ANALYSIS AND CHARACTERIZATION OF IRON DOPED SINTERED ABRASIVE MAGNETIC POWDERS WITHOUT BINDERS THROUGH POWDER METALLURGY ROUTE.

THESIS

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By

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CERTIFICATE

This is certify that the thesis report entitled "Analysis and Characterization of iron-doped sintered abrasive magnetic powders without binders through powder metallurgy route". being submitted by Mr. Shiv Dayal Dhakad to Lovely Professional university, Phagwara , Punjab, in partial fulfillment of the requirement for the award of the Degree of Master of Technology (Spl. in Manufacturing Technology) is a record of student's own work carried my supervision and guidance.

This thesis work is of desired standard and has not been submitted in any other University or Institution for the award of any other Degree

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Abstract

The mechanism of sintering and particular of the structure formation in the liquid phase sintering of sintered abrasive magnetic powder that was silicon carbide as abrasive and carbonyl iron powder as magnetic were studied, and investigated the microstructural development at three different pressures and at constant sintering temperature. The specimens were prepared through powder metallurgy route. The weight percentage of silicon carbide and carbonyl iron powder was kept uniform that is 1:1 weight/volume respectively. During the preparation of specimens the holding time was investigated to keep green compact from deforming before sintering. Micro hardness testing was performed by Vickers hardness tester and density was calculated by Archimedes principle. The changes in microstructure and its morphology were studied by using scanning electron microscopy (SEM) at different magnification. Transmission electron microscopy (TEM) was used to investigate the close surface morphology and EDS plot at single grain of sintered abrasive magnetic were studied and composition of sintered magnetic abrasive was obtained and concluded the traces of copper due to the composition of carbonyl iron powder. The structure of silicon carbide was thoroughly studied and obtained the crystalline structure after sintering. There was exponential increase in the micro-hardness at 5 ton,7 ton ,and 9 ton respectively.as well as the density also increases with increasing compaction pressure. Particle size of pure CIP ranges from 1–4 μm, most of the particles are in the range of 3 ~ 4 μm.

Keywords: Sintering, Abrasives, Morphology, TEM, SEM.

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Thank you to God for his faithfulness in my life. His promises are proven each and every day- even the bad days.

"I can do all things through Him who gives me strength." Philippians

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CANDIDATE'S DECLARATION

I, **Shiv Dayal Dhakad (Reg no-11000821)**, hereby certify that the work, which is being presented in the thesis ,entitled **"Analysis and characterization of iron doped sintered abrasive magnetic powders without binders through powder metallurgy route".** In partial fulfilment of requirement for the award of Degree of Master of Technology (Spl. in Manufacturing Technology) submitted in the Department of Mechanical Engineering at Lovely Professional University, Punjab, is an authentic record of my own work carried out during a period from August 2014 to April 2015 under the supervision of Mr. Ankit Bansal (Assistant Professor).

The matter presented in this thesis has not been submitted to any other University/ Institute for the award of Master of Technology Degree.

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LIST OF ABBREVIATIONS

S.NO	ABBREVIATIONS	FULL FORM
1	cm	Centi-meter
2	Kg-f	Kilo gram force
3	G	Average grain size
4	gm	Gram
5	G_0	Initial grain size
6	Gpa	Giga Pascal
7	J	Joule
8	K	Kelvin
9	ev	Electron volt
10	LPS	Liquid phase sintering
11	SAD	Selected area pattern
12	RS	Reactive sintering
13	SEM	Scanning electron microscopy
14	TEM	Transmission electron microscopy
15	SSS	Solid state sintering
16	XRD	X-Ray diffraction
17	Mpa	Mega pascal
18	SiC	Silicon carbide
19	EDS	Electron dispersive spectrum
20	CIP	Carbonyl iron powder

CHAPTER 1
INTRODUCTION

1.1 ENGINEERING MATERIALS

The three main categories in which engineering materials classified are polymer, metal & alloys, ceramic and glasses. polymer as well as metals and their alloys are mainly used in different application of engineering such as in structural engineering .From last four decades in the field of materials ceramics have gained a special importance as compared to polymers and metals it possess higher hardness, compressive strength, high oxidation ,corrosion resistance ,abrasion resistance, high melting point ,high wear etc.

There is need to design to advanced ceramics due to development in various materials technology enabled ceramicists for application in different fields like, space industries ,aerospace ,automobile, defense etc. The combination of primary material classes ,derived materials are formed and by using the advantageous properties of metals, ceramics or polymers forms a superior property combination ,this combination of the materials is known as composite. The composite are divided due to the presence of metal, ceramic and polymer as matrix into metal matrix composite, ceramic metal composite, and polymer matrix composite. Ceramics are refractory, inorganic materials with covalent and ionic bonds and are processed/used at high temperatures [1].

Ceramic are classified into oxide ceramics and non oxide ceramics, oxide ceramics are aluminium oxides (Al_2O_3) and zirconium oxide (ZrO_2),while non oxides ceramics are silicon carbide(Sic) boron nitride etc. Some inter metallic compounds beryllides, antimonides, arsenides, phosphides. Ceramic materials can be also classified into traditional and advanced ceramics, depending upon their final application. Traditional ceramics are the ceramic materials derived from naturally occurring raw materials, such as quartz, sand, clay etc.The application can be in making of earthward, cement, glass etc. There is need to design to advanced ceramics due to development in various materials technology enabled ceramicists for application in different fields like, space industries ,aerospace ,automobile, defense etc [2].

Advanced ceramics are being developed by tailoring the material property (control on composition and internal structure) to achieve the required property requirement for the system

by making use of materials science and technology. These ceramics usually are carbides (Sic), oxides (Al2O3), nitrides (Si3N4), non-silicate glasses, zirconia toughened alumina etc. and can be used in specialized applications as abrasives, electronic components, high temperature superconductors, automotive engines, cutting tools etc. Engineering ceramics are designed to tailor the material property (control on composition and microstructure) with an aim to achieve the desirable property requirement[3][4]. Advanced ceramics which serve as structural members (subjected to mechanical loading) and demonstrate enhanced mechanical properties under demanding conditions are generally known as structural ceramics. Structural ceramics with high melting temperatures (> 3000 °C) are generally known as ultrahigh temperature ceramics (UHTCs). Transition metal borides, carbides and nitrides are important examples of non-oxide UHTCs, which are of interest for various high temperature structural applications [3][4].

1.2 ABRASIVES

Abrasive comes from the word abrade, which means to rub off. An abrasive substance is a very hard tough material .when crushed and ground into grains like sand; it has many sharp cutting edges and points. Several common forms in which abrasive are used in metal working include the following. Abrasive cloth (also called coated abrasive) loose grain and powder abrasive. Abrasive compounds (in the form of paste, sticks or cakes) Grinding wheels , Sharpening stones Properties of abrasives. Abrasive must possess three common properties Hardness, Fracture resistance, Wear resistance. Hardness means the ability of the abrasive to cut the surface of the material being polished or ground. The fracture resistance of an abrasive is its toughness that is how well it resists breaking or crumbling during polishing or grinding when pressed against the work. The fracture resistance should be such that when the abrasive grains become dull they will break away when so broken, new sharp cutting edges are exposed. The fracture resistance of grinding wheels is related to the kind of bonding material that binds the grains together. The wear resistance of an abrasive is its ability to resist wear and stay sharp. This is related to the hardness of the abrasive material. Thus, harder abrasive materials generally are more wear resistant [5].

1.2.1 KINDS OF ABRASIVES

Abrasives are classified as either natural or artificial. Natural abrasive are minerals that occur in nature. Artificial abrasive also known as synthetic or manufactured abrasive are manmade. With the exception of diamond, artificial abrasives are harder than the natural abrasives. Artificial abrasives have largely replaced natural abrasive in metal working because of their greater hardness and wear resistance [5].

1.2.1.1 NATURAL ABRASIVES

Emery is one of the oldest kinds of natural abrasives used for metal working. It is black and is composed of a combination of corundum and iron oxide. Corundum is Aluminum oxide. Gemstones such as emerald and ruby are the purest form of corundum. Emery is about 60% corundum. Emery grains are not as sharp as artificial abrasives. The cutting action o emery is slight; therefore, it is used largely for hand polishing. Crocus is a fine, soft, red abrasive of iron oxide, or rust. It is produced artificially or found naturally and is used to polish steel surfaces to a high gloss. It is available in the form of crocus cloth or as a polishing compound known as rouge. Diamond is the hardest substance known. Industrial diamonds are known as black or borty diamonds [5].

1.2.1.2 ARTIFICIAL ABRASIVES

Pure aluminium oxide is produced by heating bauxite ore in electric furnace at extremely high temperatures. With the addition of small amounts of titanium, Greater toughness can be given to the aluminium oxide. It is broken up and crushed into fine grains for making grinding wheels, abrasive stones, and coated abrasives. Aluminum oxide abrasives are recommended for grinding and polishing materials of high tensile strength. These include carbon steels both while soft and after hardening; malleable iron; wrought iron; and tough bronze. Approximately 75% of all grinding wheels in use today are made with aluminium oxide[5].

Silicon carbide is made by heating a mixture of powdered sand, coke, sawdust, and common salt in an electric furnace. It comes from the furnace in masses of beautiful, bluish crystals. The crystals are crushed into fine abrasive grains. Then, the grains are used in making grinding wheels, abrasive stones, and coated abrasives. Silicon carbide is more brittle than aluminum oxide. However it is hard enough to cut aluminum oxide. Silicon carbide is used for polishing or grinding materials of low tensile strength. These

includecastiron,aluminium,bronze,tungstencarbide,copper,rubber,marble,glass,ceramics,magnesium,and plastics[5].

Boron carbide is produced from coke and boric acid in an electric furnace. It is harder than either aluminum oxide or silicon carbide, and can cut either of them. However; it is not as hard as diamond. It is used in stick form to dress or true grinding wheels .It is also used in powder form, instead of diamond dust, for polishing hardened steel or other very hard materials [5].

1.2.2 GRAIN SIZE OF ABRASIVES

Grain size refers to the size of the abrasive grains used in the manufacture of abrasive materials. The grain refers to the number of holes per inch .Thus; a 10 grain abrasive is one that will just pass through a 10 mesh screen inch a screen that has 10 meshes per inch [5].

Table 1 : **Properties of abrasive materials [6]**

Properties	**Aluminum oxide**	**Silicon carbide**	**Boron carbide**	**Zirconium oxide**
Molecular formula	Al2O3	SiC	B4C	ZrO2
Molar mass	101.96 g/ mol	40.10g/mol	55.255 g/mol	123.218 g/mol
Appearance	White solid	Colorless crystals	Dark grey or black powder	White powder
Odor	Odorless	Odorless	Odorless	Odorless
Density	3.95-4.1 g/cm^3	3.21 g/cm^3	2.52 g/cm^3, solid	5.68 g/cm^3
Melting point	2,072 degree centi	2730 degree centi	2,763 degree centi	2715 degree centi
Boiling point	2,977 degree centi		3,500 degree centi	4300 degree centi
Solubility in water	Insoluble		Insoluble	Negligible
Thermal conductivity	30W/m*k			
Crystal structure	Trigonal		Rhombhohedral	
Coordination geometry	Octahedral			
Refractive index				

1.3POWDER METALLURGY

The process in which blending of powder materials then compacting them into the required shape, and compact the blended powder is known as powder metallurgy, Generally known as compaction, after the compaction process the compact shape is heated in a controlled atmosphere so that the material bond together finely so that the density of the compact can be increased

which result the decrease in porosity. The powder metallurgy process carried out into four steps or can say the powder cycle from initial raw material to final product, as they are manufacturing of powder, blending of powder, compaction of powder, and heat treatment i.e. sintering. The process of compaction is mainly performed at room temperature, and the elevated temperature-process of sintering is mainly done at atmospheric pressure[7].

The two main techniques used to form powder are metal injection moulding and sintering. Recently rapid manufacturing techniques have made it possible in which metal powder for the manufacturing of products.Because in this technique the powder is melted instead of sintering to achieve better mechanical properties.

1.3.1OVERVIEW OF VARIOUS PROCESSINGS RELATED TO ABRASIVE AND MAGNETIC POWDERS

The processing of abrasives and magnetic powders includes the mixing of appropriate amounts of commercially available or in-house prepared powders (Al2O3, etc.) with desired sintering additives. After mixing, the slurry is dried and crushed down into fine particles using high energy ball milling. The mixed powder is then filled inside a die and placed inside the sintering furnace. During the sintering, the temperature increases to the desired sintering temperature, which is monitored/regulated by thermocouple or an optical pyrometer. During hot pressing, uniaxial pressure is usually applied from the beginning of the sintering cycle to obtain densification at lower temperatures as well as to obtain compact with homogeneous properties. The properties of the ceramics are strongly dependent on the microstructure, which is determined by careful selection of initial starting powders (powder, particle size and shape, purity etc.) and the sintering conditions (time, temperature, pressure, heating rate, hold time [8].

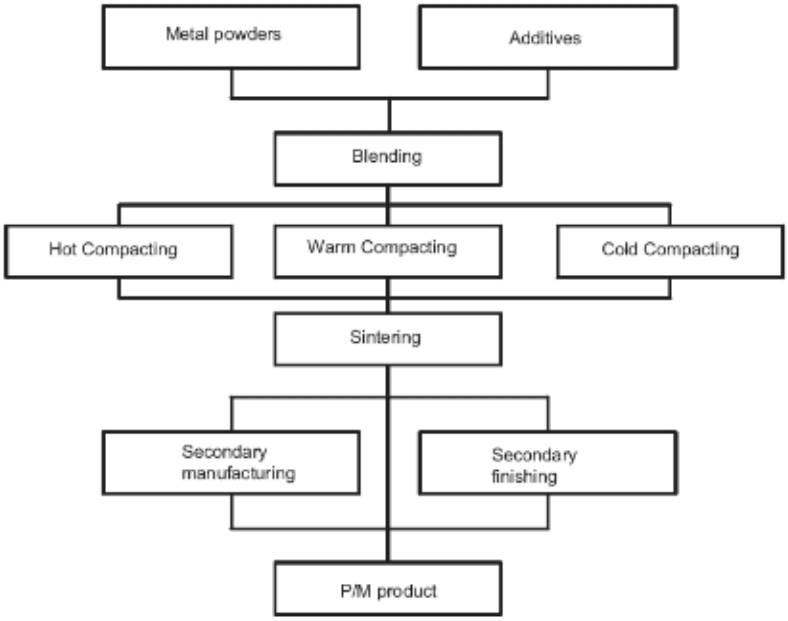

Figure 1: Simplified flow diagram indicating various operations in powder metallurgy processing

1.3.2 MIXING AND BLENDING

The process of combining different material contains different properties is termed as mixing ,such as iron and nickel, alumina and zirconia, tungsten carbide and wax. Blending is the term used for homogenizing same material chemistry but with different material characteristics such as particle. Blending is essential in powder preparation because of inherent segregation of the particles during transportation. Mixing and blending are important processing operations in powder preparation prior to compaction. Blending and mixing are accomplished by mechanical means by four ways [9].

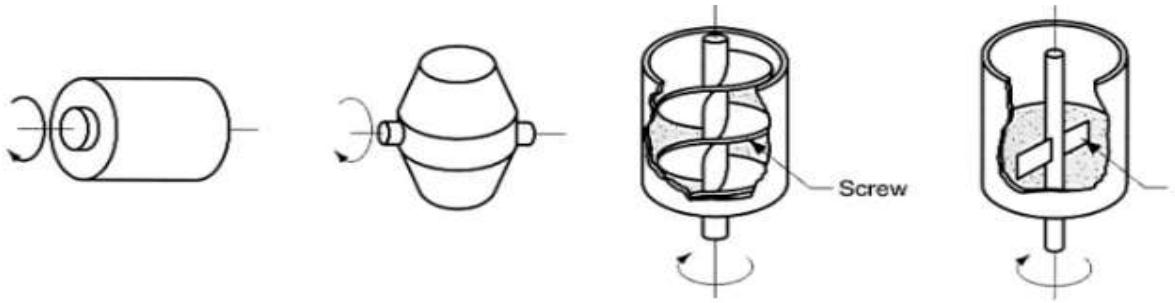

Figure 2:Blending process

Except for powders, some other ingredients are usually added:

Lubricants: To reduce the particles-die friction.

Binders: To achieve enough strength before sintering.

Deflocculants: To improve the flow characteristics during feeding.

