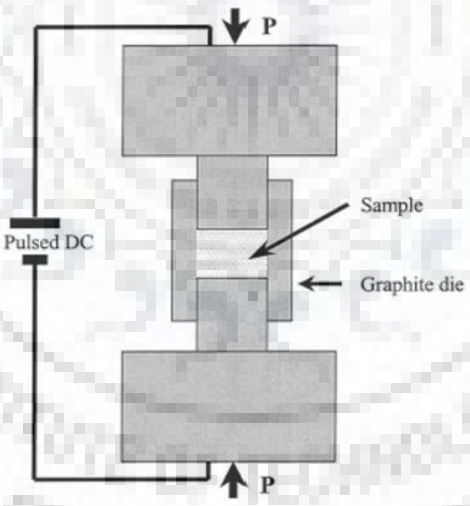


Figure 1.2. Schematic drawing of SPS apparatus [4]

The remarkable highlights of the procedure are the possibilities of utilizing quick warming rates and short holding time to acquire dense samples. Three factors that causes to the quick densification procedure can be noted below:

(i) Application of a mechanical pressure

(ii) Quick heating rates,

(iii)Use of pulsed direct current inferring that the sample are likewise presented to an electrical discipline.

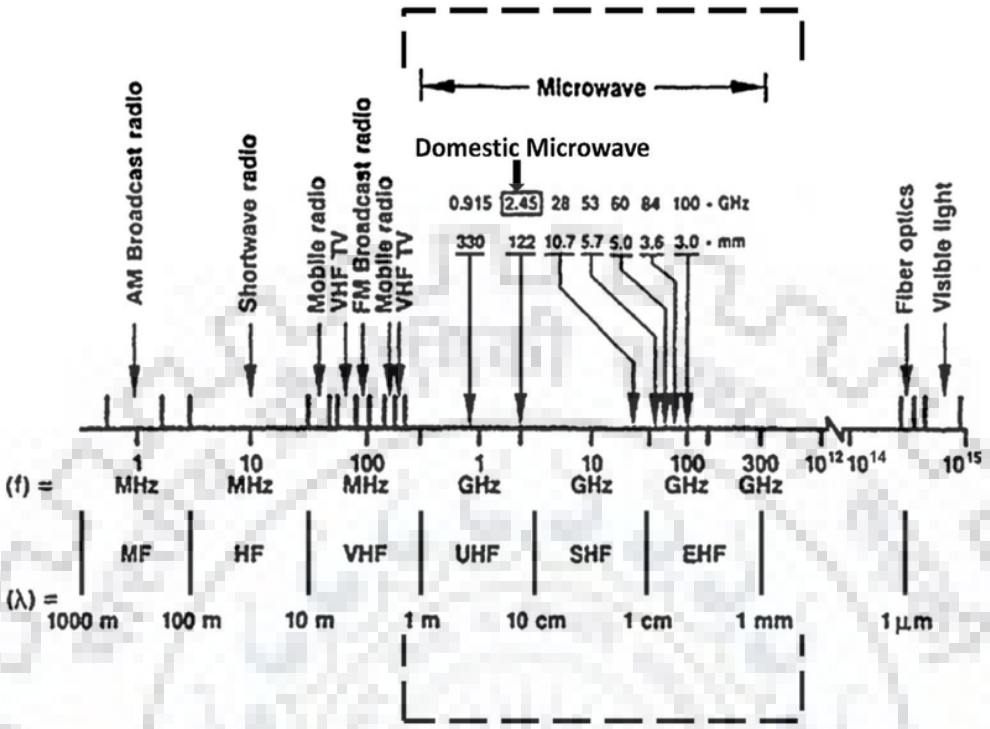
It is probably the most section stated that use of mechanical stress is regularly, worthwhile in getting rid of pores from compact samples and embellishing diffusion. The heat transfer to the compact sample from the die is tremendously efficient in this approach, due to the fact that the die itself (heated from the pulsed direct current) acts as a heating aspect. Accordingly, pulses and then plasma between the powder particles (pattern) develop spark discharges. Hence, this is the reason of calling this approach as SPS.

1.2.5 Microwave Sintering

M Gupta et al. (2007 to 2009) [9] used microwave radiation for volumetric heating of samples, which is kept inside the microwave oven. This process changes the electromagnetic energy in to heat energy. In this process, microwave susceptors are used in microwave to aid the decrease of thermal gradient during sintering. The compacted samples or billets of metal/ reinforced composites are kept in the inside the oven in crucible. There are two crucible is used. In inner crucible, compacted sample is placed and the SiC powder is kept between the gap of inner and outer crucibles, because SiC has characteristic to absorb the microwave radiation promptly and heats up rapidly. This offers incredible warmth that can thus remotely warms the compacted billets. What's more, the compacted billets themselves assimilate microwave and get warmed from inside of. Thereby, uniform distribution of heat is experienced along the whole part of a specimen, thus lowering any core-to-periphery thermal variant. On account that of this purpose, excessive sintering temperatures (~620–650 °C) can be created inside of a brief time frame (12–14 min), which can be close to near the softening purposes of Al and Mg, via the goodness of which improved wettability and reduced porosity may also be complete.

1.3 MICROWAVE MATERIALS PROCESSING

The microwaves are basically electromagnetic radiation and their radiation frequency range lies someplace within the range of 1 and 300 GHz and these microwave frequencies with quite a lot of wavelengths are utilized for a wide assortment of makes use of that are appeared in Fig. 1.3



.Figure 1.3. Electromagnetic radiations spectrum for frequency and wavelength [2]

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

Various methods were used to prepare MMCs with the objective to get the incremental improvements in their mechanical properties and their comparison with other methods of manufacturing the same. Effect of reinforced materials with their varying percentage was studied. The change in reinforcement not just affected properties but it also changed the

microstructure and grain growth of the composites. For the manufacturing of MMCs, the conventional processes such as die-casting, stir casting, friction stir process and common methods to prepare MMC were studied. After summing up, it was concluded that all these properties could be enhanced by fabricating the MMCs through MHH. Metal casting by utilising microwave radiation energy is recent ongoing projects for MWs. The procedure was created and metal cast were accounted for the procedure and cast portrayal are yet to be accounted for.

As of late, the use of microwaves in different applications has expanded numerous folds. The different handling areas where this innovation has been connected effectively is appeared in Fig. 1.4, which incorporates correspondence frameworks, sustenance handling, wood drying, improved chemical responses, vulcanization of elastic, preparing of earthenware production and metallic materials, making of different kind of steels, materials joining and its welding, squander nursing, and recuperation of substitute wellsprings of vitality.

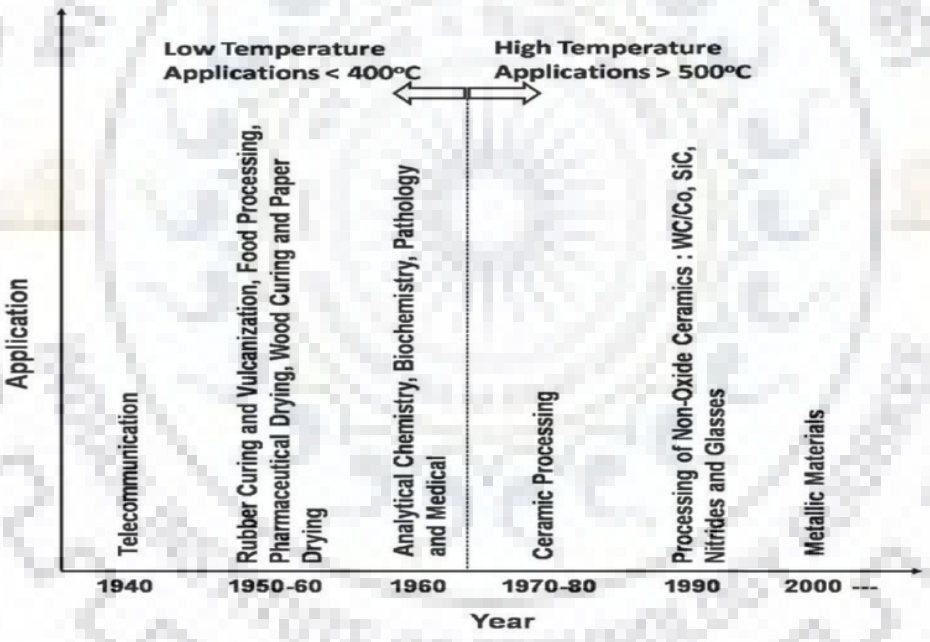


Figure 1.4 Recorded improvements appearing of microwaves in different fields [2]

Presently, it is notable that metal as powder can retain microwaves and can be prepared effectively. Further to investigate the benefits of microwaves, microwave hybrid heating was created which drove numerous scientists to process different sorts of materials for an assortment of uses in the field of material science. The different advancements in the field of

preparing of metals are appeared in Fig. 1.5, which demonstrates year astute ordered advancements utilizing microwaves. Over the most recent 65 years, microwave vitality has been used in an assortment of uses that can be categorized as low temperature (<500 ºC) applications in amalgamation and drying (1950–1970), moderate temperature (between 500–1000 ºC) uses as in sintering applications (1970–1999), and high temperature (>1000 ºC) utilizations in the cutting edge material preparing (1999–onwards). It is obvious from the advancements that, for the most part, metal tests are assuming a significant job in the handling of metal-based materials. The hypothetical and exploratory research works that were accounted for in the zone of microwave preparing of metal-based materials can be separated into four noteworthy gatherings i.e., powder metal, mass powder metal framework, mass metal, and sheet metal based on accessible writing. The hypothetical analyses and test examinations were announced for metal powders as unadulterated metal powder compacts, metal amalgam compacts and metal framework composites. The mass powder metal frameworks were accounted for applications, for example, brazing, joining, and covering/cladding. The mass metal and sheet metal preparing were accounted for by a couple of scientists as melting, casting, drilling (on metal/non-metal utilizing a metallic instrument), and warmth treatment. The hypothetical models and exploratory methodologies were likewise announced for examining compelling utilization of metallic materials amid microwave handling of non-metallic materials in other applications such as nourishment preparing, concoction union, and so on.

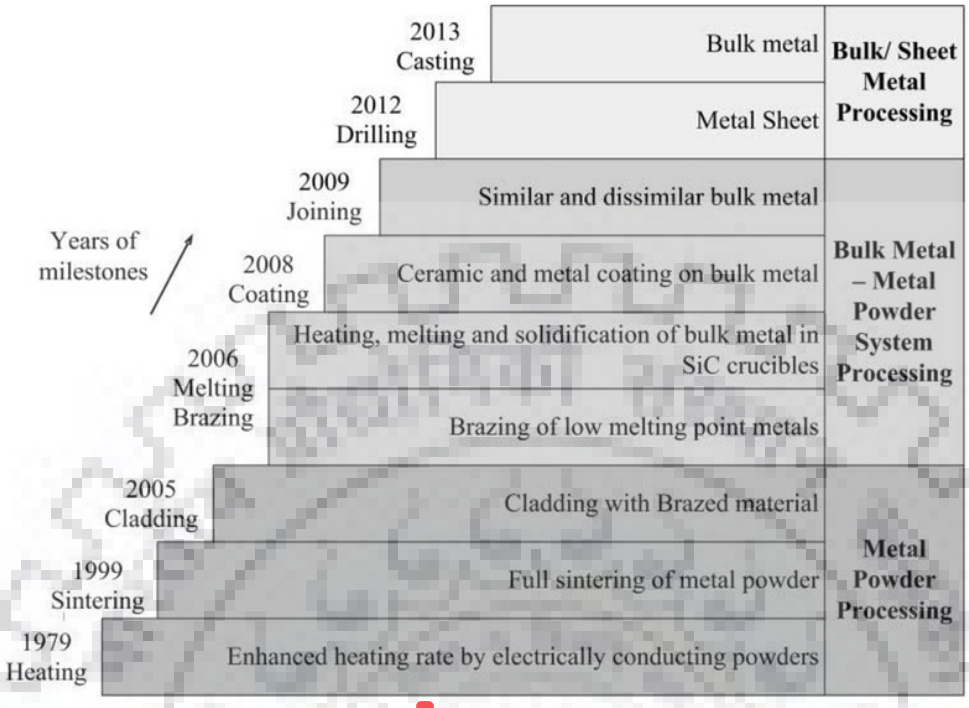


Figure 1.5 Sequential improvements in the field of microwave processing of metals [1]

1.4 MICROWAVE MATERIALS PROCESSING

Microwave heating of materials is done effectively through microwave radiation, mainly depends upon the physical properties of that material. Because the mechanical properties of materials helps in interaction of microwave radiation in that materials. Generally, materials are classified in three groups, which is shown in Fig. 1.6. Microwave interactions does not take place in transparent materials and crosses them without any heat transfer. Microwave radiations reflect back on conductors that lead to plasma formation and by this surfacial heating of conductors take place. Absorbers readily absorb the microwave, which retains and changes over these radiations into heat.