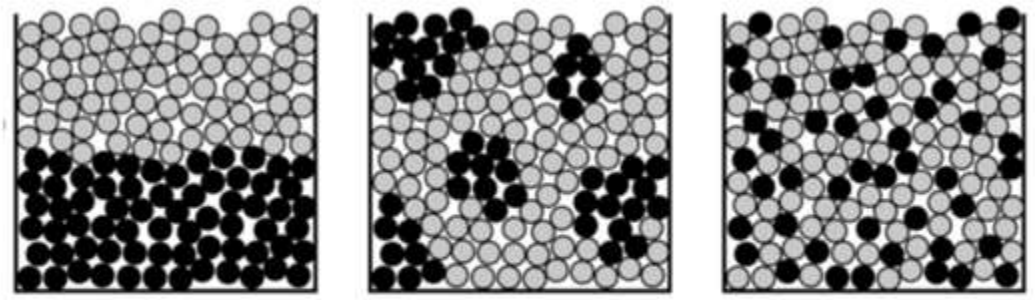

Figure 3 : Homogeneity increased after blending process

1.3.3 POWDER COMPACTION

The process in which the metal powder is compacted, due to the application of high pressure in a die. The position of tool are held in the vertical orientation consist of punch tool forming the bottom of the cavity. After that the powder is compacted into desired shape and then removed from the die of the hydraulic press. In various applications the part manufactured required very little work for their use to make very cost efficient manufacturing [10].The pressure applied is directly proportional to the density of compacted powder. The pressure ranges from 80 psi to 1000psi, pressure from 1000 psi to 1000000 psi can be obtained. The average pressure used for the compaction of metal powder is varying from 10 tons/in^2 to 50ton/in. Multiple lower punches are used to attain same compression ratio for the component of more than one level .cylindrical work piece and more complex shapes can be made by single level tooling and multiple level tooling respectively. The tool styles are divided into four major classes they are single action components, used for flat components, thin components. opposed double action with two punch motions , which is used for thicker components, double action with floating die and double action with withdrawal die .As compared to single action ,double action gives better density.

Designing of tooling must be done in such a way that it can withstand the maximum pressure without any fracture , bending or deforming. Material should possess the properties of corrosive resistive, wear resistant. By repressing and re-sintering best work piece materials can be obtained[11].

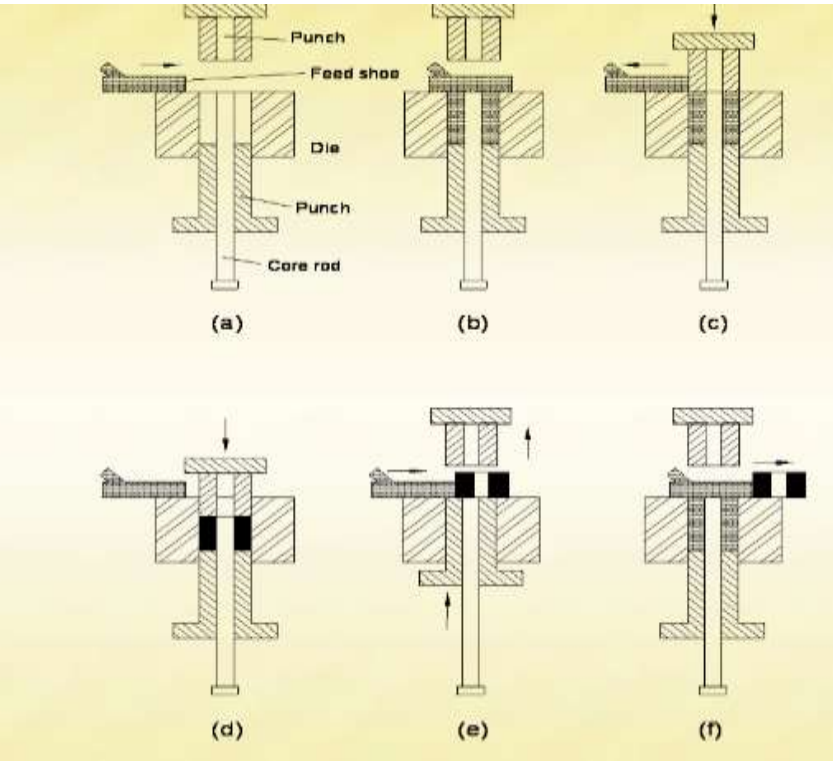

Figure 4 : Stepwise process of compacting powder in punch and die

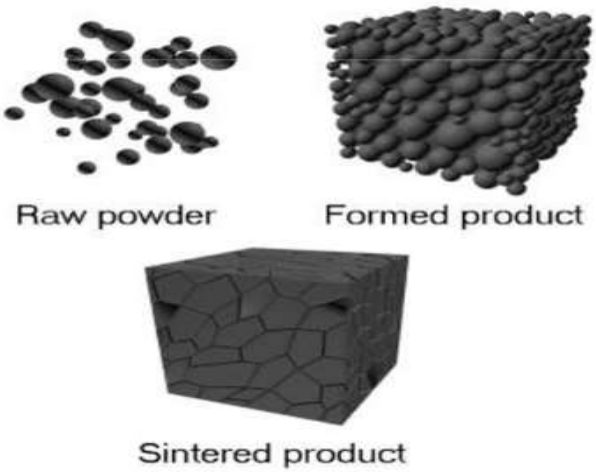

Figure 5 : Powder, Green Compact, Sintered Compact

1.3.4STARTING POWDERS

The final microstructure can be affected by the distribution in size and powder size. its agglomeration and shape i.e. its shape and grain size ,porosity, grain boundary area and in the same way physical and mechanical properties of ceramics effected .presence of bimodal distribution play an important role as provide an advantage used during processing of component ,as it allows finer particle to fill in the spaces between coarser particle. Due to which densification increased and lowers the porosity. Due to large surface area available for mass transport finer nano powders are easy to densified. Maximum densification can be achieved by close contacts amoung the particles due to enhanced neck growth during sintering. Moreover, finer the grains more are the grain boundaries, which are regions of higher energy or disorder and are responsible for higher atomic mobility. By Herring's law,

$$\frac{t(r_2)}{t(r_1)} = \left[\frac{(r_2)}{(r_1)}\right]^p$$

where, p = 2-4, t(r) = time taken to sinter a compact of uniform particle size 'r' and r1, r2 are two different particle sizes. Hence, it is beneficial to use finer sized starting powders to enhance densification (usually obtained by milling). However, the presence or formation of powder agglomerates must be avoided, since agglomerates impede the effective transfer of heat and pressure to the particles. This results in attainment of non-uniform properties across the sintered material. The shape of the powder particles also plays an important role in sintering kinetics. The spherical particles possess least surface areas and thus require higher energies for effective mass transport. On the contrary, irregular shaped particles create more contact paths, thereby enhancing the mass distribution [12].

1.3.5 SINTERING CONDITIONS

Sintering time, temperature, heating rate,and atmosphere are important variables, which need to be optimized for achieving maximum densification with improved physical and mechanical

properties. Usually at higher sintering temperatures, faster and irregular grain growth occurs. At lower sintering temperatures and lesser sintering times, more number of pores exists on the grain boundary, which impede the grain boundary movement and hence, grain growth. At higher sintering temperatures the number of pores decreases, leading to improved density of the material but with increased grain growth. Sintering time should be optimized so that considerable diffusion and mass transport occurs, leading to removal of closed porosity but without promotion of grain growth. Sintering time affects the grain growth by the following equation .

$$G = G_o + k.t^n$$

Where, G = average grain size.

Go = initial grain size.

t = sintering time.

n = growth coefficient (0.5, theoretically).

1.3.6 VARIOUS SINTERING PROCESSES

Sintering is an ancient processing technique of firing and consolidation of powders at T > 0.5 Tm, where the diffusional mass transport mechanisms lead to grain growth and finally, a dense ceramic. Almost all ceramic products and metal components, such as space vehicles, ballistic armor materials, bioceramics, bearings, valves etc. can be made by sintering. Typically, sintering involves preparation of a powder blend, followed by compaction to obtain green powder compact and finally consolidation at high temperatures. This schedule is often followed by post sintering and finishing operations. The green powder compact is flaull, as it contains a large amount of porosity. Sintering allows heating of the compact, thus forming strong solid by removal of porosity via mass transport of atoms from particles to the empty regions either via lattice diffusion or via grain boundary diffusion, thereby involving the dynamic and continuous change in the pore size and shape [12]. Two major micro-structural changes occur during sintering and they include porosity reduction causing densification and grain growth. In turning the free surfaces of the particles into grain boundaries, the total free energy of the material is reduced. This provides the driving force to eliminate the pores and increasing the density of the material. An external pressure/force is sometimes applied during sintering along with the thermal

energy to accelerate the densification process. The densification process is usually accompanied by grain growth, since it further reduces the free energy of the powder compact. At the forefront of the ceramic technology, efforts are made to sinter nano-structured materials and functionally graded components with desired properties at lower sintering temperatures in lesser times.

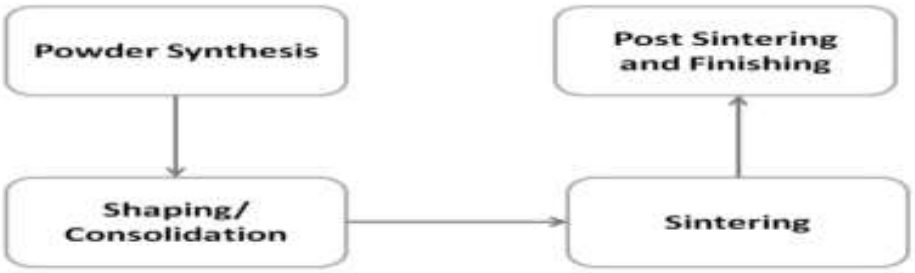

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Figure 6 :Flow Chart of various P/M processes

Figure7:Sintering Furnace

CLASSIFICATION

Sintering can be broadly classified into three categories: Solid state sintering (SSS), Liquid phase sintering (LPS) and Reactive sintering (RS).

1.3.6.1SOLID STATE SINTERING

During solid state sintering (SSS), no liquid phase is formed and the powder compact is densified truly in the solid state at sintering temperature. The solid-vapor interfacial area is continuously being replaced by solid-solid grain boundaries during SSS. The process involves diffusion to enhance the mass transport, leading to neck growth in the powder compact. SSS takes place in mainly three stages [13],

a) Initial stage: Initially, the particles (diameter D) come in contact and form a weak cohesive bond. Vapour phase transport occurs due to the surface diffusion, leading to neck formation at the inter particle region.

b) Intermediate stage: Due to continuous heating, effective neck growth occurs due to grain boundary and lattice diffusion, causing pore coarsening, thus leading to formation of interconnected pore channel. This continuous pore channel then breaks and finally the pores are removed. The theoretical densities of around 70-92 % are achieved during this stage.

c) Final stage: It is a slow process, where the pores become spherical and closed. Near theoretical density is achieved during this stage along with the presence of some isolated pores. Importantly, the grain growth takes place during the final stage of sintering.

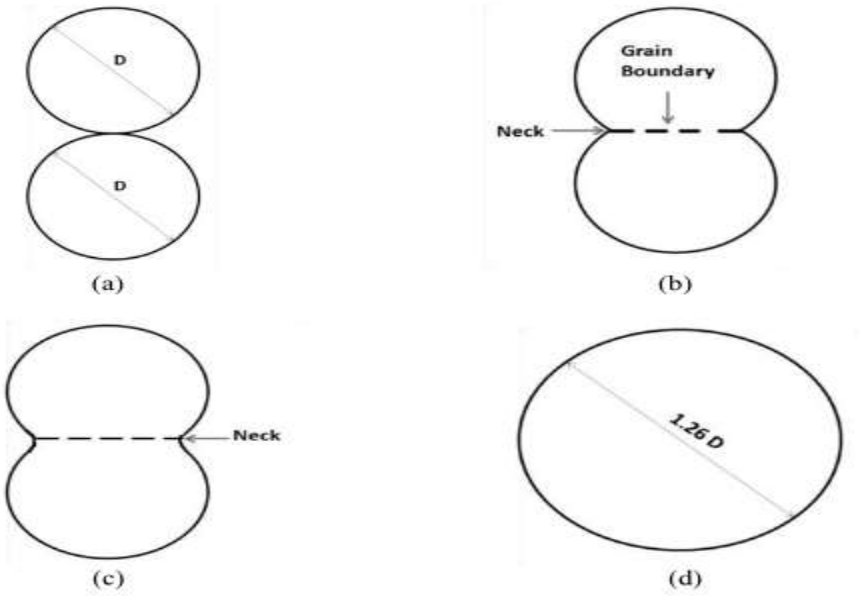


Figure 9 : Schematic illustrating different stages in SSS with the help of two-sphere model (a) adhesion, (b) Initial stage, (c) Intermediate stage and (d) Final stage.

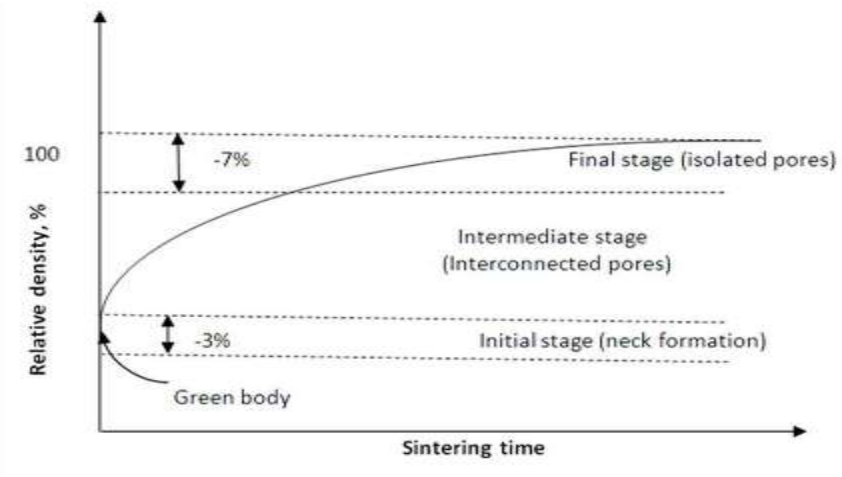


Figure 10 : Schematic illustration of three stages in solid state sintering of a powder compact

1.3.6.2 LIQUID PHASE SINTERING

Liquid phase sintering (LPS) involves formation of the liquid phase either prior to or at the sintering temperature of the powder. The liquid phase is typically formed due to oxide layer present on the surface of the powder particles or by reaction of powders with the sintering atmosphere. The molten liquid phase penetrates the particle boundary and surrounds them. The rapid mass transport due to increased diffusion in the presence of liquid phase leads to the achievement of high compact densities in difficult-to-sinter materials. LPS occurs in three overlapping stages [14].

a) Particle-rearrangement: This stage usually occurs within ~ 10 min of the formation of low-viscosity sintering liquid. The capillary action helps in driving the liquid into the pores at the solid-liquid interface. The solid particles flow under the influence of surface tension forces, presented by surrounding liquid. This causes the rearrangement of the grains into the most favorable packing arrangement, as shown in. It should be kept in mind that optimum wetting is required during LPS such that the liquid film completely spreads over the solid surface.

b) Solution-precipitation: With continued heating, the smaller solid particles dissolve in the sintering liquid. Solid phase atoms are then carried away from the contact areas by the liquid,

which then re-precipitate on the larger particles .This leads to faster grain growth and hence, densification.

c) Final Densification: Solid-solid contact is established, leading to densification of the network. In this stage, the rate of densification slows down and is finally stopped.

Reactive sintering (RS) is similar to LPS. It involves in-situ sintering reaction between the starting powders. The product phase which is mostly a liquid phase thus formed, helps in enhanced mass transport. RS occurs as a combination of two processes: reaction and densification. At higher reaction rates, the process is completed before densification is achieved. Hence, one can achieve almost full densification at lower temperatures. The selection of temperature plays an essential role during RS. Increasing the temperature promotes the formation of liquid phase. The liquid phase assists in mass transport, resulting in densification. Hence, a balance between sintering reaction and densification is required. Illustrates the balance between densification and sintering reaction to achieve higher density, before reaction interferes with the densification process.

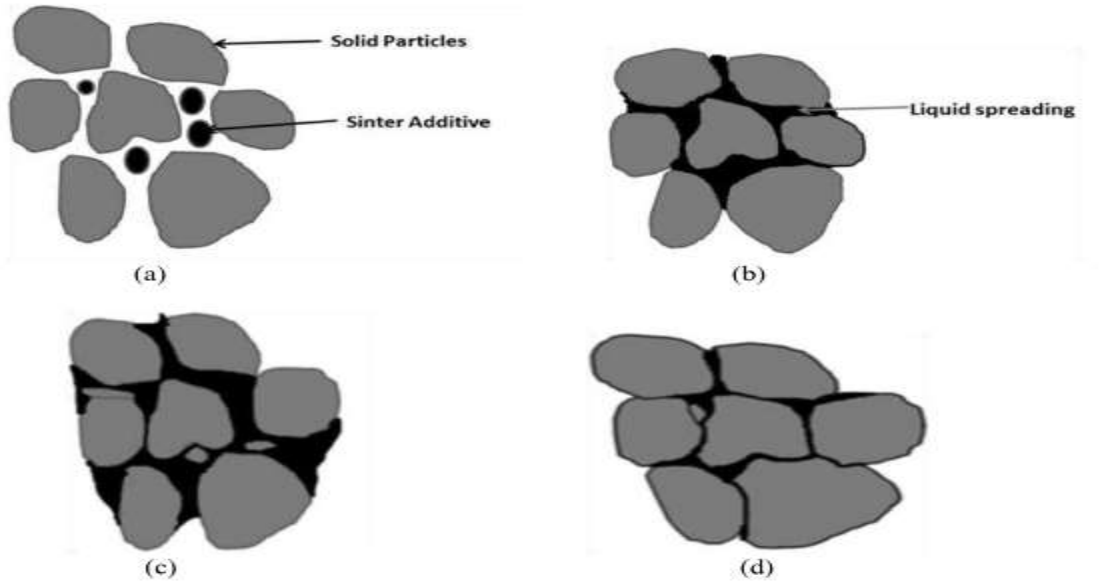


Figure 11 :Illustration of different stages of LPS (a) green body, (b) rearrangement, (c) solution precipitation and (d) solid densified compact .

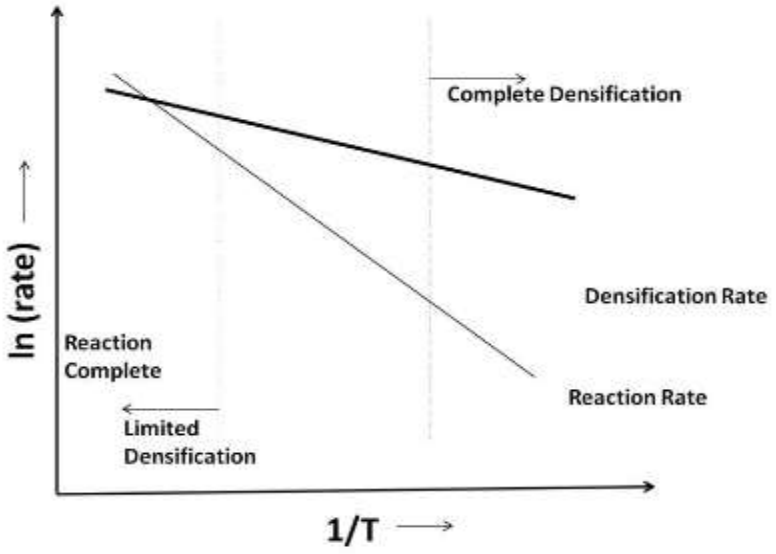


Figure 12 : Balance between densification and sintering reaction during reactive sintering.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

This chapter described the background of the research, how much work had done so far. The quality journal were reviewed to this research purpose.

2.2 REVIEWED LITERATURE

[18][Perevislov.et.al] have studied the structure formation of sintered silicon carbide and sintered silicon nitride in liquid phase sintering. Grain boundary layer ,inter-grain phase as a result of formation of structure core was also studied .Author concluded that the microstructure of the liquid phase silicon carbide material consist of an inter-grain oxide phase and SiC grains. SEM,TEM techniques were used for characterization. The data obtained on the LPS of SiC and Si3N4 make possible to model the properties of the LPS-SiC and SSN materials obtained.

[19][Gadzyra.et.al] have studied the structural characterization of SiC-C powder and Y_2O_3 and Al_2O_3 and titanium hydride during hot pressing and free sintering were studied. Done dispersion hardening with nano- sized particles of titanium and silicon. And also examined micro-mechanical properties of the composite. Author concluded that ceramics produced by free sintering had high hardness as compare to the powder produced by hot pressing .but powder produced by free sintering had inhomogeneous structure with porous areas and high density as compare to hot pressing. High density and most homogeneous structure obtained at hot pressed1850℃ temperature. Electron microscopy revealed that staring powder composition remains multiphase in hot pressing too.

[20][Nader.et.al] have studied the properties of silicon carbide, sintered at liquid phase sintering i.e. its topography, morphology and transformation in kinetics. By using Y_2O_3 as sintering binder in gas pressure sintering and pressure-less sintering in the presence of argon and nitrogen atmosphere as argon was used to decrease the temperature or for cooling. Optimized the transformation of phase to SiC from β and also studied the effect of initial content and sintering technique like atmosphere, rate, temperature etc. And investigated that the rate of transformation of phase decreases with increase in content of β in the initial powder in presence of N_2(nitrogen). Equiaxed grains with homogeneous microstructure shows in the material without phase

transformation. Microstructure developed from SiC powders were elongated grain type when phase transformation occurs. Since in LPS-SiC showed an inter-granular fracture mode, the shape, and grain size, had a significant effect on the mechanical properties. After hot pressing material exhibited stretched microstructure possessed higher bending strength and toughness of materials with equi-axed increased twice.

[21][Zhitnyuk.et.al]have studied the processing of LPS(Liquid phase sintering) for the formation of silicon carbide based ceramic by the addition of eutectic additives. And compare the reaction sintering and liquid phase sintering and obtained that the synthesized samples were similar to the specimens that is silicon carbide obtained through liquid phase sintering but had different energy efficiency.

[22][Shinoda.et.al]have investigated nano-crystalline SiC ceramics was heat treated by spark plasma sintering using Al_2O_3 and TiO_2 as additives and investigated formation of TiC phase in XRD analysis and at 20mass % addition mullite phase with preferred orientation were detected. Author investigated mullite crystal phases, TiC and alumina-silicate phases were identified. As compare to aluminium and oxygen atoms segregation of titanium atoms was observed at inter-granular glassy phases. The electrical conductivity was the same level as the Al2O3 single addition to SiC ceramics.

[23][Pujar.et.al] have investigated densification of LPS-SiC and optimized that the composition of the liquid phase plays an important role on the densification of LPS-SiC ceramics. due to optimum liquid phase composition resulted the complete densification. The degree of densification in each sintered specimen was primarily determined from the residual porosity in the specimens Total densification occurs due to the composition of the packing bed powder strongly influences volatilization of vapour species.

[24][Nagano.et.al] have investigated effect of atmosphere on superplastic deformation behaviour in nano-crystalline liquid-phase-sintered silicon carbide with Al_2O_3 and Y_2O_3 additions. And obtained there were no phase transformation or multiplication of dislocation were investigated in deformed samples .Due to the vaporization if grain-boundary phase and grain growth during experiments prevented elongation. Tension test showed that a crystalline phase was formed at the triple point and multi grain junction. There were no phase transformation or multiplication of dislocation was observed. Formation of cracks takes place in vertical direction of tensile axis in deformed samples.

[25][jing-mei Ma et.al]have studied microstructure and properties of liquid phase sintered silicon carbide and used hot pressing sintering technique for the initial powders αSic and BaAl2Si2O8 respectively. Author investigated the effect of additives on microstructure, densification, flexural strength and fracture behaviour of liquid phase sintered composite.Author concluded that the BaAl2Si2O8 plays an important role in the densification of sintered liquid phase composite. The flexural toughness and flexural strength obtained to be maximum. Crack deflection, SiC grains pull out and crack bridging were main toughening mechanism used by the author. The mechanical properties and relative densities of the sintered composite increased with increasing BaAl2Si2O8 content. Author concluded that the mechanical properties were directly propotional to the relative densities.

[26][Lin .et. al]..have prepared the magnetic abrasive by P/M route, By typically mixing of Al2O3 and iron powder at the ratio of 40:60 wt % with the average size of grain is 5 microns and compressed the mixture in cylindrical shape. Sintered the compact in vacuum furnace, these compacts were crushed after the sintering process to produce average size 150 microns magnetic abrasive. Author with these magnetic abrasive used magnetic pole of ball shaped designed with special grooves. And concluded that this special design increases the finishing efficiency and also provide good surface finish for the non-ferromagnetic material, SUS304.The best result was optimized at abrasive mass of 2gms, feed rate of 10m/min and with a working gap of 2.5mm respectively. The obtained maximum percentage improvement surface roughness was 60%. These abrasives were used for finishing of SS 304 material.

[27][Zhang et. al] have studied on magnetic abrasive finishing by sintering method and experimented on MAF (magnetic abrasive finishing) The main parameter on which author focused were, on effect of sintering temperature, particle size ratio of abrasive and magnetic, sintering time, and sintering characteristics of magnetic particles on magnetic abrasive during the finishing process, so as to achieve a better process and principle for MAF (magnetic abrasive finishing). The best result optimized by author surface roughness was 65%. These abrasives were used for finishing of SS 304 material.

[28][Shinmura et.al] prepared mixed type of magnetic abrasives by mixing of iron particles of various sizes and sintered magnetic abrasives. These mixed magnetic abrasives were used for internal finishing of SUS304 steel tubes and clean gas bomb shells. They found that the magnetic

force of the mixed-type magnetic abrasives takes the median value between those of the magnetic abrasive and iron particles. The magnitude of magnetic force increases with increasing mixed weight percentage of iron particles but the number of the cutting edges gets reduced. The obtained maximum percentage improvement surface roughness was 30%. These abrasives were used for finishing of SS 316 material.

[29][Jain et al] used loosely bonded magnetic abrasives (mixture of iron, Al2O3 and lubricant) for external finishing of stainless steel cylindrical rod of diameter 48-50 mm. It was reported that the improvement in the surface finish is 60.83% with MRR of 58.6 mg/min. These abrasives were used for finishing of SS 304 material.

[30][Kremen et. al] developed magnetic abrasives using an adhesive to bind magnetic component (iron powder) with abrasive component (diamond powder). All the three components were mixed thoroughly, dried and crushed into small particles of desired size for machining. Then by using glued magnetic abrasive powder and keeping magnetic flux density 0.4 tesla, machining time 5 minute and adding 4% of boric acid in water as cooling fluid, the effect of powder grain size on the surface roughness and MRR of a silicon wafer and tube was observed. The obtained maximum percentage improvement surface roughness was 45%. These abrasives were used for finishing of SS 316 material.

[31][Raj et al] have studied the investigation on the effect of sintering temperature and time intervals on workability behaviour of Al–SiC powder metallurgy composites during cold upsetting was attempted in the present work. Three levels of sintering temperature and time have been considered to evaluate their effect on workability behaviour. The amount of SiC reinforcement content has been varied as 0%, 10% and 20%. The experimental results were analyzed for workability under triaxial stress state condition as a function of the relative density. densification. Stress ratio is higher in Al–Sic composite with compare to aluminium due to better densification. And also compared the stress ration of the composite .concluded higher sintering temperature initiates cracks with higher fracture strain.

[32][Prochazka et al]..have studied the pressure less sintering of silicon carbide , Author synthesized b silicon carbide in the submicron range which was sintered after addition of 0·5 wt% boron and 1 wt% carbon in the temperature range 2050° ~ 2150°C to densities of 96% in

argon atmosphere. In its pure form, SiC powder will not sinter to a fully dense state. By heating at the temperature range of 1900°~ 2300°C with pressures ranging from 100–400 MPa, Author also reported that with addition of boron and carbon to submicron size b-SiC, sintering of silicon carbide to near theoretical density was achieved

[33][Zhong et al]..have studied Silicon carbide ceramics were prepared with SiC powder treated by the fluidized bed opposed jet mill as raw materials, and the effects of the ultra-fine treatment mechanism on the compaction and sintering behaviour of SiC ceramics were investigated. The results showed that the compacts had higher density and microstructure homogeneity when the sintering temperature of the compact was decreased; and that the surface microstructure, densification and mechanical properties of the sintered body could be ameliorated obviously. Author explained investigation for ultra-fine treatment of industrial SiC powder by fluidized bed opposed jet mill. The results obtained by the author that the compacts had relatively high density and microstructure homogeneity; the sintering temperature of the compact decreased; the surface microstructure, densification and mechanical properties of the sintered body can be ameliorated obviously by proper control of the sintering temperature.

[34][Lima et.al] have studied characterization and processing of Al_2O_3 and ytrrium aluminium garnet powder, Author obtained the complete YAG formation at 1400 °C, used XRD, TEM, and SEM characterization techniques for the morphology and microstructure study, Author conclude the formation of YAG obtained at 1400 °C rather the conventional 1600°C and pellets are prepared through powder metallurgy route.

[35][Tartaj et al] have studied continuous procedure for the preparation of homogeneous iron oxide doped alumina spherical particles is described. The method was based on the hydrolysis with ammonium hydroxide of liquid aerosols formed by spraying iron and aluminum nitrate aqueous solutions. Author found that the temperature of such a transformation is lowered due to increase in iron oxide content. Densification studies show the presence of iron oxide has a good effect on the sintering behavior of compacted powders. Thus un-doped samples did not achieve full density even after heating for 2 h at temperatures as high as 1500°C.

[36][Shamsuddin et al] have studied on fabricating and characterizing composites of iron-chromium alloy reinforced with 5–25 wt. % of alumina particles fabricated using powder

metallurgy method. The diffraction patterns of XRD reveal the influence of varying weight percentage of alumina. Comparisons on the mechanical properties are also being made on the unreinforced iron matrix (0 wt. %). Homogenous distribution of particles of alumina has obtained from microstructure which indicates the compatibility between reinforcement and matrix. Density, Porosity and Micro-hardness were measured using Archimedean testing and Vickers hardness tester respectively. Highest hardness obtained at 20 wt% of alumina. The composites were prepared by powder metallurgy route.SEM and XRD were used for microstructure and morphology respectively. Hardness, density, porosity and shrinkage were affected by varying the weight percent of alumina particles.

[37][She et al]....have studied the Submicron silicon carbide (SiC) was sintered to about 98% of its theoretical density by using alumina and yttria as sintering additives.Formed eutectic liquid by LPS between Al2O3 andY2O3 at sintering temperatures. Highly inter-granular fracture behaviour and microstructure studies by SEM. Author explained the effect of residual tensile stress from thermal expansion mismatch at primary and secondary phase. The volatilization of liquid phases at sintering temperature may also cause a decrease in sintered density. An increased amount of sintering additives may favour both the evaporation of liquid phases and the reactions between SiC and additives, leading to incomplete densification. Due to decrease in grain size hardness increased i.e. with decreasing sintering temperature as well as hardness decreased with increases the amount of binders fraction of grain boundary phases, having lower hardness than SiC grains.

[38][Tomilina et al]…have studied strong porous ceramic based on silicon carbide has been obtained at a diminished roasting temperature. The effect of the roasting temperature, the amount of the binder, and the grain size of the filler on the ceramic and filtering properties of the material is described. The ceramic can be used as a base for filtering elements. Author observed that the mean density changed insignificantly with the variation of the roasting temperature. The disperse silicon carbide oxidized and formed active silica that reinforced the composition quite considerably.

[39]*[Pribytkov et al]* have studied volume changes during solid phase sintering and the structure and phase composition of materials sintered of the binary and ternary powder foundry alloys of the Al–Cr(Si,Ti) system are investigated. The powders for sintering were obtained by self

propagating high temperature synthesis (SHS) in the mixtures of elementary powders of four target compositions with the subsequent crashing of the cakes and sieving of fine fractions. When sintering all the pressings under study, volume shrinkage, which monotonically increased with an temperature rise and the duration of the isothermal holding, take place. The results of structural investigations of the sintered materials, which were performed applying the methods of X-ray structural analysis, electron probe microanalysis, optical and scanning microscopy, are discussed jointly with the results of investigations of the volume variations during sintering. Author concluded that the rather dense materials of all studied compositions can be obtained by the solid phase sintering of the binary and ternary powder SHS foundry alloys Al– Cr(Si, Ti). To decrease the residual porosity, the compaction pressures of powder performs ≥800 MPa and the duration of the isothermal holding during sintering ≥8 h at temperatures close to the melting point are required. It is difficult to determine the real values of the final porosity of the sintered materials.

[40][Nepomnyashchii et al] have studied magnetic abrasive powders , prepared by mechanical mixing of starting components in the presence of surfactants and adhesive materials. It is shown that with respect to operating properties the abrasive powders prepared are on a par with known powders prepared by spraying, sintering, grinding, and other known methods. A processing scheme for forming mechanical mixtures using SAS and organic adhesive substances provides quite a high level of operating properties and is not limited to choice of system components. SAS and organic adhesive materials fasten abrasive particles to a ferromagnetic base due to a chemisorption bond. During MAT magnetic frictional and absorption forces arise, which facilitate fastening of abrasive to a matrix surface and a surface being treated, and provide a high level of MAM operating properties.