Material properties for microwave absorption are the complex. The relative permittivity of space, medium and loss tangent shown by Eqs. (1) and (2) [1, 3].

$$\varepsilon = \varepsilon_0 (\varepsilon' - j\varepsilon'') = \varepsilon_0 \varepsilon' (1 - j \tan \delta) \quad (1)$$

$$\tan \delta = \frac{\varepsilon''}{\varepsilon'} \quad (2)$$

Where

ε=electric constant in medium

ε_0=electrical permittivity in space

ε'=electrical permittivity in medium

ε''=dielectric loss factor

j=electrical polarizability

δ=dielectric loss tangent

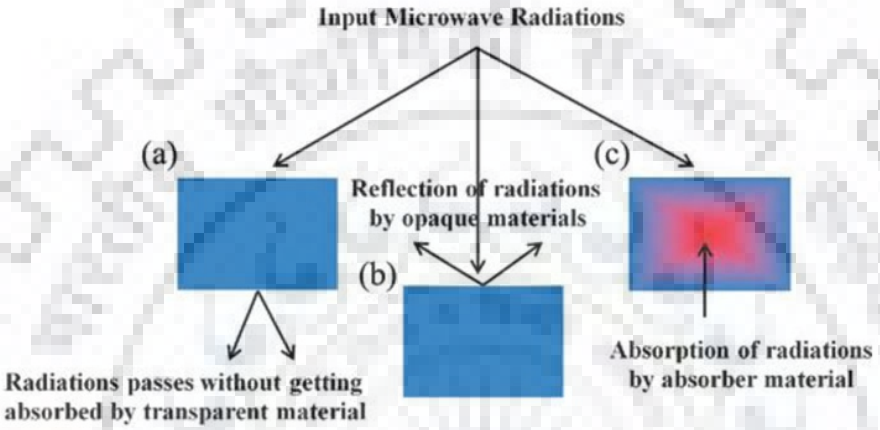


Figure 1.6. Microwaves–materials interactions [1]

The ability of a material to transfer microwave energy into heat is defined by dielectric loss factor, and for measuring, the polarizability of material is dielectric constant. The increase in temperature because of absorption of microwaves is ruled by means of Eq. 3.

$$\frac{\Delta T}{\Delta t} = \frac{2\pi f \varepsilon_0 \varepsilon'' |E|^2}{\rho C_P} \quad (3)$$

The dielectric associations of materials are additionally portrayed by two parameters: power dissipation (P) and depth of penetration (D) of microwaves. The consistency of heating profile relies upon these variables.

The power dissipation is communicated by Eq. (4).

$$P = \frac{1}{2}\omega . \varepsilon_0 . \varepsilon'' . E^2 . V . e^{-2\alpha z} \quad (4)$$

Where, E = electric field through the surface,

V = volume,

$\omega = 2\pi f$ frequency,

z = distance into the specimen and

α = attenuation constant.

The consistency of warming inside the material relies on the depth of penetration on which the episode power is decreased to half of introductory esteem. Depth of penetration is communicated by Eq. (5)

$$D = \frac{3\pi_0}{8.686\pi \tan\delta\left(\frac{\varepsilon'}{\varepsilon_0}\right)^{\frac{1}{2}}} \quad (5)$$

The correlation of the penetration depth in terms of frequency is expressed by Eq. (6)

$$D = \frac{C}{2\pi f\sqrt{2\varepsilon'}\left(\sqrt{1+\tan^2\delta}-1\right)^{\frac{1}{2}}} \quad (6)$$

The higher estimations of t and electrical permittivity decrease the depth of penetration at a specific wavelength of microwave radiation. Surficial warming is due to high frequency and high estimations of dielectric properties so for the volumetric warming moderate dielectric property is required.

At room microwaves radiation of 2.45GHz reflect for mass metallic compounds because of smaller estimations of skin profundity, which results in impression of radiations. The skin depth of materials in connection to microwave handling is characterized as the profundity into the materials from the surface at which the estimation of episode microwave control drops to 1=e (36.8%) times the surface esteem. A significant parameter gives a comprehension of the upper thickness breaking point of materials to be handled through microwave radiation in a proficient way. In any case, it is conceivable to expand the skin depth (δ) of specific material at a specific recurrence by changing the temperature-subordinate parameters, i.e., resistivity and magnetic permeability as shown in Eq. (7).

$$\delta = \sqrt{\frac{\rho}{\pi f \mu_r \mu_0}} \quad (7)$$

Where

δ=skin depth (mm)

ρ=resistivity (μΩ-cm)

f=frequency of microwaves (GHz)

μ=magnetic permeability (H=m)

μ_0=absolute permeability (H=m)

μ_r=relative permeability

Microwave radiations for better coupling with various metallic materials with the idea of hybrid heating was proposed by analysts which is talked about in the next segment.

1.5 HYBRID HEATING BY MICROWAVE

The effort, which is done for microwave samples (materials) preparations, was principally completed by utilizing local heating by microwave with a recurrence of 2.45 GHz. The immediate warming of samples by microwaves radiations may experience central issue like thermal instabilities that may create thermal runway into the prepared materials. The traditional method of warming prompts poor microstructures of surfaces, while microwave-warming mode causes poor microstructures of centres due to distinction in temperature inclinations in various territories. These slopes can prompt extreme temperature no consistencies and may cause cracks inside the prepared materials. To conquer the issue of thermal instabilities, research specialists have created hybrid heating system because it involve heat exchange marvel which includes both ordinary and heating through microwave radiation. Materials, which are having higher dielectric misfortunes at room temperatures are utilized as an infrared warming source. These sources are names as subsector, which retain the energy of microwave positively, accomplish temperature in high amount, and then use to heat the metallic material, which is in powder form by means of traditional ways. This case is the best example of mixed heat transfer, utilize for material heating which are having less dielectric misfortunes like composite materials. Fig. 1.7 is showing the two way heating of powder samples, which lead to reduce the time of heating and give uniform heating.

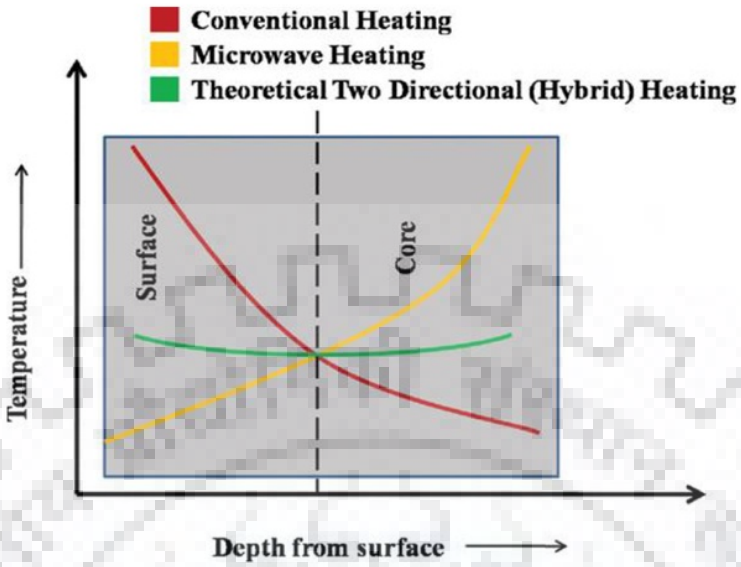


Figure 1.7 Temperature and distance profile for traditional, microwave, and two directional hybrid heating [2]

Preceding the year 1999, the believe was that MMC reflect microwave radiations and the electron mist are created sharp corners because of constrained entrance of microwave radiations in MMC and that create plasma development which leads to sparks generation. The initial effort of researchers revealed that connection of microwave energy with metallic powders for upgrade of warming rate by including a couple of percent amount of metals powders which are having electrically conducting property was amid in preparing of refractory ceramics. Very few researchers through sintering, casting, brazing of chosen metallic materials until 2008, did processing of metallic powder by means of microwave energy. Intensive inquiries about were brought out for warming and sintering of metallic powders with compelling utilization of microwaves energy resulting in improved metallurgical and mechanical properties in compacted samples.

1.6 ADVANTAGES OF MICROWAVE MATERIAL PROCESSING

The points of interest offered by microwave heating phenomena are numerous over traditional heating because of the following reasons:

- The direct ingestion of microwaves inside materials permits volumetric heating which produces upgraded diffusion rates, diminished power utilization, and lower process time. The immediate exchange of energy wipes out losses, which is related to heating of furnace,

furnace walls etc. These give higher rates of heat movement in contrast with traditional techniques and higher temperatures can be accomplished in shorter time as shown in fig 1.8.

- It was analysed that the defects present in the heated samples prepared by using microwave energy was less because of higher heating and diffusion rates in the process. Enhancement of different parameters gives improved microstructure for example temperature rate of heating and temperature angles. The procedure of quick warming in microwave preparing gives a few advantages, for example, high sintered thickness and better microstructures contrasted with moderate heating traditional procedures. These better microstructural advancements, better normal grain measure, higher densification parameters, and lower porosity imperfection result in improved mechanical properties contrasted with traditional processing strategies [1-3]. The prerequisites for microwave sintering incorporate high temperature, better warming rates, uniform temperature slopes, and uniform warm circulation inside the sample. Sintering through ordinary routes does not give uniform heating and temperature slopes, which powers to keep the conservative at high temperatures and for higher handling times. Its moderate warming rates combined with its higher handling time has prompted the distortion and coarsening of compacted samples.

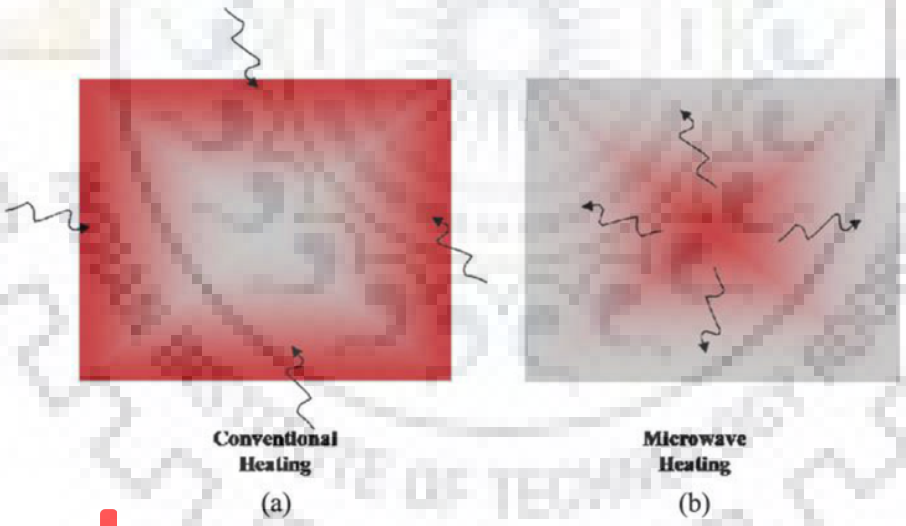


Figure 1.8 Heat generation pphenomenon in (a) traditional heating (b) microwave heating [2]

- The volumetric heating phenomenon promote selective and uniform heating of materials that causes reduce time in processing temperature , lowers down the heat affected zones and ecofriendly as comparison to other traditional methods.

Chapter 2

LITERATURE REVIEW

2.1. WORK BRINGING OUT THE DIFFERENT SIZE OF THE REINFORCEMENT USED

There are various experiments have been performed to find out the ultimate results after creation of metal/matrix composites. Some of result of these kind of experiments are discussed below:

Vaidya et al (1996) [4] showed how the variation of the SiC particle reinforced with Mg alloy AZ91 based composite by volume fraction, effects the High cycle fatigue properties of it. AZ91D Mg alloy metal matrix composites processed by two methods one is squeeze casting and other is extrusion and reinforced by either 15 μm or 52 μm size SiC particles, mixed at both 20% and 25% volume fraction in the composite. SiCp increased the strength and modulus of AZ9lD, with finer reinforcement which provides more strength to the metal matrix composite. Reinforcement with 20 or 25 volume fraction of 15 μm SiCp gives higher fatigue performance with respect to monolithic AZ91D.

W.L.E. Wong et al (2006) [6] reported that use of hybrid length of the reinforcement improves the thermal stability and hardness of the composite when compared the singular reinforced material. SiC particles were reinforced in pure magnesium powder which was mixed in a RETSCH PM-400 mechanical alloying machine at a speed of 200 rpm for 30 minutes and then compacted using uniaxial compaction machine and then sintering process was formed by using microwave. Sintering time was 25 minutes for this experiment. Sintered Mg composite reinforced with SiC particles of two different amount shown better hardness and thermal stability in comparison to monolithic reinforced composites.

K.K. Deng et al (2012) [9] reported about the various results regarding the size of particle on microstructure and mechanical characteristic of SiC/AZ91 magnesium alloy composite. Authors concluded that uniformly distribution of particles depends on the particle size. The larger size of SiC are more uniformly distributed in magnesium matrix in the same condition of volume fraction. Particle distribution and grain refinement is important factors that influence the mechanical properties of MMC, Submicron SiC/AZ91 reinforced MMC exhibits good mechanical characteristic at 2% volume fraction, but at 5% and 10% volume fraction of micron

SiC/AZ91 composite shows better strength. Also related optical micrographs of the experiment has been shown in Fig 2.1.

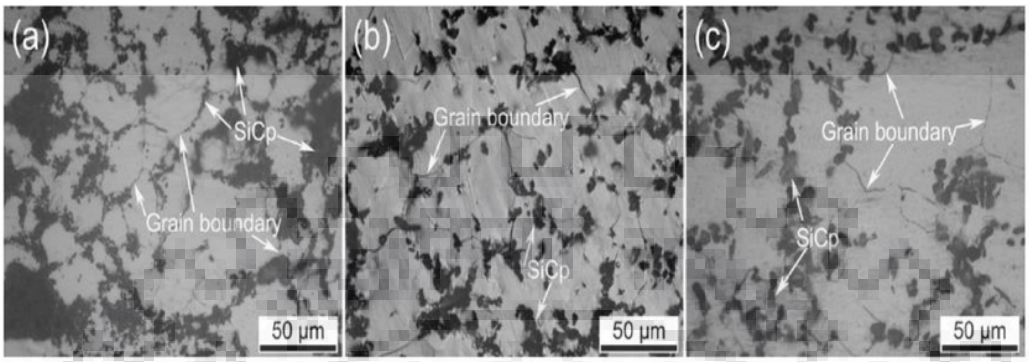


Figure 2.1 Optical micrographs of casted SiCp/AZ91 Mg metal matrix composites in 10 vol.% of SiC particle size of (a) 0.2 µm, (b) 5 µm and (c) 10 µm, respectively [9]

2.2 INTERACTION OF METALS AND MICROWAVES

Mishra and Sharma (2016) [3] represents the wonderful explanation regarding heat interaction between different materials and microwave which is very helpful to understand the exact heat interaction conditions inside the microwave which pictorial illustration is presented in fig 2.2. Mechanism that cause interaction of microwave energy in various materials like metals, non-metals and composites talked about utilizing reasonable illustrations.

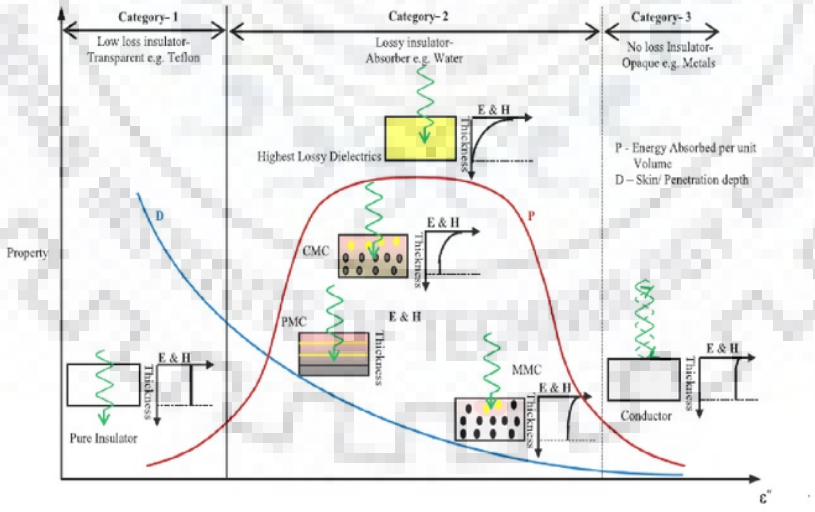


Figure 2.2. Microwave interaction with different types of materials [3]

It has been seen that while microwave energy absorption three losses are played significant role in the heating of metal based materials i.e. magnetic field based loss effects, dipolar loss and conduction loss that led to the electric field effects in microwave heating of non-metals. Difficulties in handling of composites have been distinguished from the accessible literature.

2.3 CHARACTERISTIC OF AZ91 MG ALLOY

Mondet (2016) [9] investigated the influence of $Al_{11}Mg_{12}$ precipitation by sintering temperature and grain size of sintered samples and showed how SPS technique is better than other conventional sintering processes. Fine intergranular $Al_{12}Mg_{17}$ precipitates found in sintered as shown in Fig 2.3. On high sintering temperatures, the populations of precipitate gradually dissolve until their complete dissolution above 380 ºC. A very few amount of $Al_{12}Mg_{17}$ precipitate found in between 380 ºC and 500 ºC. The impact of $MnAl_4$ precipitates were negligible impacted and may be examined that their size and fraction remained unreacted in the sintering process. From the fig 2.4, it was clearly visible that on increasing sintering temperature hardness decreases. The paper also pointed that in SPS process, the lack of ductility, under tensile testing, remains an issue for AZ91 Mg alloys processed by SPS.

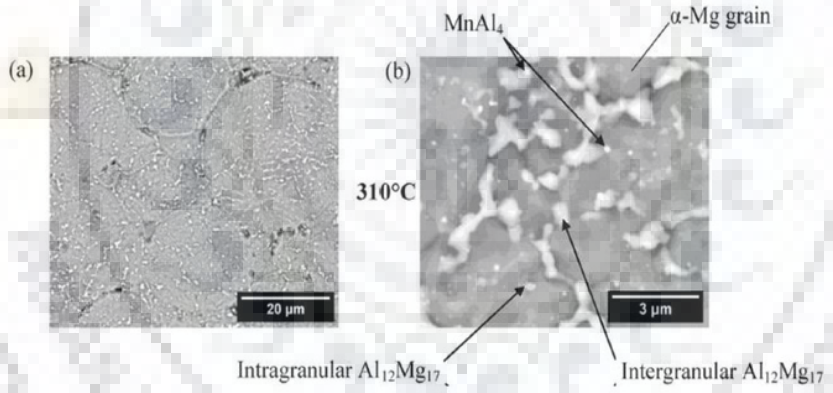


Figure 2.3. SEM images for AZ91 Mg alloy at 310 ºC [9]

Trojanová (2009) [21] did study on AZ91 Mg alloy reinforced with SiC particles and then additional amount of Si particles were added. It resulted the formation of an ‘‘in situ” composite (Mg–Mg_2Si) which gave a strong bonding between Mg_2Si and the matrix interface as it is shown in SEM images (Fig. 2.5). The effect of reinforcing of SiC and Mg_2Si particles found to be decreased with increasing in temperature. Mechanical properties were also analysed in this study at different temperatures. A ductility improvement was found at 200 ºC and temperatures over 200 ºC.

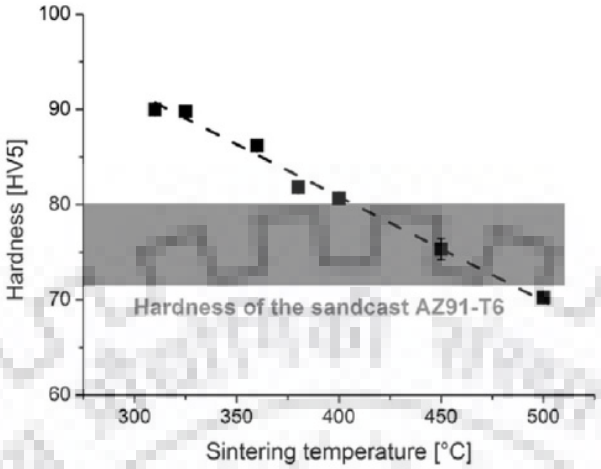


Figure 2.4 Vickers hardness AZ91 mg alloys at different sintering temperature [9]

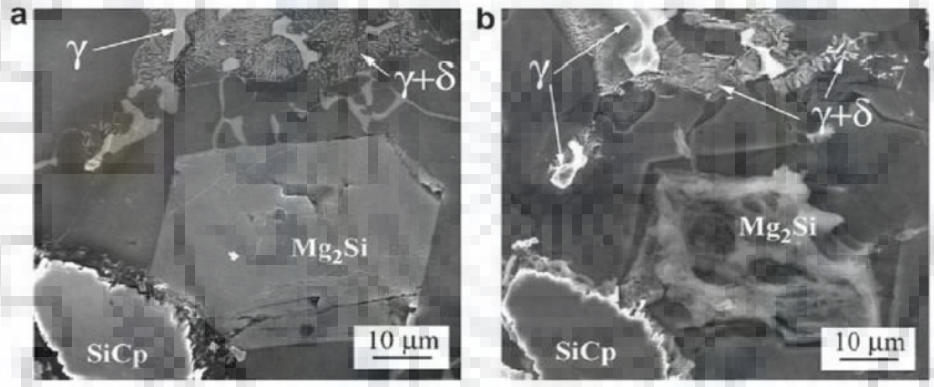


Figure 2.5 SEM images shows SiC particle with primary Mg_2Si coarse dendritic crystal after using glycol etching (a) and deep etching (b) [21]

2.5 MICROWAVE VERSUS CONVENTIONAL SINTERING

Oghbaei and Mirzaee (2010) [22] gave a brief of central parts of microwave hybrid and hybrid sintering and after that its favourable circumstances against the traditional techniques. Some microwave sintering applications are likewise referenced which so far have displayed favourable circumstances of this novel process.