[41][Ashuri et al]…have studied characterization of severely deformed new composites fabricated by powder metallurgy including a stage of mechanical alloying formed a new composites having a binary matrix of Al–4Cu reinforced with TiO2 nano-particles were investigated. Composite was formed by using 2 wt% and 8 wt% of TiO2 reinforcement particles used powder metallurgy and mechanical alloying fabrication. Powders are mixed by ball milling techniques and morphology and phases were studied during densification .mechanical properties enhanced due to increase in reinforcement particle. There is drastic in micro structural evolution

and mechanical properties changes after the application of twist and severe plastic deformation process also studied. due to increase in twist extrusion led to increase in high strength as well as high yield strength and also led to development of ultrafine grained nano-structure and distribution of more homogenous of the reinforcement particles within the structure, The maximum allowable passes of extrusion was four, beyond which the materials did not endure plastic deformation and failed.

LITERATURE GAP

In previous research, researchers had not much work on sintered abrasive magnetic powder, especially with carbonyl iron powder .In this research green compact formed without using any binder were investigated as carbonyl iron powder also act as good additives with silicon carbide.

CHAPTER 3

SCOPE OF THE RESEARCH

This research "Analysis and characterization of iron dopes sintered abrasive magnetic powder without binders through powder metallurgy route". The powder manufactured at three different pressures and on increasing sintering temperature have application in magnetorheological finishing (MRF), The sintered powder can be used as tool for MRF, In past recent years MRF gained interest of many researchers, There is need to develop a good abrasive, which can make a good abrasive bond and the finishing should be accurate. Formerly Silicon carbide was used but with iron particle not with carbonyl iron powder. The sintered magnetic abrasive particles can be used as tool in abrasive machining, which can increase the efficiency of machining as compared to its conventional abrasive bond. Sometimes in rheological machining abrasive bond is made from other abrasive material did not homogeneously mixed with the gel or the other pastes ,due to insolubility of abrasive particles in rheological fluid. And can be used for surface finishing in micro level more accurate finishing. Nowadays surface finishing up-to Nano level is a challenging task and need appropriate magnetic abrasive for better finishing. As conventional methods were not able to satisfy the quality requirement.

CHAPTER 4

OBJECTIVE OF THE REASEARCH

This research aimed to produce a sintered abrasive magnetic powder ,which possess better abrasive properties as well as magnetic properties. The main objectives during the research are as follows

1) To optimize the mixing ration of abrasive powder with magnetic powder.
2) To optimize the holding time without binders , so that the green compact cannot deform from its shape or break.
3) To characterize density of sintered specimen
4) To study the behaviour of its morphology due the effect of sintering temperature and pressure.
5) To study the mechanism of liquid phase sintering (LPS).
6) To study the working principle of characterization techniques i.e. scanning electron microscopy (SEM),Transmission electron microscopy (TEM).
7) To study the particle size, grain Size. geometry of the particles and optimize their diffraction pattern.
8) To study the composition variation after sintering by using Electron dispersive spectrum.

CHAPTER 5

MATERIALS

5.1 INTRODUCTION

In this research work of characterization and synthesis of abrasive magnetic sintered powder ,abrasive powder used is Silicon Carbide and magnetic powder carbonyl iron powder are used, due to their properties of magnetic and abrasive and the description of their properties as follows:

5.2 SILICON CARBIDE (SiC)

The only stable compound at atmospheric pressure in the Si-C equilibrium system is silicon carbide.it was early observed by jons Berzelious in 1824, Since the properties and potential of the material was not thoroughly understood at that time. Around 1885 Eugene Acheson introduced the growth of polycrystalline Sic with an electric furnace. He was the one to recognize it as a silicide of carbon and gave it the chemical formula SiC. Naturally occurrence of SiC is only found in meteroites that's why SiC must be manufactured with elaborate furnace techniques as it cannot be mined. Silicon carbide is also knowns as carborundum, it is a compound of silicon and carbon represented by chemical formula SiC. Occurance is naturally in the form of extremely rare mineral moissanite.it has been mass produced since 1893 used as an abrasive.BY sintering process grains of SiC can be bonded together to form a very hard ceramic, which are widely used in many applications required high endurance, for example car brakes, clutches and ceramic plates in bulletproof jackets.

Silicon carbide exists in about 250 crystalline forms, The polytropes are defined as the large family of similar crystalline structure characterized by the polymorphism of SiC . They are variations of the same chemical compound that are identical in two dimensions and differ in the third. Thus, they can be viewed as layers stacked in a certain sequence

Silicon carbide have polytropes like α-SiC and β-SiC. Alpha silicon carbide is the most common polymorph, Formed at temperatures greater than 1700 °C and has a hexagonal crystal structure. The beta silicon carbide with a zinc blende crystal structure similar to diamond formed at temperature below 1700 °C. α-SiC have wide application areas as compare to β-SiC. β-SiC have higher surface area than α-SiC due to which there is increasing interest in its use as a support for

hetero-genous catalyst. Pure SiC is colourless. Due to presence of iron impurities the colour turned from brown to black in industrial products. Due to the passivation layer of silicon di oxide which forms at the surface results the formation of rainbow like luster of the crystals. Due to its high melting point or high sublimation temperature approximately 2700 °C makes it useful for bearings and furnace parts. Silicon carbide does not melt at any known pressure. It is also highly inert chemically.it has very low coefficient of thermal expansion due to which no phase transition occurs and causes discontinuities in thermal expansions .In the arts, silicon carbide is a popular abrasive in modern lapidary due to the durability and low cost of the material. In manufacturing, it is used for its hardness in abrasive machining processes such as grinding, honing, water-jet cutting and sandblasting. Particles of silicon carbide are laminated to paper to create sandpapers and the grip tape on skateboards. In 1982 an exceptionally strong composite of aluminium oxide and silicon carbide whiskers was discovered. Development of this laboratory-produced composite to a commercial product took only three years.

Table 2 Properties of Silicon Carbide

Physical/Mechanical	**SI Units**	**Values**
Density	g/m^3	3.21
Porosity	%	0
Color	-	Black
Flexural strength	Mpa	550
Elastic Modulus	Gpa	410
Bulk modulus	Gpa	220
Poission'ratio	-	.14
Compressive strength	Mpa	3900
Hardness	Kg/mm2	2800
Fracture toughness	Mpa $m^{1/2}$	4.6
Melting temperature	°C	2072
THERMAL		
Thermal conductivity	Wm/°K	120
Coefficient of thermal expansion	10^{-6}/°K	4
Specific heat	J/Kg °K	750
Electrical		
Volume resistivity	Ohm cm	10^2-10^6

Table 3 :Properties of major SiC polytropes

Polytype	**3C (β)**	**4H**	**6H (α)**
Crystal structure	Zinc blende (cubic)	Hexagonal	Hexagonal
Space group	T_{2d}-F43m	C_{6v}^4-P63mc	C_{6v}^4-P63mc
Pearson symbol	cF8	hP8	hP12
Lattice constants (Å)	4.3596	3.0730; 10.053	3.0810; 15.12
Density (g/cm3)	3.21	3.21	3.21
Bandgap (eV)	2.36	3.23	3.05
Bulk modulus (GPa)	250	220	220
Thermal conductivity (W cm−1K−1)	3.6	3.7	3.9

Table 4 Mechanical properties of LPS-SiC as a function of different sintering aids.

Reference	**Technique**	**Sintering additives**	**Hardness(GPa)**	**Fracture toughness**	**Density**
Chen et.al	Pressure-less sintering SiC	Al2O3 + HoO3	17.47	3.68	Up to 3.764 g/cm3
Chen et.al	Pressure-less sintering SiC	Al2O3 + SmO3	17.1	4.6	92.6%
Hidaka et Al	Hot pressing at 1950°C and P=39 MPa	(Al2O3 + Y3+ ions)	19 - 21	5.9	95-98%
Scitti et al	Hot pressing at 1850-1950°C	Al2O3 + Y2O3	22	2.95 – 3.17	3.24 g/cm^3
Wang et .al	Pressure-less sintering β-SiC at 1850°C	Y2O3 in (Al2O3 + Y2O3)	22	4.3	98%
Mulla et.al	Pressure-less sintering β-SiC at 2050°C	Al2O3		6	97-98%

5.3CARBONYL IRON POWDER

Carbonyl iron is a highly pure (97.5% for grade S, 99.5+% for grade R) iron, prepared by chemical decomposition of purified iron penta carbonyl ,It usually has the appearance of grey powder, composed of spherical micro-particles. Most of the impurities are carbon, oxygen, and nitrogen .

In electronics, carbonyl iron is used to manufacture magnetic cores for high-frequency coils, and in production of some ferrites. Spherical particles manufactured of carbonyl iron are used as a component of the radar absorbing materials used in military, in stealth vehicles for example. Other uses are in powder metallurgy, metal injection molded parts, and in various specialty products.

Powdered cores made of carbonyl iron have high stability of parameters across a wide range of temperatures and magnetic flux levels, with excellent Q factors between 50 kHz and 200 MHz. A popular application is in broadband inductors, especially in high-power applications. Particles of carbonyl iron (20-40%) suspended in a carrier fluid (60-80%) are used as a magnetorheological fluid.

The molecular formula of carbonyl iron is $Fe(Co)_{5,}$ Manufactured using the carbonyl decomposition process. Uniform microscopic spheres with only traces of carbon, oxygen and nitrogen . High purity with superior electromagnetic properties .

Table 5 :Properties

S.No	PROPERTIES	SI/METRIC
1)	Atomic number	26
2)	Molecular weight	195.9 g/mol
3)	Density	7.87g/cm^3
4)	Specific heat	12 cal/g- °C
5)	Melting point	1536 °C
6)	Boiling point	217 °F
7)	Thermal conductivity	.12 cal/s-°C
8)	Brinell hardness	82-100
9)	Solubility in water	Insoluble
10)	Crystallography	Cubic structured, body centred

CHAPTER 6

REASEARCH METHODOLOGY AND EXPERIMENTAL PROCEDURES

6.1 INTRODUCTION

This chapter explained the experimental procedures followed during the "Analysis and characterization of iron doped sintered abrasive magnetic powders without binders through powder metallurgy route". For processing, microstructure determination, characterization, and testing procedures standard techniques were used. For the preparation of powders,ball milling and particle size analysis were used. Characterization techniques included scanning electron microscopy(SEM),transmission electron microscopy(TEM) were used. Micro-hardness was determined by indentation tests.

6.2 STARTING MATERIALS

Silicon carbide

In this research work of "Analysis and characterization of iron doped sintered abrasive magnetic powders without binders through powder metallurgy route" silicon carbide was used as abrasive and carbonyl iron powder was used as magnetic powder. Silicon carbide 400 mesh size was used and the purity was 99%.The functionalized silicon carbide was characterized with SEM, TEM.

Carbonyl Iron Powder

Carbonyl iron powder (manufacturer BASF) of soft grade was used. contains 99.5% of iron particles or Fe ,.03,.01,..1-.25 of carbon, nitrogen and oxygen respectively. The ferromagnetic properties of CIP were very good.

Fig 13 Carbonyl iron powder at electronic weighning machine

6.3POWDER PROCESSING

Initial powder silicon carbide and carbonyl iron powder were taken uniformly and mixed homogenously manually. After mixing manually the mixed powder was milled through ball milled contains tungsten balls .milled for 2 hours. In most cases, two batches for each mixture were made: one bottle was ball milled dry; the other ball milled in ethanol (hereafter termed "wet" milled). When soft agglomerates were formed, they were broken up by mortar and pestle prior to sintering. Four powder samples were prepared through ball milling.

Fig 14 Ball mill with tungsten carbide ball for mixing of powders homogenously

6.4COMPACTION OF POWDER

The powder was compacted due to the application of high pressure, the hydraulic press was used .The die was in cylindrical shape, having dimension of internal diameter of 30mm while the external diameter was of 40mm.The maximum load capacity of the hydraulic press was 15 ton, The milled powder was placed in a die and the pressure was applied to form a green compact. In this research work the specimens were prepared at three different pressures. Primarily the powder was placed in die and pressure was applied and the first pressure value was 5 ton ,again the powder was placed in die and the value of pressure was increased by 2 ton i.e.7 ton and finally the value of pressure was increased by 2 to i.e. 9 ton. Three green compacted specimens were prepared.

Fig 15 Manually operated hydraulic press

6.5 SINTERING

Sintering time, temperature, atmosphere and heating rate are important variables, which need to be optimized for achieving maximum densification with improved physical and mechanical properties. Usually at higher sintering temperatures, faster and irregular grain growth occurs. At lower sintering temperatures and lesser sintering times, more number of pores exists at the grain boundary, which impede the grain boundary movement and hence, grain growth. After the

compression, the green compact was taken and hold for some time ,holding time is one the parameter. The parameters were temperature, pressure, holding time etc.Liquid phase sintering was done, Liquid phase sintering (LPS) involves formation of the liquid phase either prior to or at the sintering temperature of the powder. The liquid phase is typically formed due to oxide layer present on the surface of the powder particles or by reaction of powders with the sintering atmosphere. The molten liquid phase penetrates the particle boundary and surrounds them. The rapid mass transport due to increased diffusion in the presence of liquid phase leads to the achievement of high compact densities in difficult-to-sinter materials.

The green compact was allowed to sinter in tubular furnace in a controlled argon atmosphere with temperature increasing 5°C per minute reaches a temperature 700 °C and hold one hour for complete degasification. Then with the same rate the temperature was increased up-to 1200 °C and holds for three hours at this temperature for getting complete phase transformation. After the completion of process the cooling of furnace was done in argon atmosphere upto the temperature cools down to 25 °C. The sintered products were then crushed in ball mill with tungsten carbide balls to obtain the sintered magnetic abrasives. Hence sintered magnetic abrasives composed of ferromagnetic phase and abrasive phase which are combined firmly

6.6 DENSITY CALCULATION

Apparent Density

The apparent densities of the samples were measured by dividing the sample mass by the sample volume. The sample volume was calculated from dimensions measured .The mass was acquired by weighing the sample. Apparent Density for all the samples were measured by this method.

Archimedes Method

The absolute densities of the samples were assessed by the Archimedes method, The Archimedes method was performed by placing the sample disks in a drying furnace at ~120°C for 24 hours, followed by measurement of the dry weight. Next, the samples were submerged in deionized water, suspended from metal mesh to secure samples and prevent cracking, and boiled for 5 hours. The samples were again weighed while submerged in deionized water. Finally, the saturated samples were removed from water, brushed with a damp cloth to remove surface water,

and weighed for the saturation 35 weight. The pore volume, the geometric volume, and the percent porosity were calculated according to Equation

$$\text{Volume}_{pores} = \frac{\text{weight}_{saturated} - \text{weight}_{dry}}{\text{Density}_{Fluid}}$$

$$\text{Volume}_{geometric} = \frac{\text{Weight}_{saturate} - \text{weight}_{submerged}}{\text{Density}_{Fluid}}$$

$$\%\text{Porosity} = \frac{\text{Volume}_{pores}}{\text{Volume}_{geometric}}$$

6.7 MICRO HARDNESS

Microindentation was performed on vickers hardness tester .In Vickers test, the load is applied smoothly, without impact, and held in place for 10 or 15 seconds. The physical quality of the indenter and the accuracy of the applied load (defined in E 384) must be controlled to get the correct results. After the load is removed, the two impression diagonals are measured, usually with a filar micrometer, to the nearest 0.1 μm, and then averaged. The Vickers hardness (HV) is calculated by: HV = 1854.4L / d2 where the load L is in grams-force and the average diagonal d is in μm (although the hardness number units are expressed in units of kgf / mm2 rather than the equivalent gf / μm2)

Fig 16 Vickers hardness tester

BALL MILLING CRUSHING

After micro indentation and density measurement the solid sintered specimen was again placed in ball mill for crushing.

6.8 SCANNING ELECTRON MICROSCOPY(SEM)

Characterization and morphology of the crushed powder sample was performed by Scanning electron microscopy conducted on (SEM model LEO 435V.Typical microscope setting was used for sintered powder samples.. sintered samples were 15Ev,and all the images were taken at different magnification.

Fig 17: Scanning Electron Microscopy (SEM)

SAMPLE PREPARATION FOR SEM

Sample preparation is very important step to obtain the desired results. since the specimens used were in the powder forms , so there was need to prepare the sample for SEM. Samples were prepared by following method .A small amount of powder was added in ethanol solvent then a small amount of suspension was taken from the suspension and was dropped on the carbon tape stick on the stub, And kept for 10-15 minutes under infrared lamp for drying. The reason to use carbon tape as it makes a good adhesive base for the powders, sprinkle the powder lightly with a spatula, press lightly to seat and to remove loose material fromm the top surface of the powder spray it with canned air.

6.9 TRANSMISSION ELECTRON MICROSCOPY (TEM)

Powder characterization to a very high level was done by using transmission electron microscopy in central instrumentation facility IIT ROORKE on Tecnai G2 20 S-TWIN FEI Netherland.it offers very high performance for characterization upto nano level and it includes STEM,CCD cameras, EDX and energy filters. Sample preparation was most important task which was to be done carefully .