Madhan (2019) [24] this repot comprises the production of Al_2O_3-SiC ceramic composites by using conventional and microwave sintering process. The sample was prepared by mixing of x

wt. % SiC, (x=5, 10, 15 & 20) in Al_2O_3 powder and milled for 180min. Then the samples were compacted at 60MPa for 30sec and sintered by both methods. After sintering hardness, densification, grain size, phase variation and micro structure were examined &comparison were made for the methods.

Wong and Gupta (2007) [23] used the concept of hybrid heating to develop magnesium composites with different amount of Cu particles mixed with it. The process was done by using frequency 2.45 GHz and power 900 W for microwave generation. The concept of using susceptor also applied for hybrid heating of the compacted sample In this it also revealed that microwave processing has capability to make denser product than other conventional ones and gives less porosity in the sintered product. Cu particles blended with the matrix gives more hardness, increase yield strength, elastic modulus, ultimate tensile strength of the metal matrix composites. It was also observed that the tensile properties also increased by processing through microwave energy route

2.6 LITERATURE GAPS AND OPPORTUNITIES

Composites are become the lead demand in various applications such as automobile, aerospace, defence and medical science. They have specific properties, which required prominent processing techniques so that final product can fulfil the desired customer's needs. However, there are various existing processing techniques are present still material efficient, energy and time efficient process are required to develop. Consequently, microwave material processing is a revolutionary process for the current scenario as it gives the better mechanical, chemical, physical properties than other conventional processes. The operation procedure is not complicated, requires less floor area. From the literature survey, microwave sintering process is suitable for development of new composites with adequate attributes.

It is also observed that MMC can be easily processed by using susceptors in MW i.e. hybrid heating phenomenon. As Mg based alloy are very famous among researchers due to its valuable properties. There is much work has been performed or Mg and Al based alloys in conventional as well non-conventional routes of processing. However, AZ91 Mg alloy reinforced with SiC particles with different weight percentage has not been attempted through microwave sintering. Hence, in this work, development of AZ91/SiC composites using microwave sintering has been proposed and successfully developed with some beneficial results.

2.7 OBJECTIVES AND SCOPE

The objectives of this work are as follows:

I. Development of AZ91/SiC composites through microwave sintering

Commercially available magnesium-aluminium-zinc alloy (AZ91) of 80 μm particle size have reinforced by SiC powder of 80 μm size. This composite have been developed in a microwave oven at 2.45 GHz frequency and 900 W power consumption.

II. Density measurement of AZ91/SiC composite

Theoretical density and experimental density have been measured through Archimedes principle of density measurement.

III. Characterization of developed AZ91/SiC composite

Optical, SEM/FESEM & EDS measurements have been performed and results have been reported. Micro hardness test has been performed to measure the hardness of microwave sintered AZ91/SiC composite.

IV. Results of above are then evaluated as well as easy of process is validated.

Chapter 3

MATERIAL SELECTION AND ITS APPLICATION

Material selection for any application is the most important factor as it decides the performance and efficiency of the final product where it used. According to applications and their performance, material should be choose and processed by suitable manufacturing process. In this work, Mg based alloy has been used for development of MMC by adding SiC as reinforcement. The brief explanation why these materials have been selected is given in next paragraph.

3.1 BASE MATERIAL

Magnesium comes under the lightest structural metal. Magnesium alloys are 33% lighter than aluminium, 61% lighter than titanium and 77% lighter than stainless steel making them promising competitors as trade material for these metals. As far as accessibility, magnesium is the sixth richest component in the world's hull containing 2% by mass and third most dissolved mineral in seawater with an accessibility of 1.1 kg/m^3. Its plenitude in planet earth is sixth most accessible in earth outside, third most accessible in seas, and fourth most inexhaustible cation in the human body. Moreover, simplicity of reusing, predominant explicit mechanical properties, high damping capacity, and critical electromagnetic protecting ability are a portion of the characteristics that warrant the across the board utilization of magnesium based materials in both building and biomedical applications. Different points of interest related with magnesium based alloys include:

i. Requirement of energy is low in processes where solidification requires
ii. It can be easily processed by using traditional solidification methods.
iii. Better machinability gives higher tool life generally 5-10 times higher than hard materials.
iv. Ability to be processed using conventional plastic deformation processes at speeds matching with that of conventional structural materials.

Above points, present the interesting facts about Mg based alloys that to be incorporated in designing applications with insignificant infrastructural ventures and varieties, basic prerequisite that influences basic leadership of industrialists to deliver better-required materials. Moreover, decreasing in cost of magnesium and the ability to decrease carbon

impression are factors that are setting off the substitution of aluminium based alloys step by step and continuously over a decade ago. The cost related factors guarantees steadiness of market costs of the item without stressing customer's pocket. Likewise, worldwide are moving to reduce the carbon dioxide outflows by 2 billion tons to maintain the temperature ascend to inside 2°C from the preindustrial levels as proposed in Paris understanding will fuel the expanding utilization of Mg based alloys in not so distant future. It is broadly foreseen that magnesium will supplant aluminium in 2020s in a similar manner as how aluminium supplanted steels in 1980s.

3.2 TYPE OF REINFORCEMENT

Magnesium based composites generally use ceramic reinforcements. They range from carbides, borides, or oxides. From all the ceramic reinforcements, the most broadly explored and utilized reinforcing ceramics with pure magnesium and commercial grade magnesium alloys is the SiC particulates. SiC particles are mostly chemically stable in many molten Mg alloys and SiC particles has generally better wettability with Mg in contrast with the other ceramic type of reinforcements [1, 2].

Chapter 4

EXPERIMENTAL WORK

4.1 MIXING

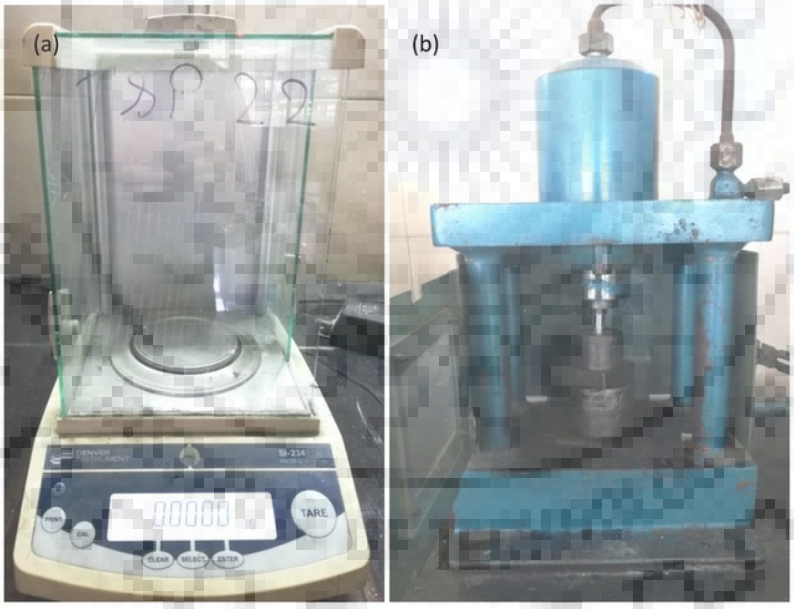
AZ91 Mg alloy (80micron particle size) was uniformly mixed with SiC (80 micron particle size) in 5%, 10% &15% by weight in V blender machine and mixing time was taken as 4 hours for ensuring the uniform mixing of powders.

Figure 4.1. V- blender machine

4.2 COMPACTION

Before compaction,the measured powder was mixed with Polyvinyl alcohol (PVA) as PVA is binder for the billet preparation.So firstly PVA is melted on hot magnetic (Fig 4.2) plate and mix(5% by weight for each sample), properly so that no lumps was taken place and then weighted each sample to make h=5mm,d=13mm, pallet in press of maximum capacity 25T at a 9T pressure.

Figure 4.2. Hot magnetic plate machine



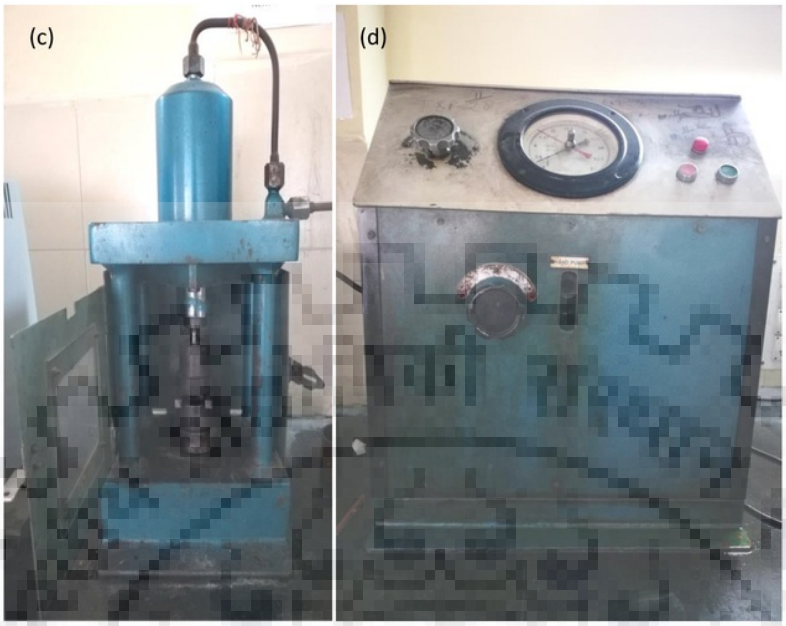


Figure 4.3. (a) Weighing machine, (b) Hydraulic jack during compaction of powdered sample, (c) Hydraulic jack during removal of compacted sample and (d) Input segment of Hydaulic jack machine

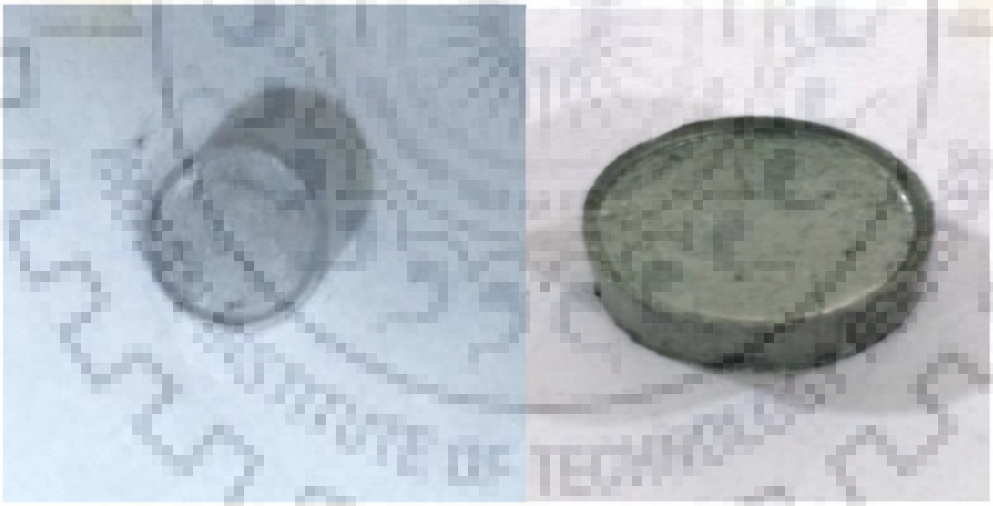


Figure 4.4. Images of compacted samples

4.3 PREPARATION OF MICROWAVE SINTERING SETUP

Peapered sample or billet was kept inside the Alumina crucible(99.7% of alumina from Ants ceramics Mumbai). As shown in the Fig 4.5, there are two crucible and SiC is kept between them to as a Susceptor for the sample. Alumina glass wool is used as an insulator or the process. After this, the whole setup was kept inside the microwave and the base material is refractory brick for it.

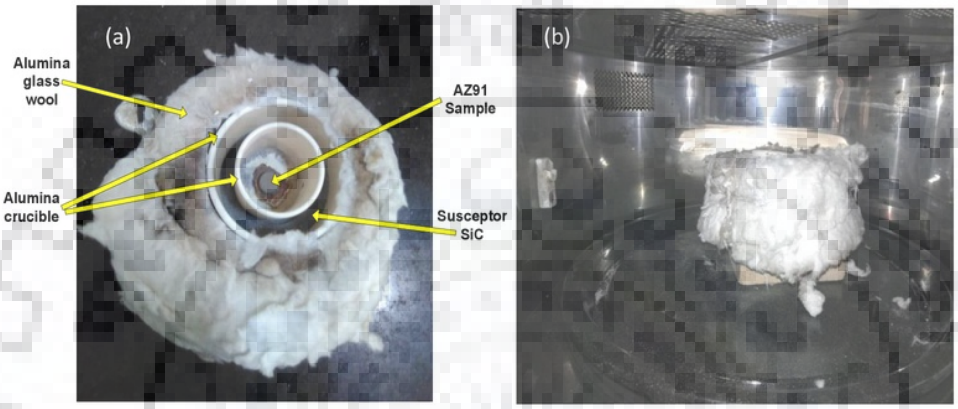


Figure 4.5. (a) Microwave sintering setup, (b) Setup inside the microwave

4.4 MICROWAVE SINTERING

Sintering is done in microwave at 900W for various time periods on domestic microwave which is shown in Fig 4.6.

Figure 4.6. Domestic Microwave applicator

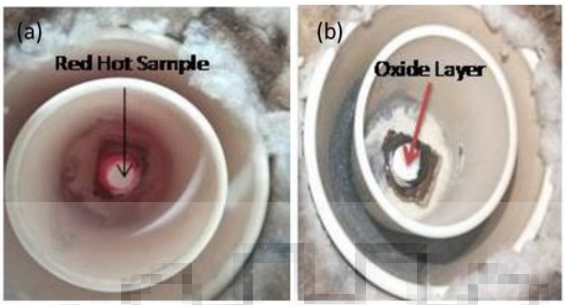


Figure 4.7. (a) Showing the red hot sample after microwave sintering, (b) Oxide layer formation on sintered sample

Table 1. Table for comparision of microwave sintering at various temparature

Diameter	Height (before sintering)	Sintering time (min)	Sintered sample
1.3 cm	0.5 cm	13	
1.3 cm	0.5 cm	10	
1.3 cm	0.5 cm	8	
1.3 cm	0.5 cm	5	

Sintered samples at sintering time 8 min is used for futher analysis because at 13min and 10 min compacted samples were burned which was not desired for futher analysis and at 5 min, sintering was not done completely as shown in table 1.

The sintered samples are shown in table 2 with their short names which has been used in results and discussions for convinient purpose.

Table 2. Showing all the 4 different sintered sample's images and their short names

Samples	Image of Sintered Sample	Short name
Pure AZ91 Mg Alloy		Sintered sample 1
5% SiC + AZ91 Mg Alloy		Sintered sample 2
10% SiC + AZ91 Mg Alloy		Sintered sample 3
15% SiC + AZ91 Mg Alloy		Sintered sample 4

4.5 FAILED EXPERIMENTS

Issues faced

- Availability of AZ91 Mg alloy in form of fine sized powder was challenging because it is a costly material and has been made for specialised order in larger quantity (as compared to our requirement)
- Compaction of the powder sample was really a tough job because AZ91 Mg alloy easily react with atmospheric air and form oxides which reduces its binding capability. Mg alloy has good binding capability at 450 MPa pressure. Usually Mg alloy does not

require any binder but due to oxide formation, we used binder PVA for compaction and after mixing PVA in powder sample, compaction was done before it react to the environmental air.

- During compaction of powder, punch and die got distorted due to high pressure applied on it repeatedly. Precise machining was done on the punch and die to make it perfectly align again.
- We use domestic microwave, which does not show any temperature at any time of the heating process. That's why it was difficult to find out perfect time and temperature for the heating process. The sintering time was chosen on the basis of hit and trail method.

Figure 4.8. Trials for microwave sintering but not following

4.6 SAMPLE PREPARATION

Grinding and polishing

Coarse grinding was carried out on different sintered samples using the Silicon carbide sand papers of grit size 150, 220, 400, 600, 800, 1000 and 1200. Water was used as the lubricant during the grinding process. Further fine grinding was carried out using the grit size of 1500

and 2000. During grinding it was observed that cracks on the sample surface are unidirectional, that means all the scratch present on the surface are in one single direction, as any change in the direction of the polishing will lead to randomness of the scratch positioning which will hinder the view of the microstructure of the surface when viewed in the microscope. Also, it was ensured that the sample is parallel to the paper surface.

Once all the marking become unidirectional, the sample is then polished by turning it by 90°. Bearing this the mind, the surface is polished from all the sand paper grades in the same direction from beginning till the end until the surface becomes scratch free i.e. no scratch is left on the surface of the sample.

After fine polishing by sand paper of grade 2000, the samples were next polished on velvet cloth. For this 0.25 µm diamond paste is used together with the diamond lubricant. This method of polishing leads to mirror like finish on the surface on the sample. Care should be taken so that the finished or polished surface should not come in contact from any rough surface or substance as it might lead to scratch formation on the surface which is undesirable.

Figure 4.9. Polishing machine

Chapter 5

RESULTS AND DISCUSSION

5.1 DENSITY

Theoretical Density

As we know the density of AZ91 Mg alloy, and rest samples density is calculated by following formula:

$$\rho_{th} = w_1 * \rho_1 + w_2 * \rho_2$$

Where, ρ_{th}= theoretical density

Density of SiC = 3.21g/cm^3

Weight of sintered sample was measured using Archimedes principle by using weighing machine which is shown in Fig 5.1. Experimental density of sintered samples is calculated by following formula:

$$\rho_s = (\rho_w * W_s) / W_{w,}$$

Where, Ws = Weight of sintered sample

W_o = Weight of sintered sample within water

W_s = Weight of displaced water

ρ_s = Experimental density

$$W_s = Ws - W_o$$

$$\%Porosity = (1- \rho_s/ \rho_{th})*100$$

After all calculations, the result is written in the table 3 such as theoretical density, experimental density and percentage porosity of the sintered samples.

Table 3. Comparision table for theoretical and experimental density and porosity in the sintered samples

Sample	Theoretical Density (g/cm^3)	Experimental Density (g/cm^3)	%Porosity
Sintered sample 1	1.81	1.571	13.2
Sintered sample 2	1.88	1.627	13.4
Sintered sample 3	1.95	1.714	12.1
Sintered sample 4	2.02	1.735	14.1

Figure 5.1. Weighing machine which support Archimedes principle

5.2 OPTICAL MICROSCOPE ANALYSIS

Sintered Samples number 1, 2, 3 & 4 were made to undergo optical micrographs. AZ91 alloy has mainly two phases αMg and $\beta Mg_{17}Al_{12}$. These phases exist along and adjacent to grain boundary. Following optical micrograph shows the diffusion of SiC particles in sintered samples, taken at 100X. From these it can be inferred that sintered sample 3 & 4 has got better diffusion of SiC particles at the boundary as compared to the other two because of they have mixed with higher amount of SiC particles. Fig (5.2-5.4) below shows the distribution of the SiC particles.

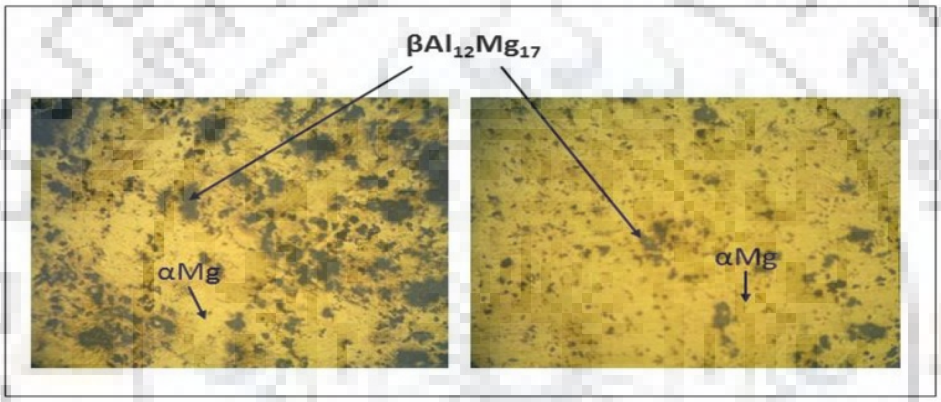


Figure 5.2. Optical micrograph showing the two main phases of sintered sample 1 using 100X magnification

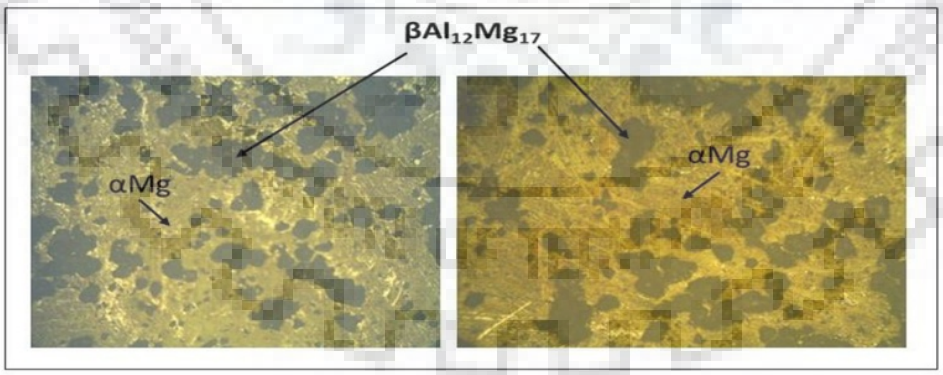


Figure 5.3. Optical micrograph showing the two main phases of sintered sample 2 using 100X magnification

It has been observed that sintered sample 3 and 4 has got agglomerations of the SiC particles (fig 5.4)

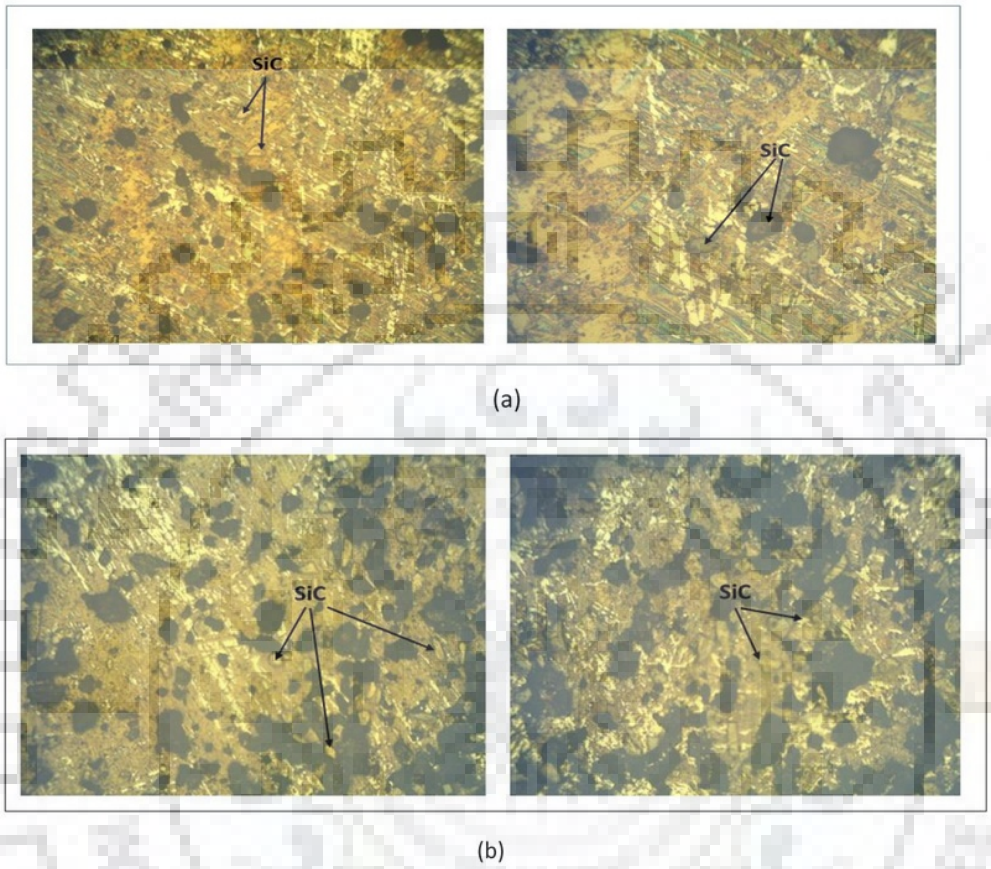


Figure 5.4. (a) & (b) Showing distribution of SiC particles of sintered samples 3 & 4