Fig 18 Transmission Electron Microscopy (TEM)

SAMPLE PREPARATION FOR TEM

The grids stored for tem were kept in blue boxes will be using dark side place TEM stub on sticky dark side and the sample , the grid should be kept in container and the sample was placed on stub, then stirrer water was added and allowed to set for a specified amount of time, Filter paper used whip the water from the grid and again the water was added after whipping by filter paper, After that the sample was taken and placed in grid box carefully as to avoid the bending of tip of the specimen.

TEM IMAGING

Transmission electron microscopy imaging was carried out with Tecnai (G2 20 S-TWIN FEI Netherland).The accelerating voltage was 100Ev.Spot size generally at 1 or 2 ,and the alpha setting was 2 or 3,depending on the type of magnification. A condenser aperture of 100μm was employed to sharpen the beam .Image contrast and selected area diffraction patterns were obtained with the aid of the objective aperture. Both single and double tilt specimens holders allowed one to obtain bright field images and diffraction patterns respectively. Generally ,the selected area aperture singled out a particular grain in the poly-crystal so as to obtain a diffraction pattern composed of discrete spots, as opposed to rings.

6.10 DIFFRACTION PATTERN ANALYSIS

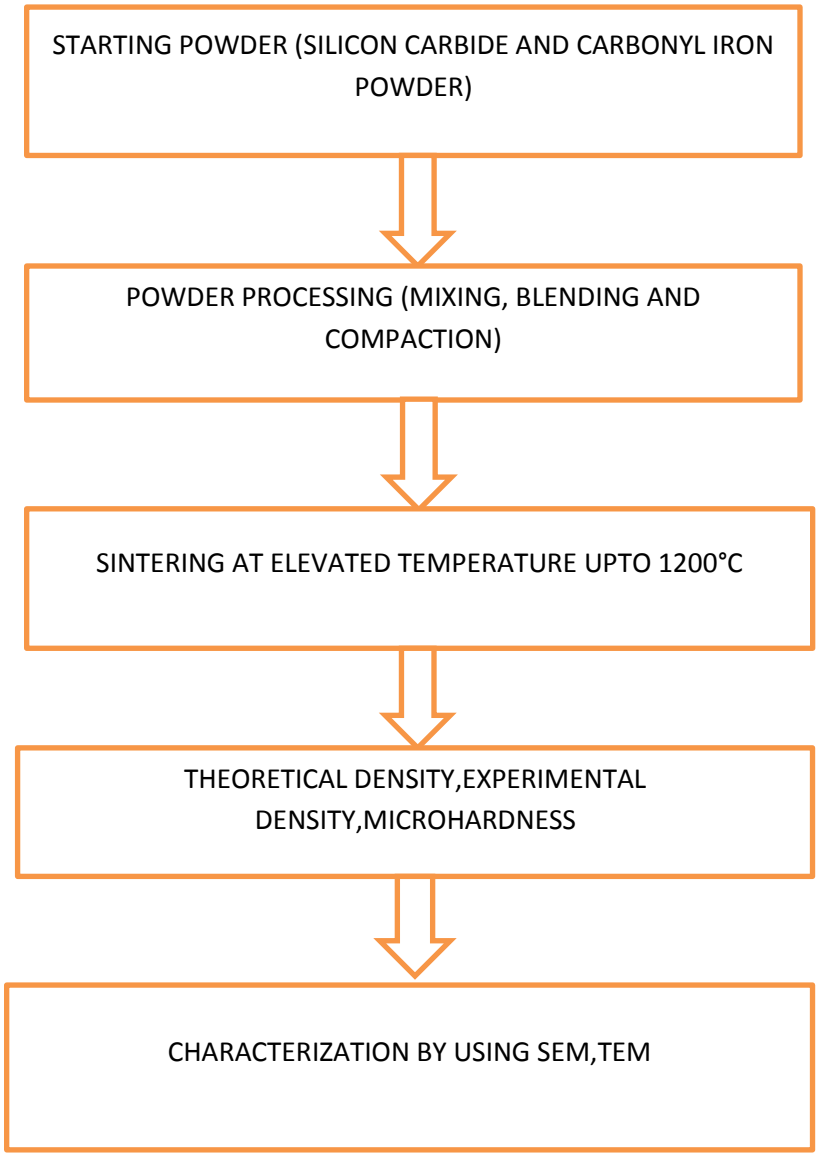
Diffraction patterns obtained during TEM imaging enabled the determination of composition and phase of the sample material. The *d*-spacing for the material was determined by measuring the radial distance between diffraction spots, or the radius of the diffraction rings. The relevant equation for such analysis is shown below.

$\pi L = rd$

Where λ is the wavelength of the electron, *L* is the camera length, *r* is the radius of the diffraction ring, and *d* is related to the lattice constant.

Selected area diffraction (SAD) is a TEM technique to obtain diffraction patterns that result from the electron beam scattered by the sample lattice. Obeying the bragg's law, the electrons are scattered elastically by the lattice, therefore, we can index the diffraction spots in the pattern and identify the phases in the sample and study their structures. Typically, the area size of the sample been selected by SAD aperture is 0.5-1 μm, so the microstructure of the sample could be fine selected and tested.

6.11 FLOW CHART OF THE METHODOLOGY



CHAPTER 7

RESULTS AND DISCUSSION

7.1 INTRODUCTION

This chapter described the results investigated from the research , The result parameter described step by step.

7.2 DENSITY

1) ***Theoretical Density***

Theoretical density was calculated by using mass/volume formula ,as the initial powder was taken 10gm of abrasive and 10gm of magnetic ,but after the completion of all the processes the mass of sintered specimen remains 17.8gm ,18.2gm and 18.5 gm for 5ton,7tonand 9ton specimens respectively .The volume of the cylindrical specimen calculated by using ,volume of cylinder. The graph shown below representing the densities as the compaction load increases the densities increases exponentially.

Table 6: Theoretical density at5, 7,9 ton specimens respectively.

S.no	Compaction load(Kg-f)	Density ($gmcm^{-3}$)
1	5 ton	2.51
2	7 ton	2.59
3	9 ton	2.62

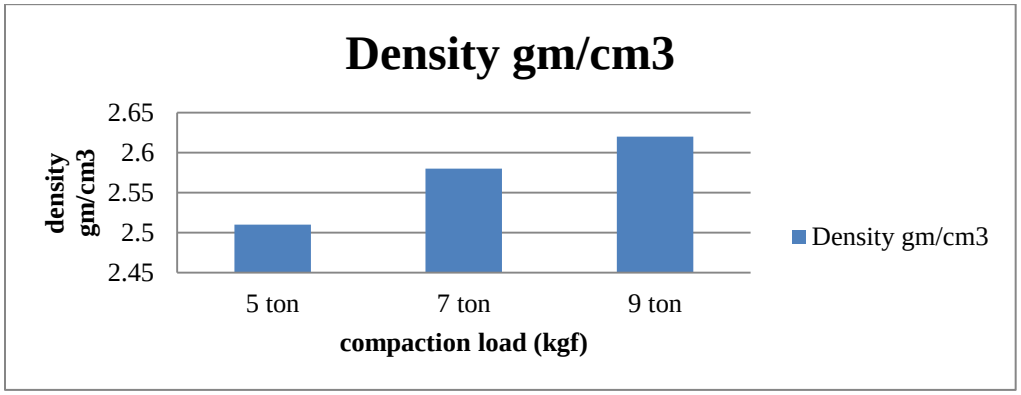


Fig 19:Graph of Theoretical density

2) **Density calculated by Archimedes principle:** Experimental densities were calculated by using Archimedes principle of water immersion method , The result shows the densities increases with increasing compaction load at constant temperature.

Table 7: Experimental Density at 5,7,9 ton respectively

S.No	Compaction load (Kgf)	Density($gmcm^{-3}$)
1	5 ton	3.42
2	7 ton	3.47
3	9 ton	3.53

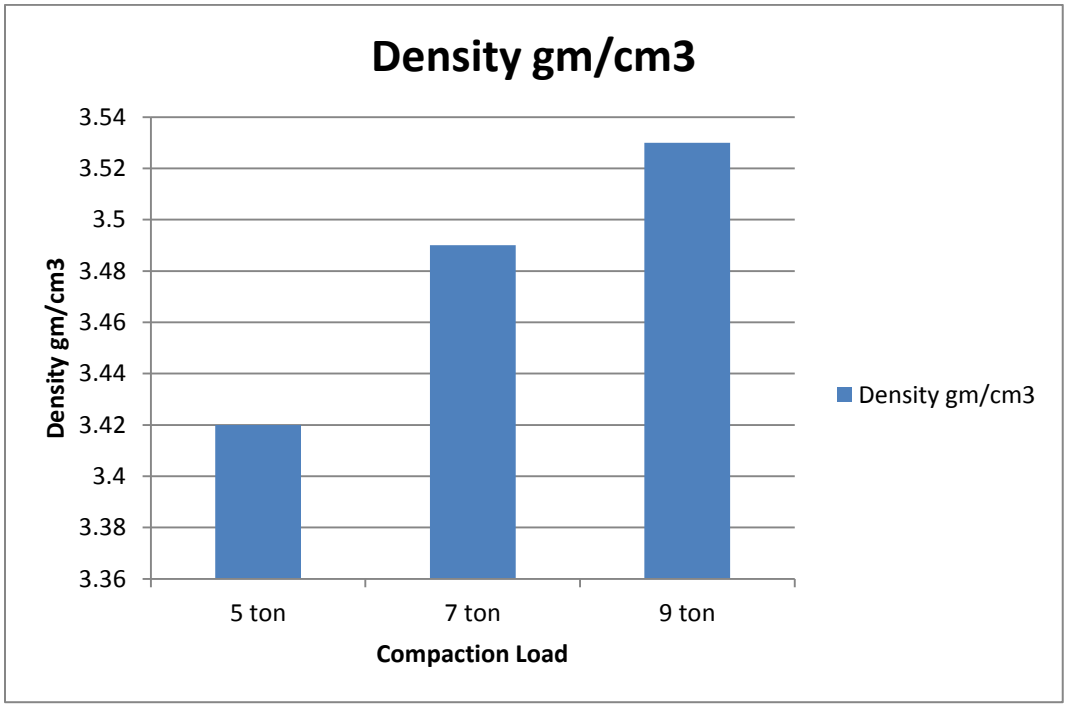


Fig 20: Graph showing the experimental density

3) **Comparison between theoretical and experimental density**

The below graph shows the comparison between the theoretical density and experimental densit , As there was much variation between both the values. Experimental densities comes nearer to the abrasive and magnetic powders densities.

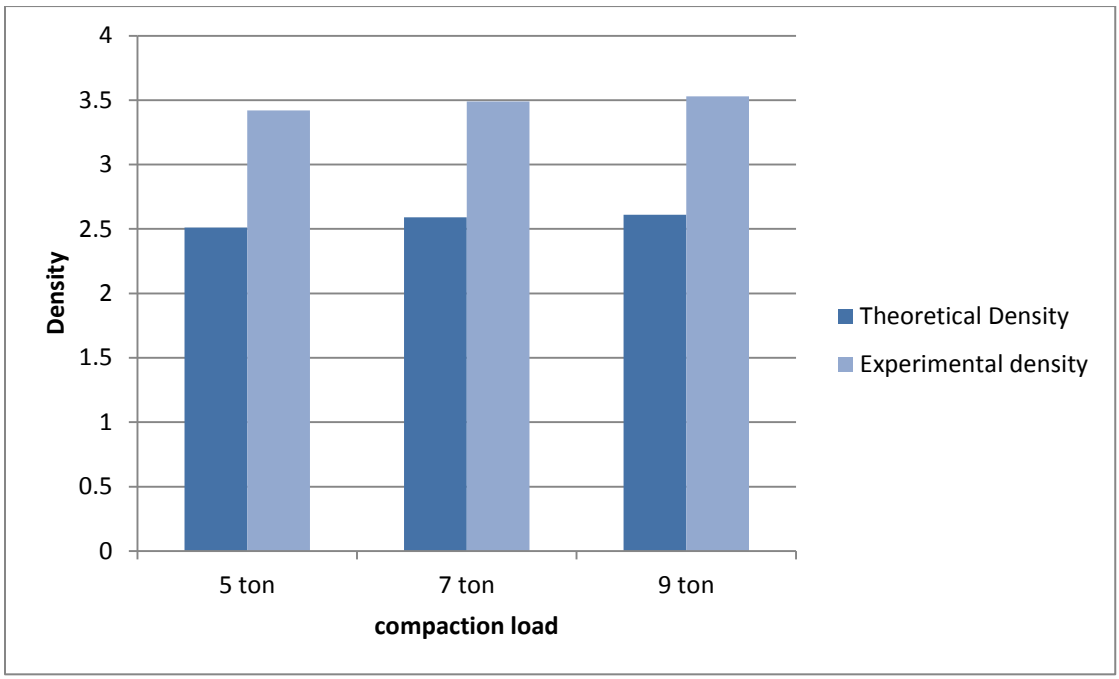


Fig 21 Graph comparision between experimental and theoretical density

7.3 MICRO-HARDNESS

Micro hardness was calculated at Vickers hardness tester of pyramid shape indentor at 10 kg applied. The specimen was cylindrical shape with 30mm diameter and 10 mm thickness respectively. The table shown below shows the value of Vickers hardness no, since there was exponential increase in the value of micro hardness. Micro hardness is directly propotional to the compaction load. The graph is plotted between compaction load and vickers hardness number.

Table 8 showing the value of micro hardness

S.no	Compaction load	Micro hardness(Kg-f)
1	5 ton	647
2	7 ton	672
3	9 ton	693

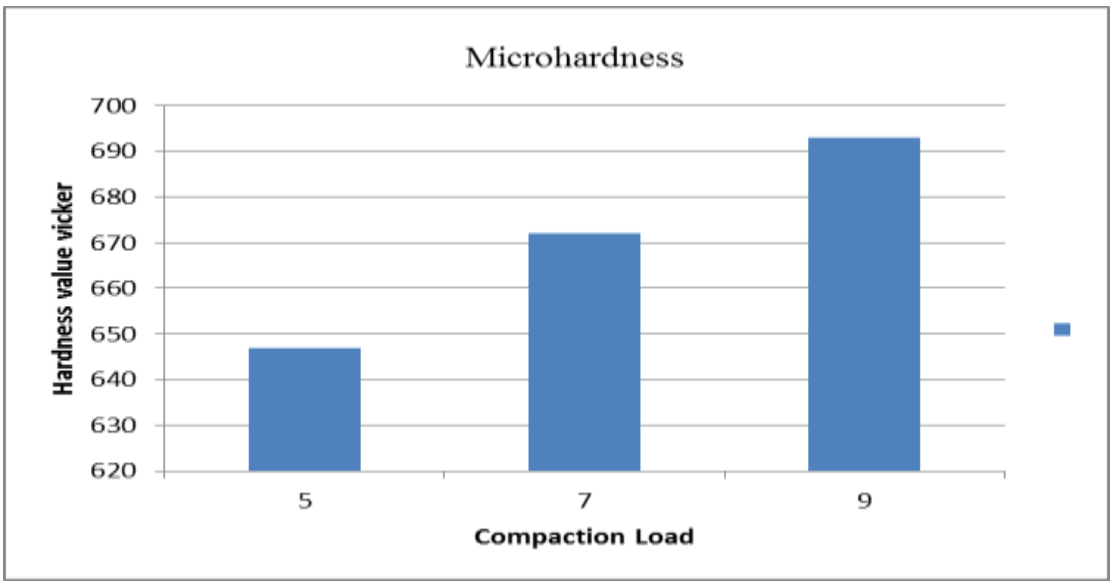


Fig 22: Graph plotted b/w Vickers hardness and compaction load

7.4 SCANNING ELECTRON MICROSCOPY (SEM)

Characterization of sintered abrasive magnetic was done by using scanning electron microscopy**.**

Silicon carbide

Morphology of silicon carbide

The SEM micrograph of as received silicon carbide ,The SEM topography of silicon carbide show that ,the sharp edges of silicon carbide can be seen of crystalline structure, the particle size was not uniform ranges from 4-12um respectively. The average particle diameter was estimated by drawing a line on the micrograph, counting the number of particles that were crossed, and dividing the number of crossings from the length of the line

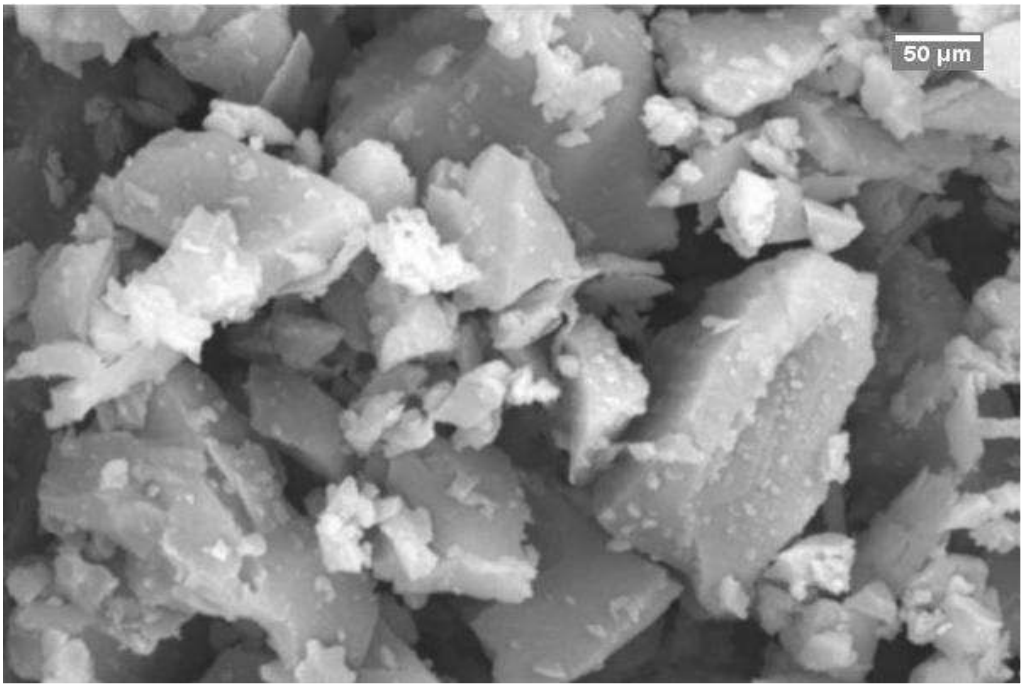


Fig 23:SEM micrograph of as-received SiC powder containing agglomerates.