5.3 SCANNING ELECTRON MICROSCOPE (SEM)

Typical microstructure of the microwave sintered samples are shown in the figures. Microstructure of the sintered samples are having of the δ-phase which is solid solution, of Al in the Mg. It also shows the discontinuous precipitate $Al_{12}Mg_{17}$ i.e. comes under β-phase. In each sample, precipitate $Al_{12}Mg_{17}$ can be noticed at the grain boundaries along with, fine $MnAl_4$ precipitates. So from the SEM images we can say that the dark areas in the SEM micrograph corresponds to the αMg phase whereas the lighter areas show the $\beta Al_{12}Mg_{17}$.

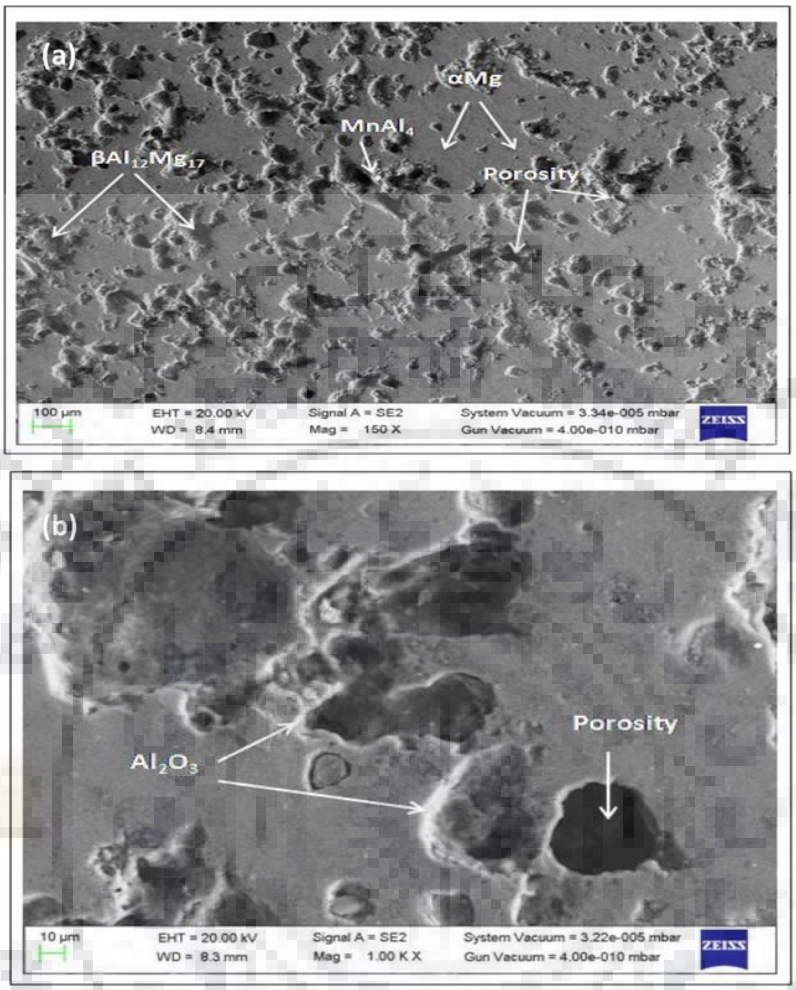


Figure 5.5. SEM images for sintered sample 1: (a) represents the different phases (b) showing oxide formation (Al_2O_3)

For better compaction of powder samples PVA was used. During sintering, PVA evaporate at 230° C as it is its meting point temperature. So due to evaporation of PVA it causes porosity on that positions. And the porosity is clearly seen in each sample.

The addition of SiC powder in AZ91 Mg alloy leads to ascent in the Mg_2Si phase, which is formed as eutectic particles in the form of Chinese script type, or present as a primary Mg_2Si coarse dendritic particles. The Mg_2Siphase is located generally in the δ-phase grains interior, and also islands of the entrapped δ-phase are often present in some Mg_2Si coarse dendritic crystals.

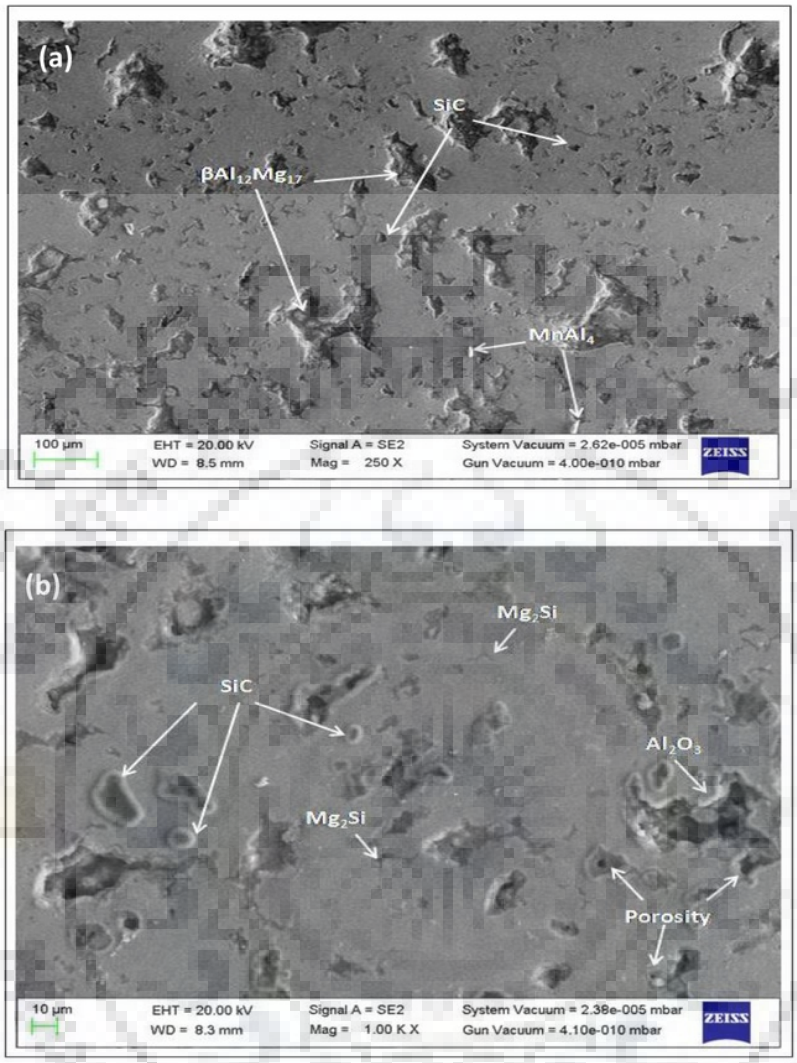


Figure 5.6. SEM images for sintered sample 2: (a) represents the different phases (b) showing the other different phases on 10µm area

SEM analysis was done to analyse, the chemical composition of the AZ91 Mg alloy/SiC interface. The excess of SiC can lead to the formation of Mg_2Si phase. The segregation of Zn has not been perceived [21].

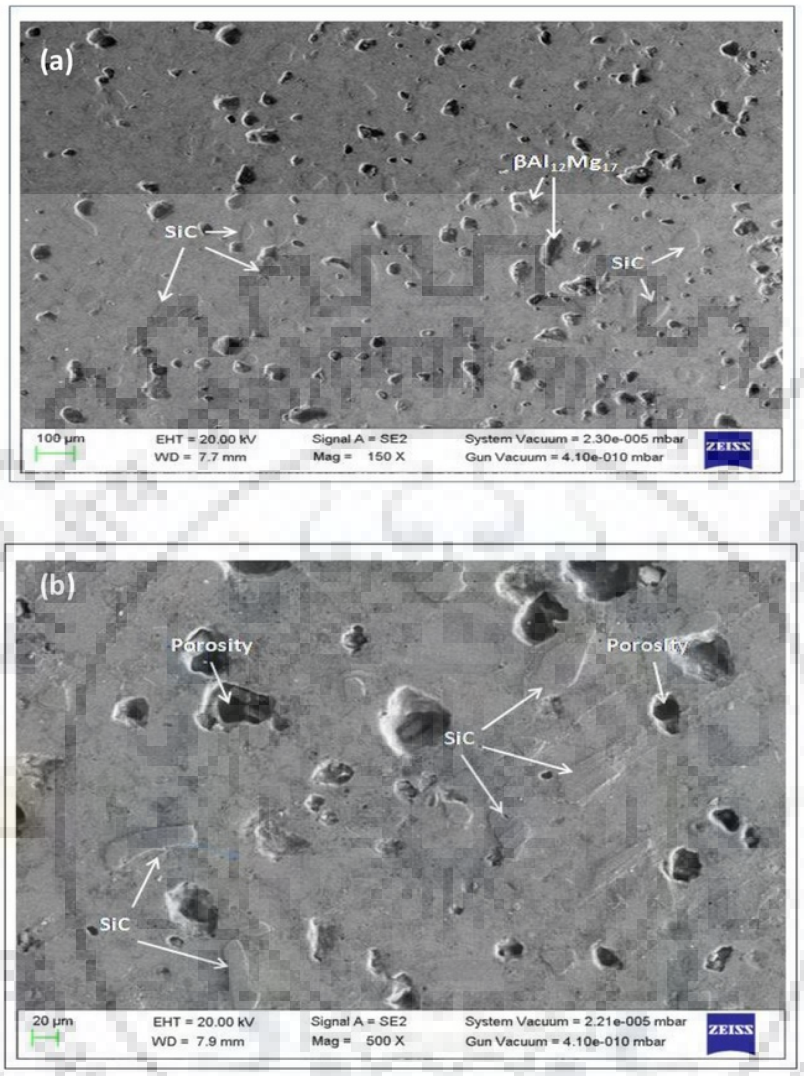


Figure 5.7. SEM images for sintered sample 3: (a) & (b) represents the different phases on 100µm & 20µm area

Sintered sample 3 is not showing the clear picture of the presence of Mg_2Si from Fig 5.7 but can observed the diffusion of SiC particles whereas sintered sample 4 depict the presence of all phases easily from Fig 5.8.

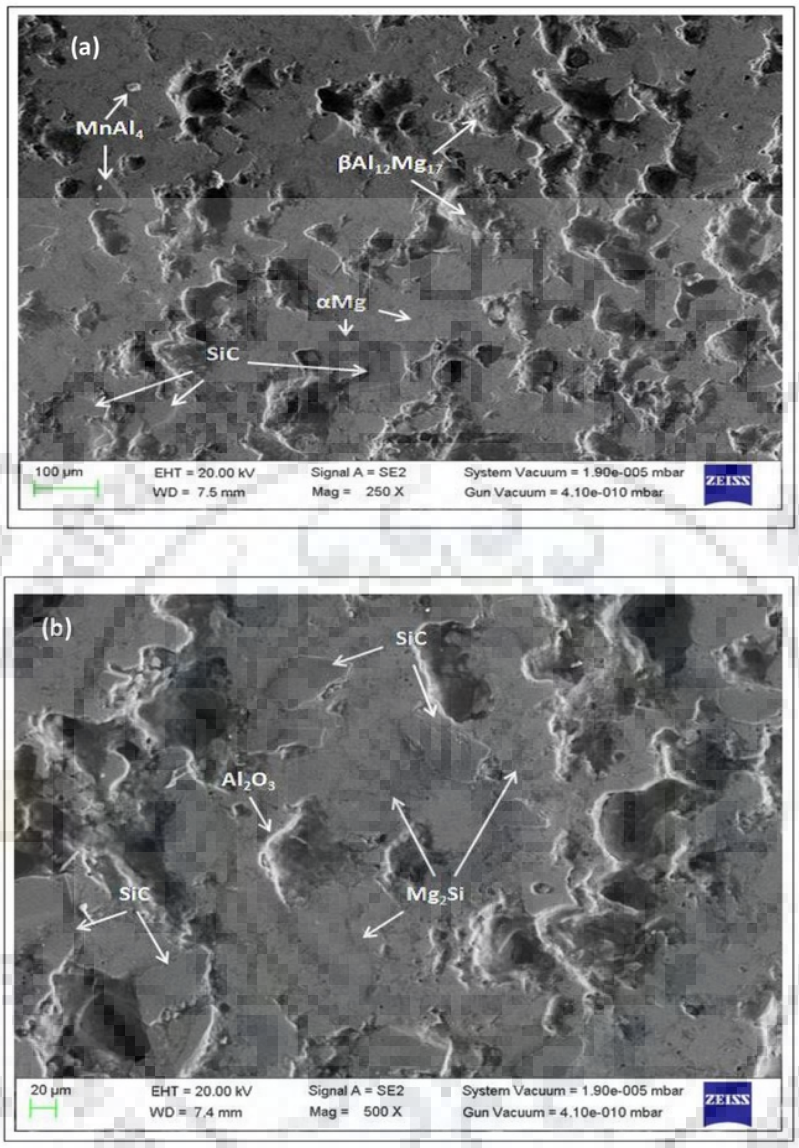


Figure 5.8. SEM images for sintered sample 4: (a) & (b) represents the different phases on 100µm & 20µm area

5.4 ENERGY DISPERSIVEX-RAY ANALYSIS (EDX)

EDX analysis was done on the sintered samples to ascertain the composition of the different phases present in the microstructure of the microwave sintered samples. The selected locations (spectrum) for elemental analysis and their corresponding results are presented in Fig 5.9, 5.10, 5.11 and 5.12. The X-ray elemental composition in Fig 5.9 (a) reveals that at spectrum 2

position presence of Mg & Al with contributions of approximately 38.17% and 36.38% respectively which indicate the presence of $\beta Al_{12}Mg_{17}$as indicated in SEM image Fig 5.9 (b). The X-ray composition study at spectrum 3(Fig 5.9. c) indicates the presence of C, O, Mg& Al elements (Fig 5.9. d)

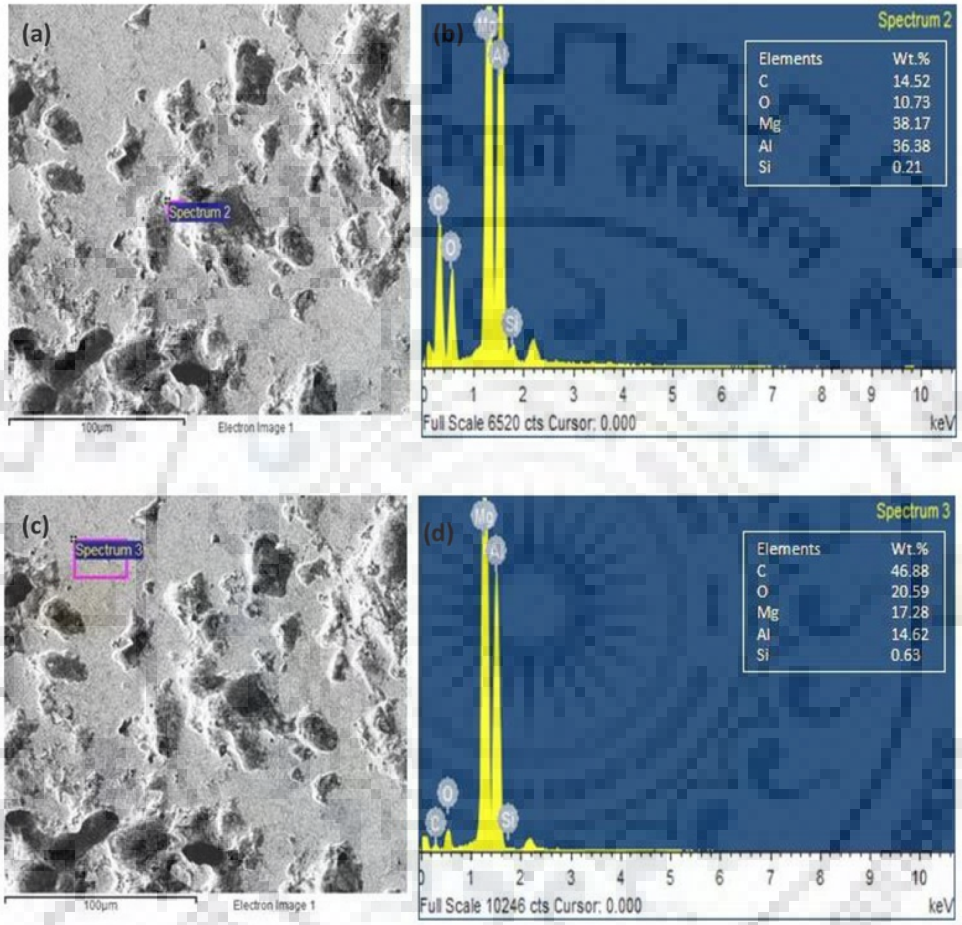


Figure 5.9. Typical EDS analysis of the sintered sample 1: (a)& (c) SEM image representing locations of EDS analysis; EDS spectra at (b) spectrum 2(d) spectrum 3

The X-ray composition study at spectrum 1(Fig 5.10. a) for sintered sample 2 indicates the presence of C, O, Mg& Al elements (Fig 5.10. b). It shows the formation of $\beta Al_{12}Mg_{17}$ and Mg_2Si phases.

In Fig 5.10. (d), it depicts the formation of SiC phase.

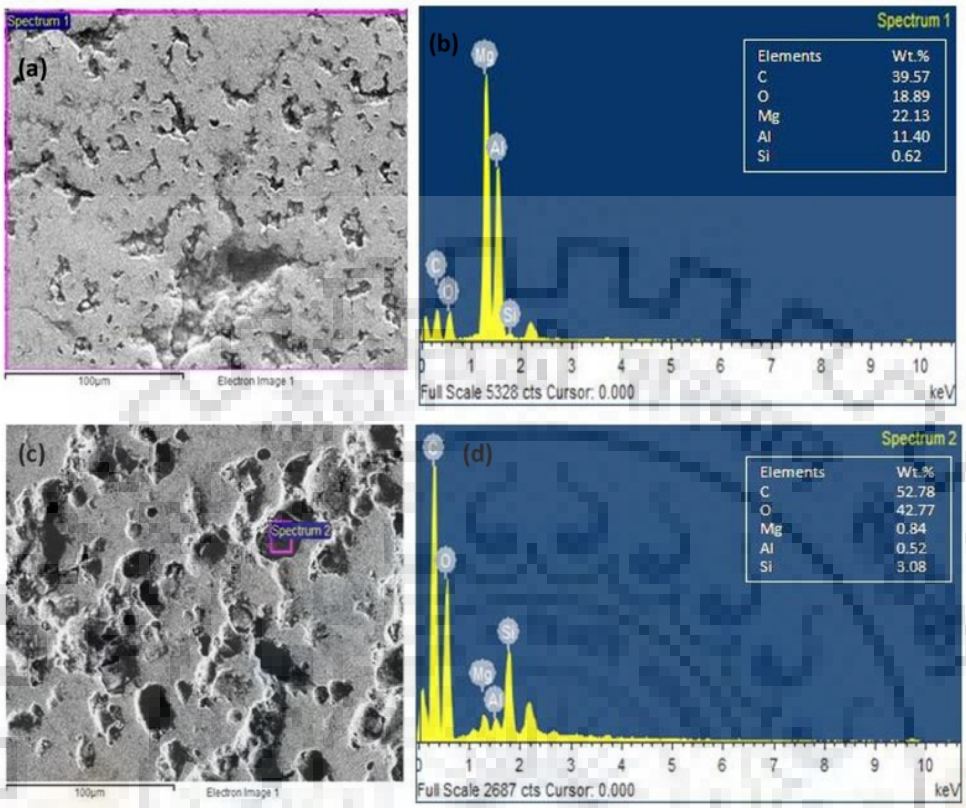
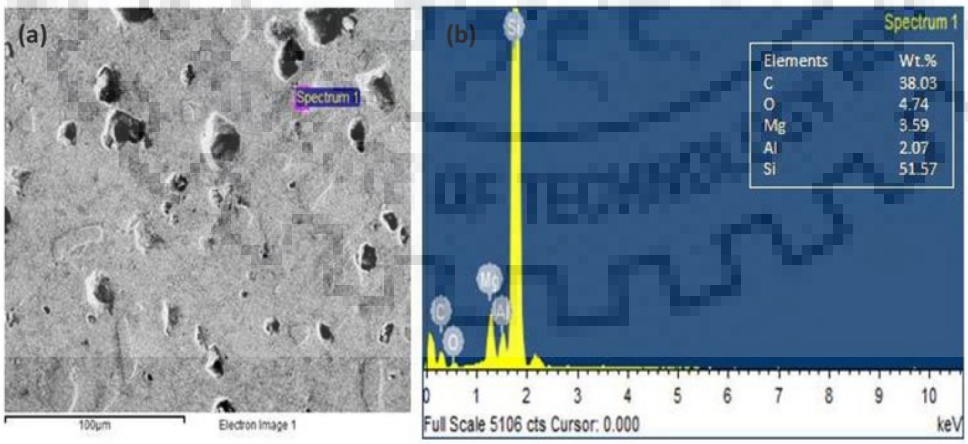


Figure 5.11. Typical EDS analysis of the sintered sample 2: (a)& (c) SEM image representing locations of EDS analysis; EDS spectra at (b) spectrum 1(d) spectrum 2

The X-ray composition study for sintered sample 3, both figures show the formation of SiC phase as it is also indicated in SEM images (Fig 5.11. b & d)