CARBONYL IRON POWDER (CIP)

Characterization of carbonyl iron powder

Characterization of powders was done by using scanning electron microscopy (SEM). Spherical morphology revealed by SEM topography of CIP. It shows that the CIP particle size lies between 1µm to 4µm and maximum particles lies between 3µm to 4µm. The average particle diameter

was estimated by drawing a line on the micrograph, counting the number of particles that were crossed, and dividing the number of crossings from the length of the line

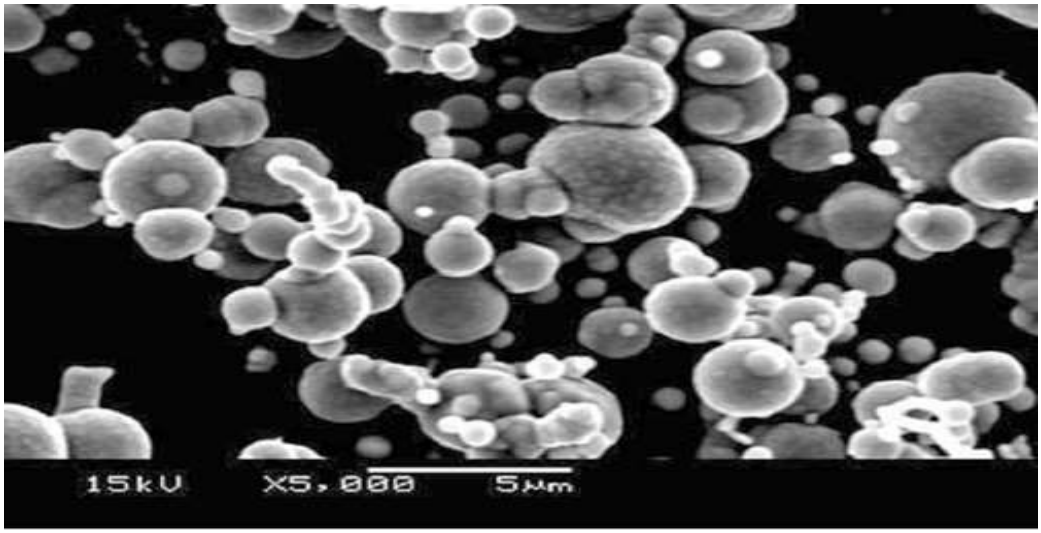


Fig24:SEM micrograph of carbonyl iron powder pure

Morphology of un-bonded abrasive magnetic

The morphology of un-bonded abrasive magnetic has been studied using SEM,The SEM micrograph shown below describing the sharp edges of abrasive can seen clearly that is of silicon carbide and the spherical morphology of carbonyl iron powder cab also clearly seen. The particle were not of uniform shape ,there were variation in the particle size, as the powder was milled thoroughly for homogeneous mixing and particle comes in uniform shape. The CIP particle were not fully diffused on the silicon carbide particles surface.

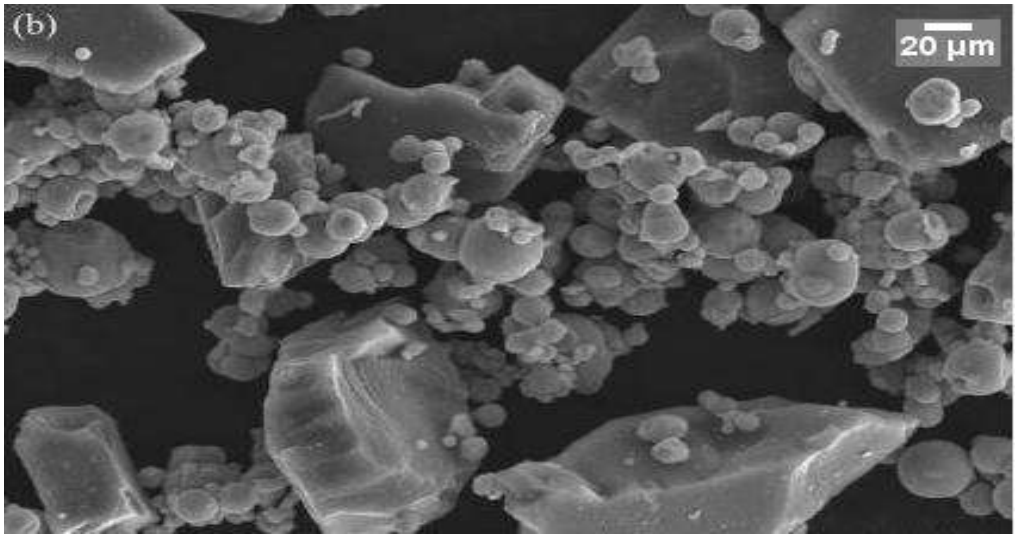


Fig 25: SEM micrograph of un-bonded abrasive magnetic shows the CIP and SiC particles clearly

SEM MICROGRAPHS OF SAMPLE 1

The SEM micrograph of first specimen compacted at 5 ton pressure and sintered at 1200 ®C , Micrographs taken at different magnification shows the particles of sintered abrasive magnetic powder. The carbonyl iron powder diffused at the surface of abrasive particle i.e. silicon carbide and formed abrasive magnetic powder. The particle size varies in size , non-uniform particles present at the surface. The size ranges from 1µm to 14µm respectively. The next image show the grain boundaries and edges of the particles ,analysed by the use of image j software, on the basis of analysis the total no particles present were 988,and diameter ranges from 14nm to 2132 nm respectively. As the compaction load was 5 ton so,the particles were not compacted closely. And due to crushing in ball mill the particle doesn't come in uniform particle size.

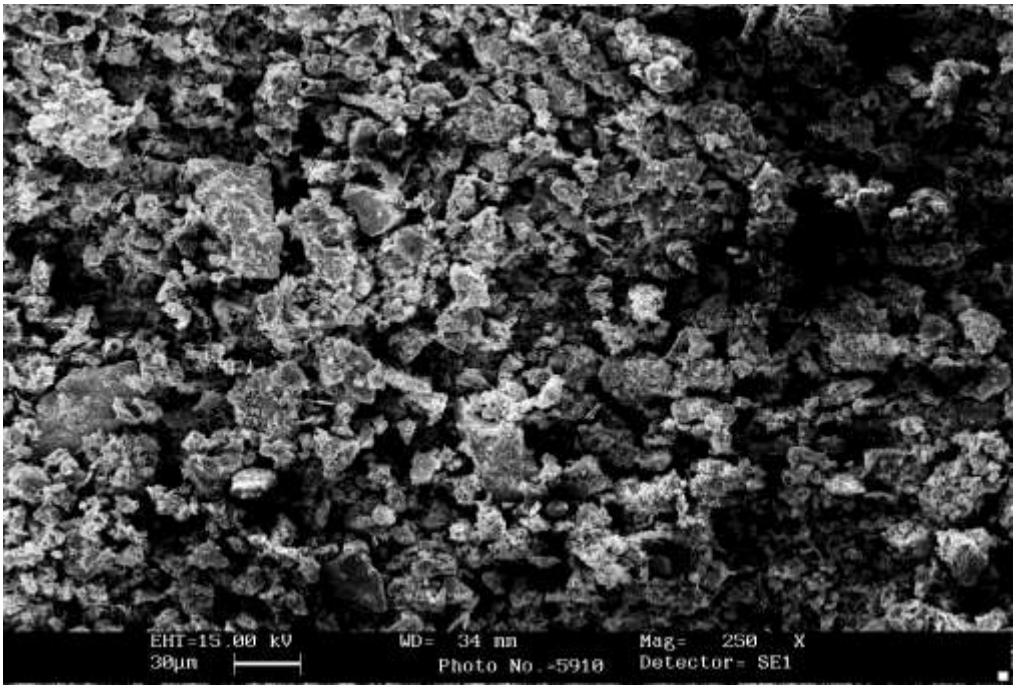


Fig 26:SEM micrograph at 5 ton compaction load showing the agglomerates

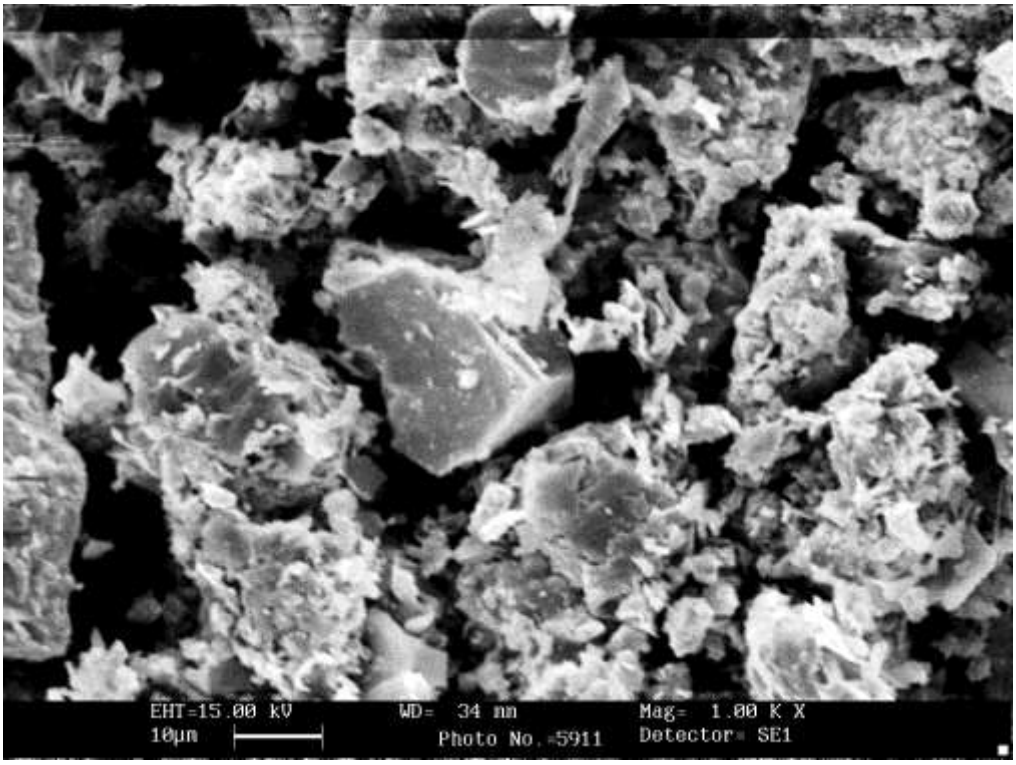


Fig 27: SEM micrograph clearing showing the abrasive and magnetic particles at higher magnification .

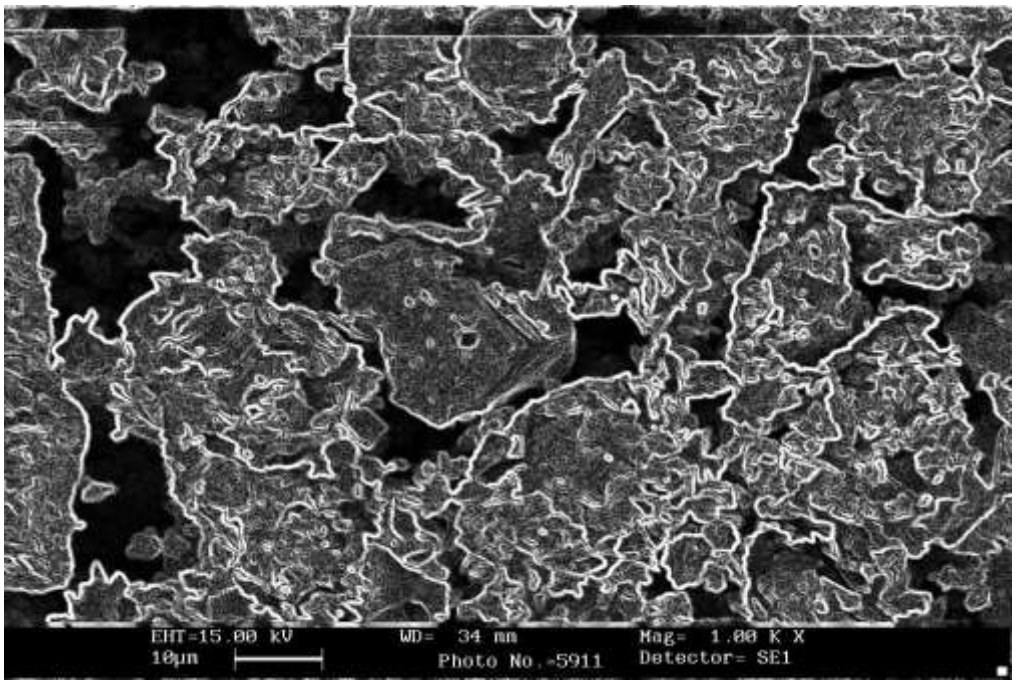


Fig 28 :SEM micrograph showing the grain boundaries and edges of sintered abrasive magnetic particles .

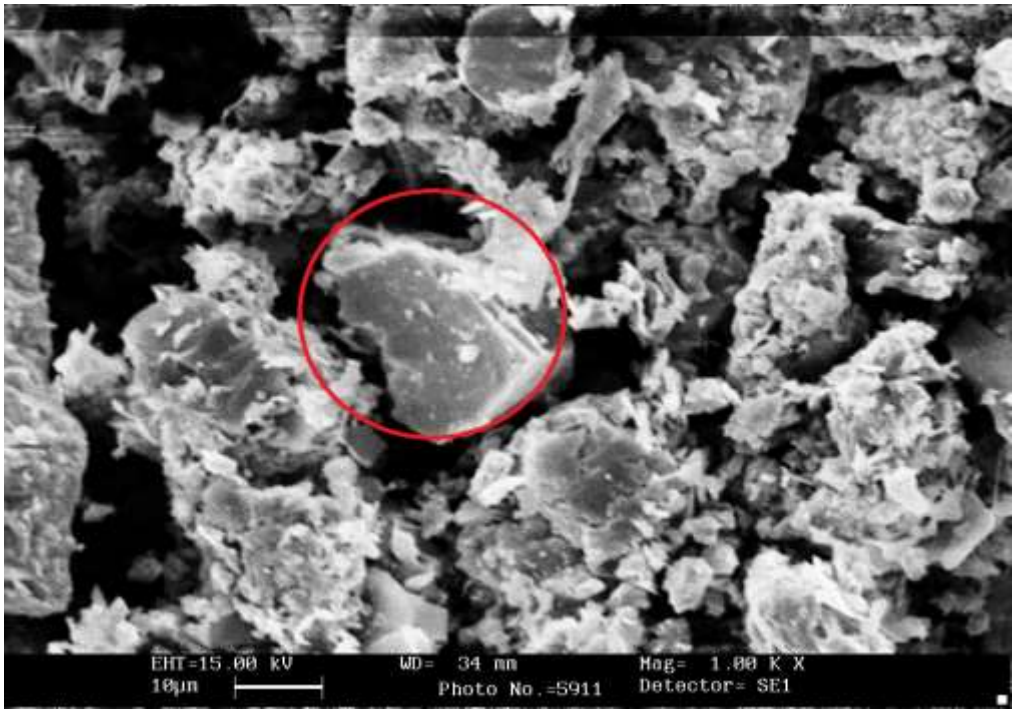


Fig 29:SEM micrograph at higher magnification sintered at 1200°C.

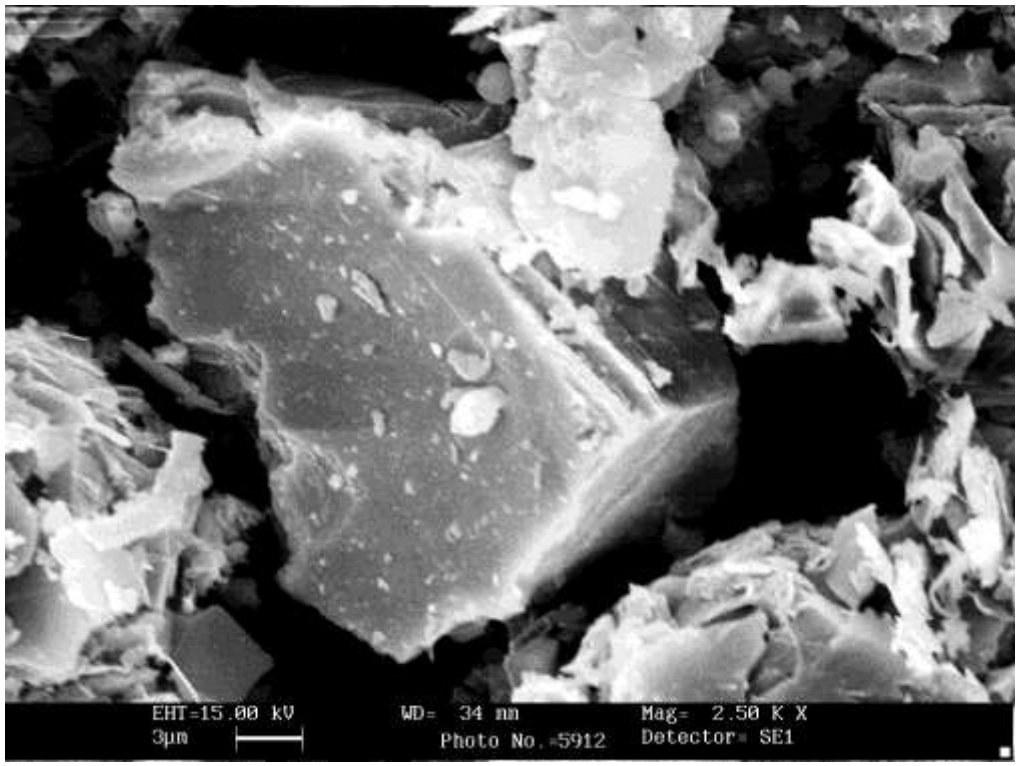


Fig 30:SEM micrograph at higher magnification. Shows the abrasive particle, and can be seen the sharp edges and the formation of fracture due milled at high speed on ball mill.

SEM MICROGRAPH OF SAMPLE 2

The SEM micrograph of Sintered abrasive magnetic was compacted at 7 ton pressure and sintered at elevated temperature upto 1200 °C in argon atmosphere. due to slowly increasing temperature the degasification takes places due to which the porosity of the grains decreases and increases the density, The phase transformed completely. The particle size, grain size measured with straight line method, since the pressure was very high ,so the magnetic particles diffused on the surface of abrasive and liquid phase sintering was used ,so the carbonyl iron powder itself act as binder as it melts which acts as a sintering additive for the bonding of silicon carbide .The particle size ranges from 2µm to 10µm respectively. The size of the particles was not uniform, In the next figure, SEM micrograph at higher magnification ,the red circle denoting the magnetic particles diffused at the abrasive particle, the topography of carbonyl iron particle was spherical, the particle size was 4µm.since the pressure was more than the pressure applied on the first pellet ,here it can be clearly seen the particles were uneven and the bond between the particle was very strong and compacted.

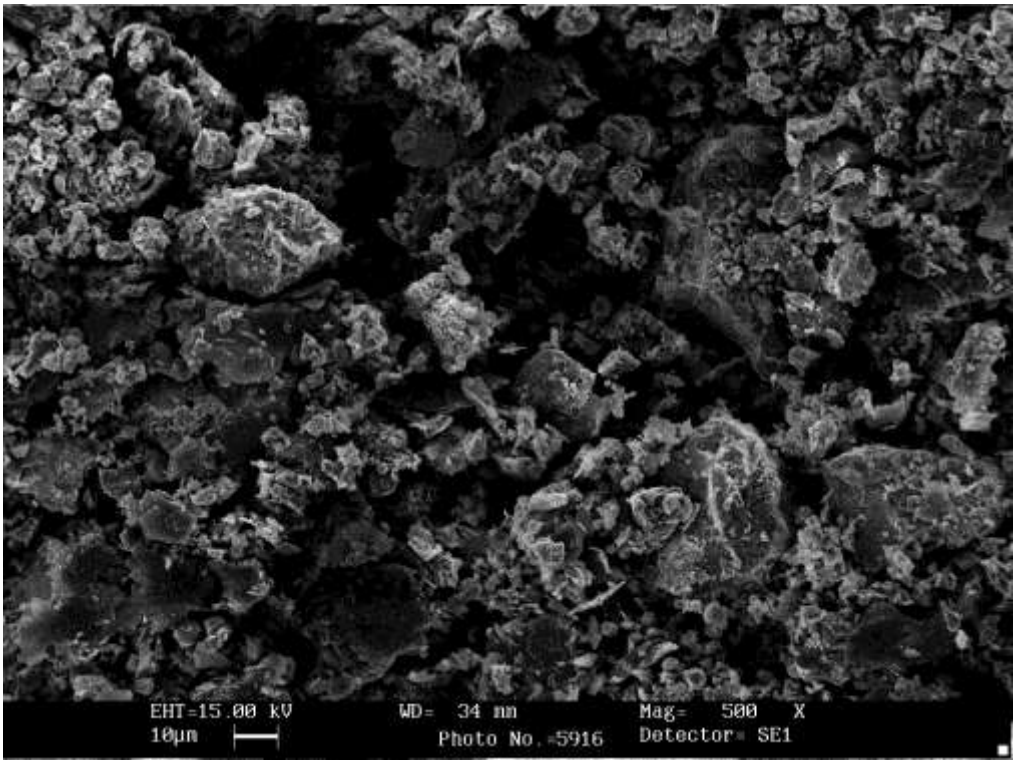


Fig 31 :SEM micrograph compacted at 7 ton pressure shows the compacted grains.

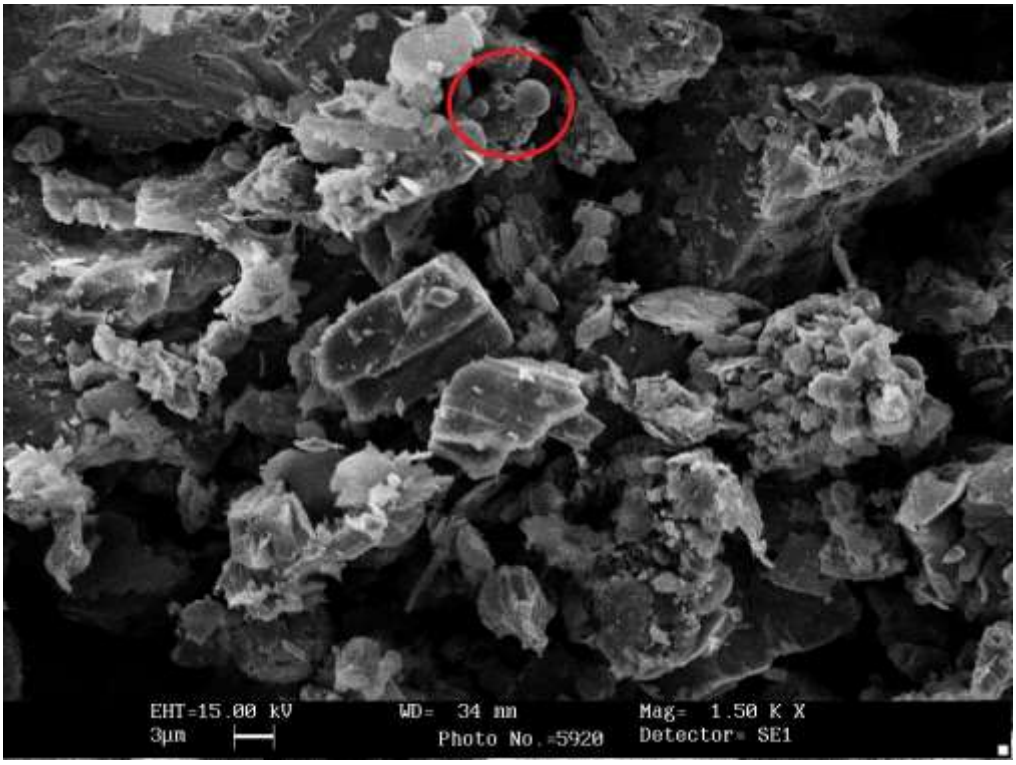


Fig 32: SEM micrograph showing the CIP grain diffused at the surface of SiC ,compacted very densely due to high pressure.

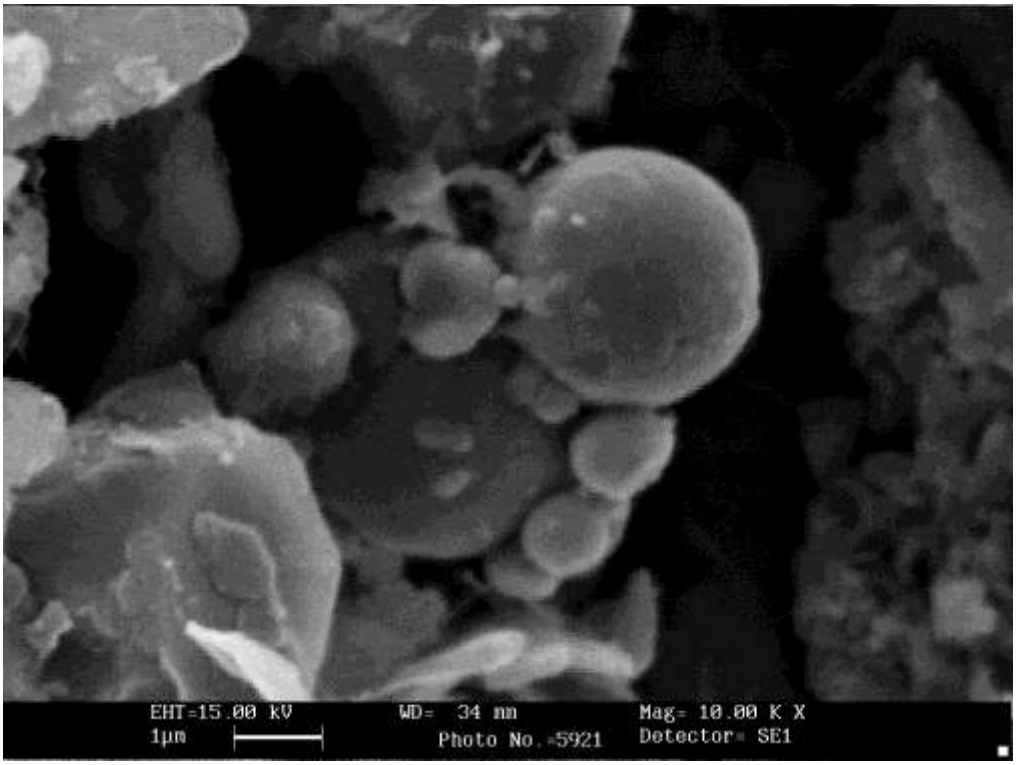


Fig 33: SEM micrograph showing the CIP particle at higher magnification, the topography can be clearly seen.

SEM MICROGRAPH OF SAMPLE 3

The next pellet was compacted at 9 ton pressure and at elevated temperature up-to 1200°C,since the pressure was very high due to which the density increases as the compaction between the particles was very good. The particle were very dense .Grain size, particle size was calculated by using straight line method. The particle size ranges from 4µ to 15µm.since the pressure was increasing there was increase in grain size of the particles. The SEM micrograph shown below representing the compacted pellet at 9 ton, the particle were uneven and CIP particles diffused very strongly on the abrasive surface.

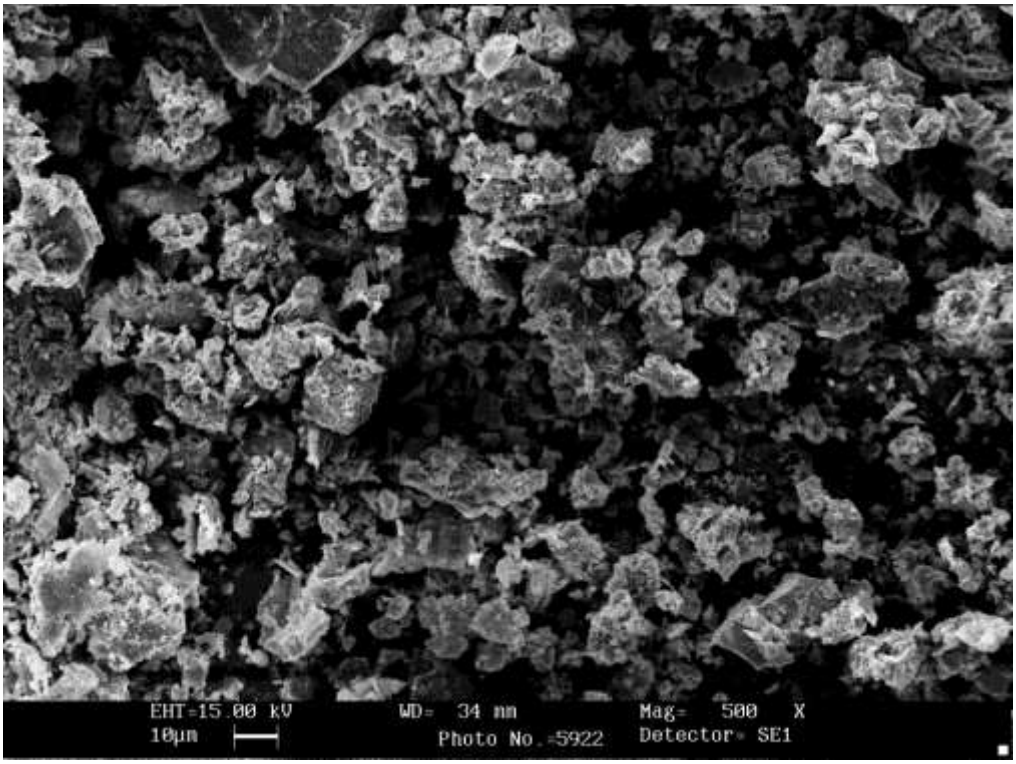


Fig 34: SEM micrograph compacted at 9 ton showing densely compacted SiC agglomerates.