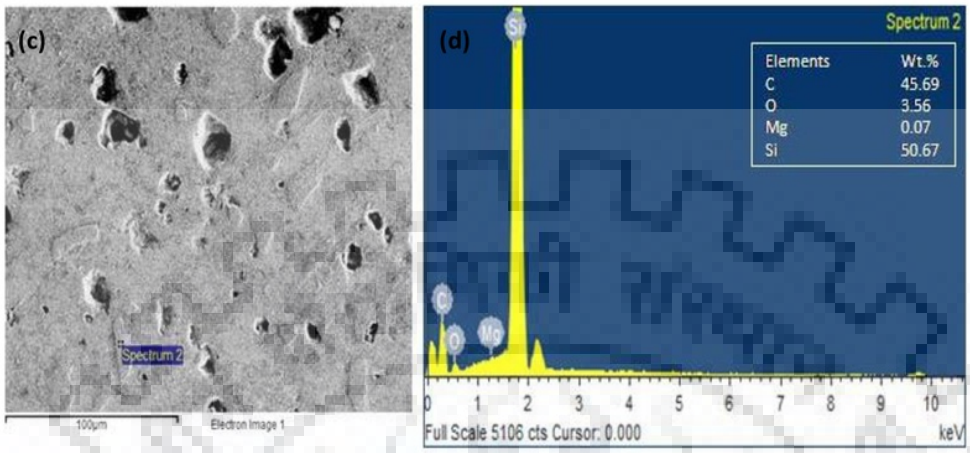
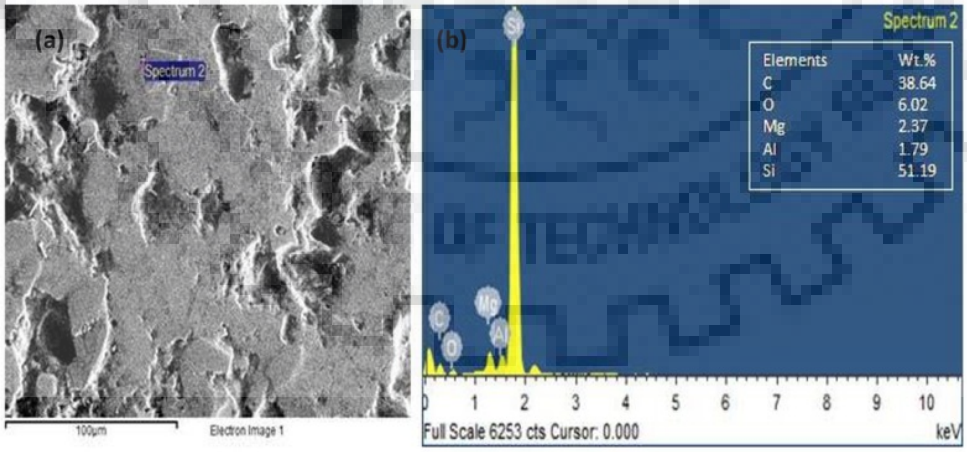


Figure 5.11. Typical EDS analysis of the sintered sample 3: (a) & (c) SEM image representing locations of EDS analysis; EDS spectra at (b) spectrum 1(d) spectrum 2

The X-ray composition study for sintered sample 4 for Fig 5.12 (a) show the presence of C and Si with contribution of approximately 38.66% and 51.19% respectively which indicates the formation of SiC phase as it is also indicated in SEM image (Fig 5.8)

In Fig.5.12 (b), it shows the presence of C, O, Mg, Al and Si in significate amounts which indicates the formation of Al_2O_3 (oxide formation) $\beta Al_{12}Mg_{17}$ and Mg_2Si phases.



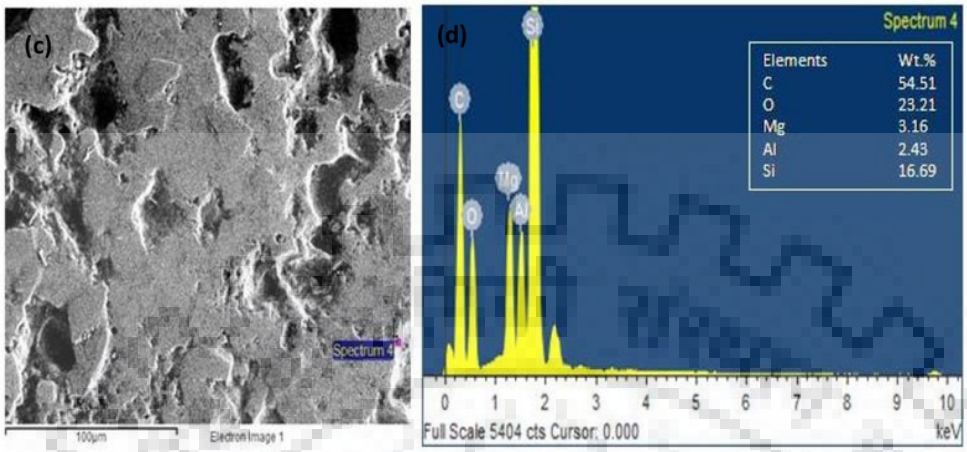


Figure 5.12. Typical EDS analysis of the sintered sample 4: (a)& (c) SEM image representing locations of EDS analysis; EDS spectra at (b) spectrum 2(d) spectrum 4

5.5 MICRO HARDNESS

Micro hardness analysis was done on the sintered samples. Load applied was 20 gm with dwell time of 10 seconds. Mean micro hardness for the sintered samples 1, 2, 3 & 4 were found to be 106.3 HV, 132.6 HV, and 115.7 HV & 89 HV respectively. From this result and above graph (Fig 5.13) we obverse that hardness increases to in addition of SiC but for sintered sample 4, it is lower than sintered sample 1 which is pure AZ91 Mg alloy. Micro hardness impression in sintered sample 1, sintered sample 2, sintered sample 3 and sintered sample 4 is shown in Fig 5.13

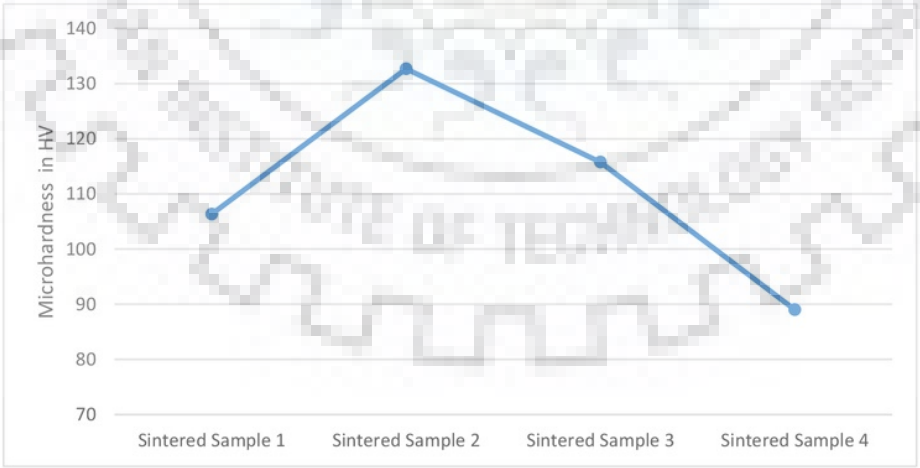


Figure 5.13. Micro hardness for sintered sample

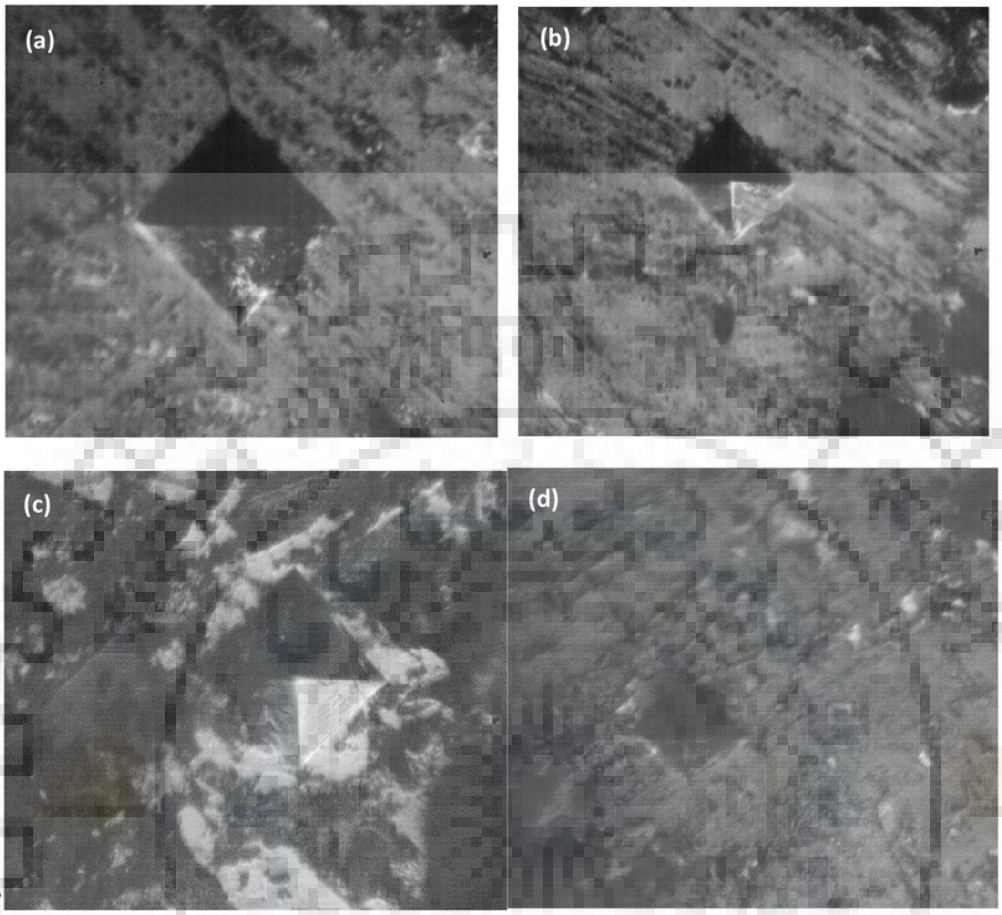


Figure 5.14. Shows the micro hardness impression in (a) sintered sample 1, (b) sintered sample 2, (c) sintered sample 3, (d) sintered sample 4

Chapter 6

CONCLUSION

Increasing demand of high yield strength materials, Mg and its alloys fascinates researchers. As of now it finds major application in automobile and aerospace industry. There are several traditional modes to sinter the Mg, its alloys and composites. In this article, an eco-friendly way of sintering is explored for Mg alloy by means of microwaves. The main conclusions extracted from this experimental study are the following:

i. Mg alloy easily reacts with the atmospheric air which leads to oxide layer formation and it reduce the binding strength of powder Mg alloy and strength of sintered solid bulk.

ii. Polyvinyl alcohol (PVA) was mixed in the mixture of AZ91 Mg alloy powder and SiC powder which causes porosity in microwave sintered samples.

iii. Domestic microwave does not give any idea of temperature at any particular time so it was difficult to analyse the temperature at which the compacted samples were sintered in microwave.

iv. Experimental density of the sintered samples are in the range of 85 to 90% of the theoretical density.

v. From optical micrograph, SEM images and EDS analysis, it is observed that on increasing the amount of SiC particles in AZ91 alloy more Mg_2Si and SiC phases are appeared in the sintered samples.

vi. Micro hardness results shows that for sintered sample 2(5% weight % of SiC in AZ91 Mg alloy) the hardness is more that the rest of sintered sample, So on increasing the amount of SiC particles in AZ91 Mg alloy hardness increases but after a certain percentage it reduces as sintered sample 3(10% weight % of SiC in AZ91 Mg alloy) but it has more hardness to sintered sample 1(Pure AZ91 Mg alloy) but Sintered sample1 micro hardness is more than that of sintered sample 4(15% weight % of SiC in AZ91 Mg alloy)

Chapter 7

FUTURE WORK

- Try to compact the powder without using any binder will give better density and less porosity.
- Flash sintering give the exact temperature of any time during microwave sintering process will give the better idea of sintering time and its corresponding temperature.
- Fracture properties will be analysed for the sintered samples.

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