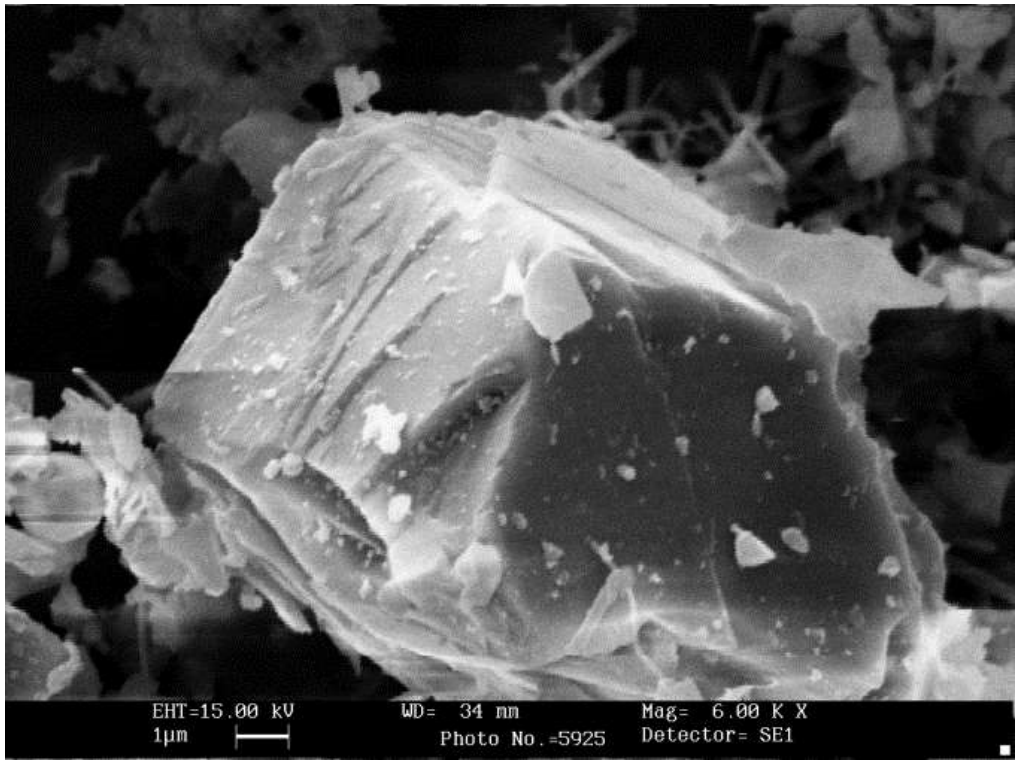


Fig 35: SEM micrograph at higher magnification showing the geometry of abrasive particle with sharp edges

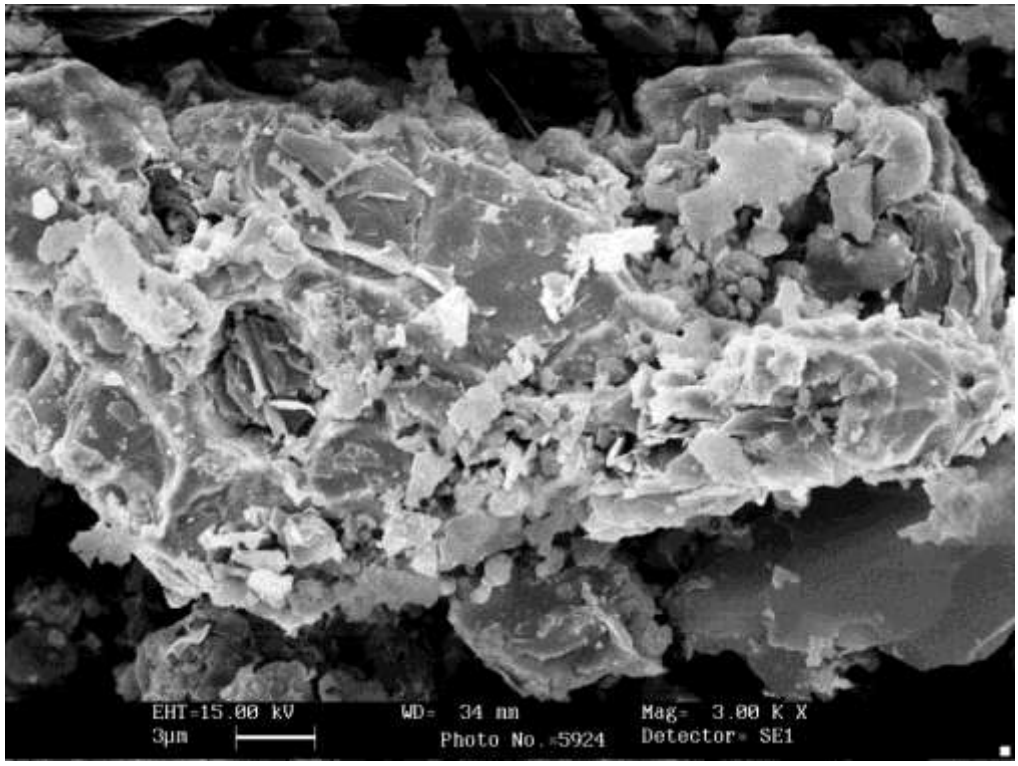
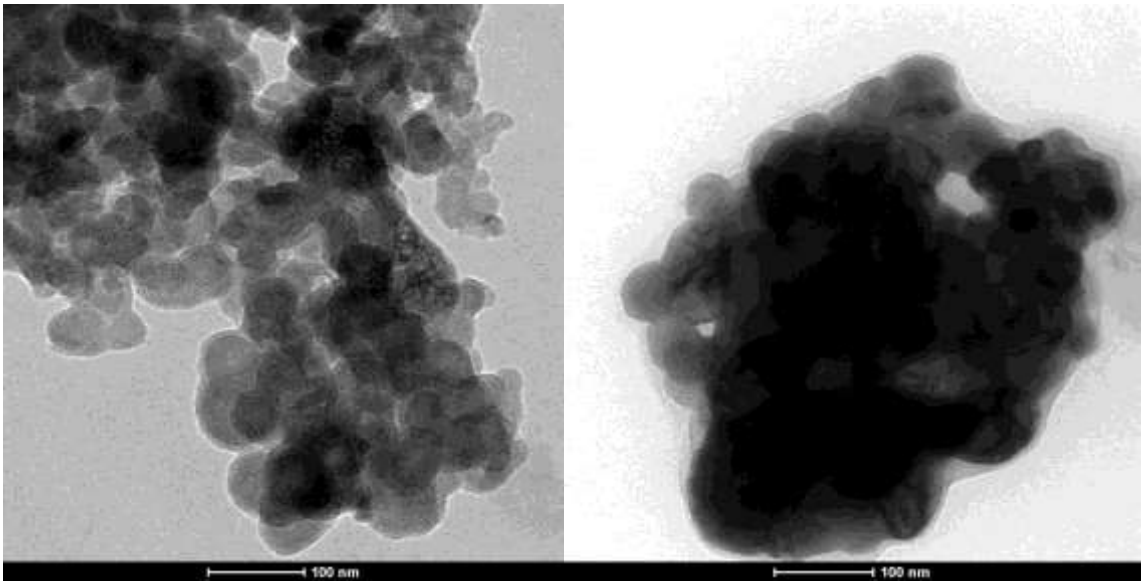


Fig 36:SEM micrograph showing the densely packed structure of sintered abrasive magnetic grains, it can clearly be seen the abrasive and magnetic particle , particles are compacted densely due to high pressure.

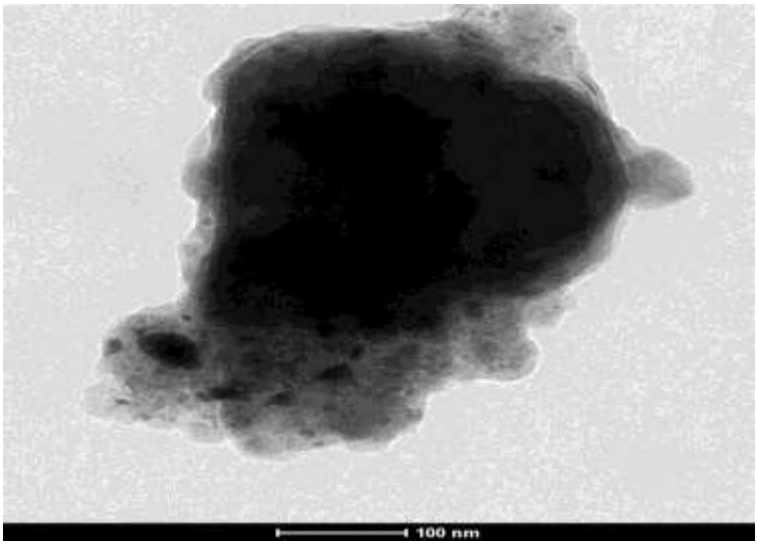
The result investigated from the SEM micrographs of 5,7,9 ton respectively showing that the grain size and particle size increases with increase in compaction load and the agglomerates deposition on the abrasive surface also increases. The topography was compacting with increasing pressure, SEM micrograph at 5 ton showing the disputed geometry, while at 7 ton the geometry was somewhat compacted compared to the 5 ton specimen while the most compacted geometry can be seen in 9 ton compacted pressure. Since the silicon carbide is a ductile material and carbonyl iron is brittle ,when the composition form the fracture can be described by using griffths theory of fracture in which the material elongate or stretched.

7.5 TRANSMISSION ELECTRON MICROSOCPY (TEM)

Nano level characterization was done by using transmission electron microscopy. The TEM micrograph shown below representing the sintered abrasive magnetic powders. TEM micrographs below were compacted at 5 ton,7 ton and 9 ton were taken at 100 µm respectively. The incident direction of the electron beam was nearly perpendicular to the compressive plane of the each sample (in other words, the electron beam was almost parallel to the LPS compressive direction). Most of the grains were equi-axed nano grains with the size of ~100 nm. Some large grains with the size of~300 nm were occasionally observed. In fig (a) the grains can be seen clearly the bright surface in the micrographs due to less dense grains, while the dark spots occurs due the presence of stresses as the ,as the beam of light incident at the surface, the light which passes through the particle can be seen as bright while the incident light which was reflected back due to dense and strongly bonded grains shows the dark. while the TEM micrograph at 7 ton shows the densely compacted structure, due to high compaction pressure the bonds between the grains was very strong as only dark phases can seen ,while in TEM micrograph at 9 ton was most densely compacted as the pressure was very high only dark phases can be seen and at the edges the carbonyl iron particle can be seen.



(a) (b)



(c)

Fig 38 TEM micrographs of sintered abrasive magnetic powder at (a)5ton,(b)7 ton,(c)9 ton respectively.

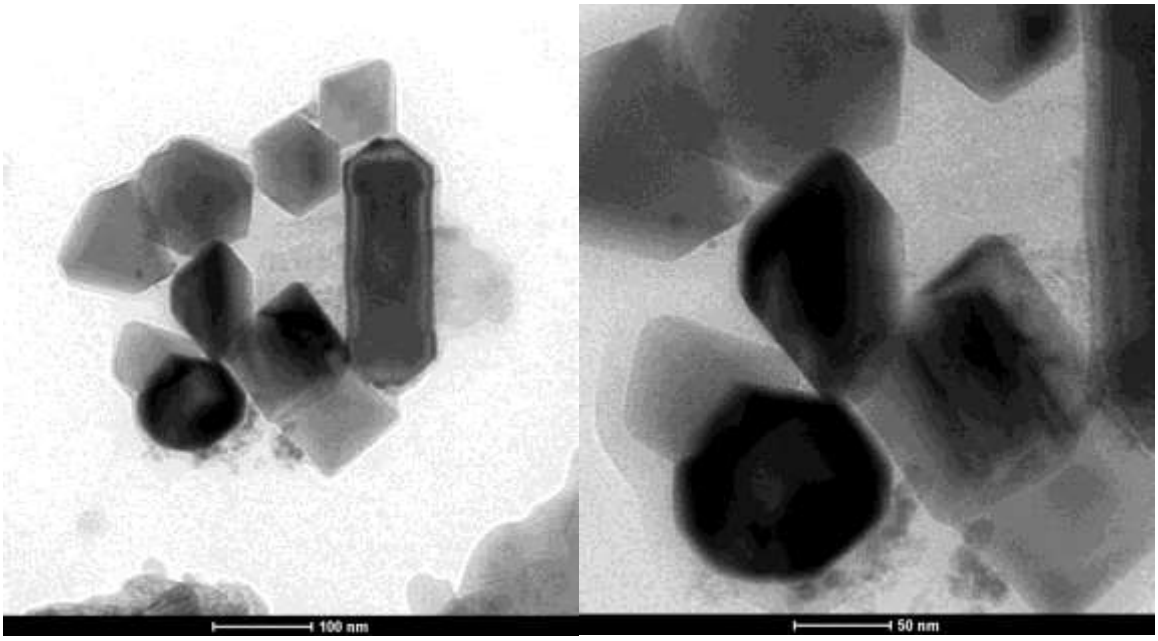


Fig 39 (d) TEM micrograph of sample 1,Grains of abrsaive can be clearly seen.

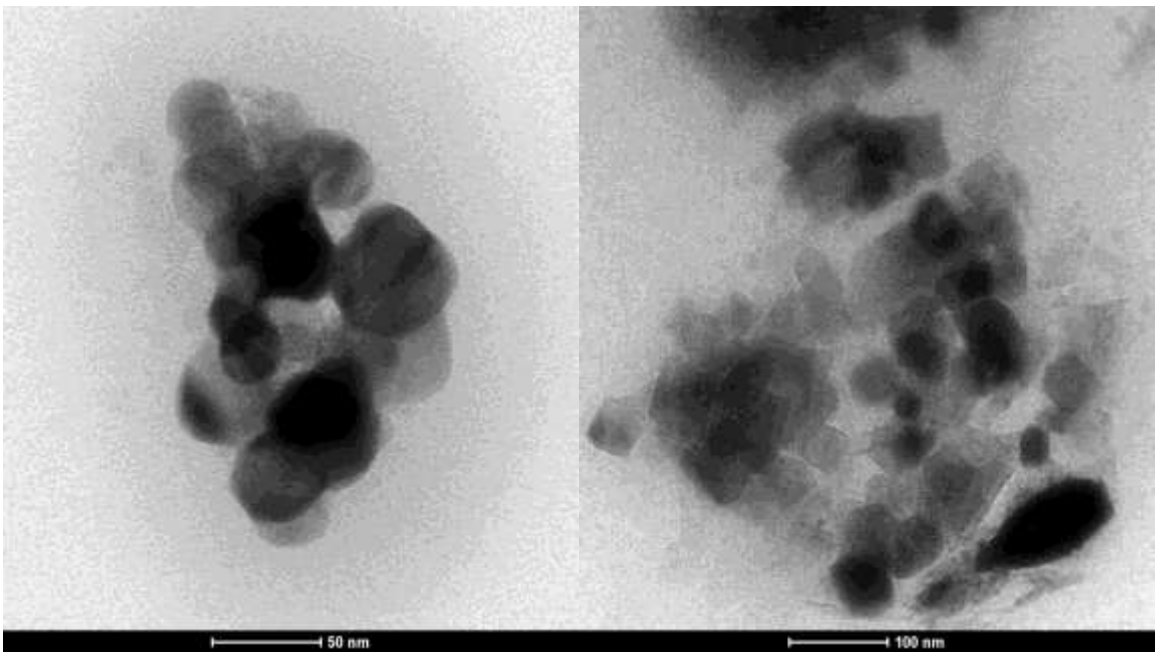


Fig 40 (e)TEM micrograph of sample 2,The grain are comapcated,the dark grains bonded densely.

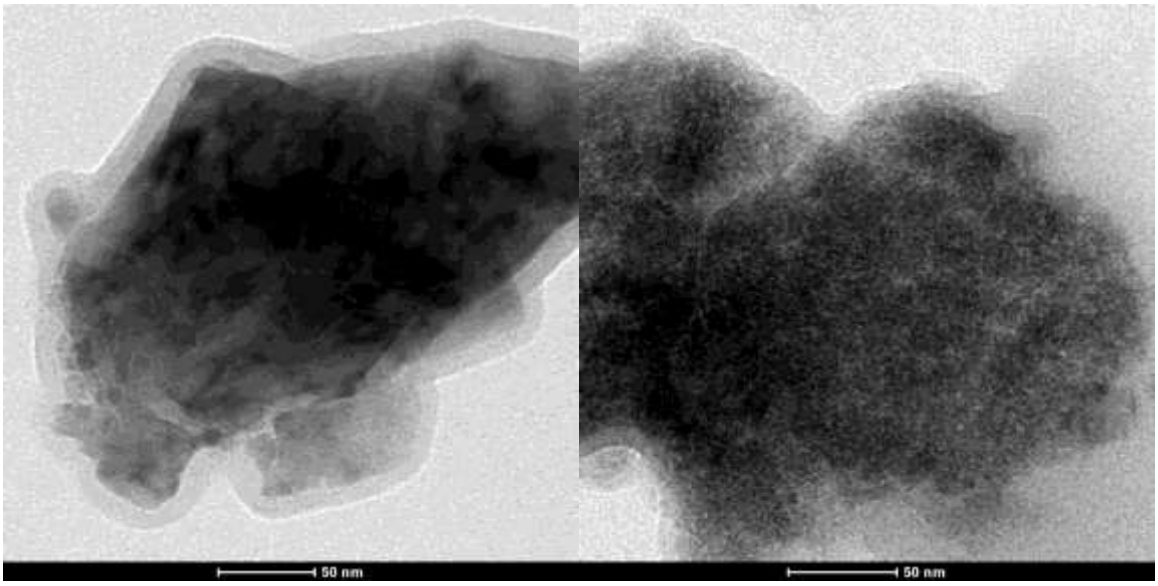


Fig 41 (f) TEM micrograph of sample 3,the structure is very densely packed ,and on the second image the CIP can be clearly seen as the process was liquid phase sintering ,the CIP particles melt and diffused at the surface of abrasives.

7.6ELECTRON DISPERSIVE SPECTRUM (EDS) ANALYSIS

SAMPLE 1

TEM micrograph and EDS spectrum of the 5 ton compacted specimen is shown below,the graph represent the no. of particles at different energy levels.The results optimized from the graph shows the composition contains C,O,Si,Fe,and Cu repsectively.since the compostion optimzed is same as the compostion of sintered abrasive magnetic but the presence of copper shown in higher extent ,The reason behind the presence of copper due to the sample grid which was palced in TEM for characterization,when the ray of light incident as it firstly crosses the copper grid as a result the copper present in highet counts on EDS spectrum.The quantum numbers distributed as C has unitary shell K ,O has unitary shell K ,Si has K,Land M shells,Fe has K,Land M shells.

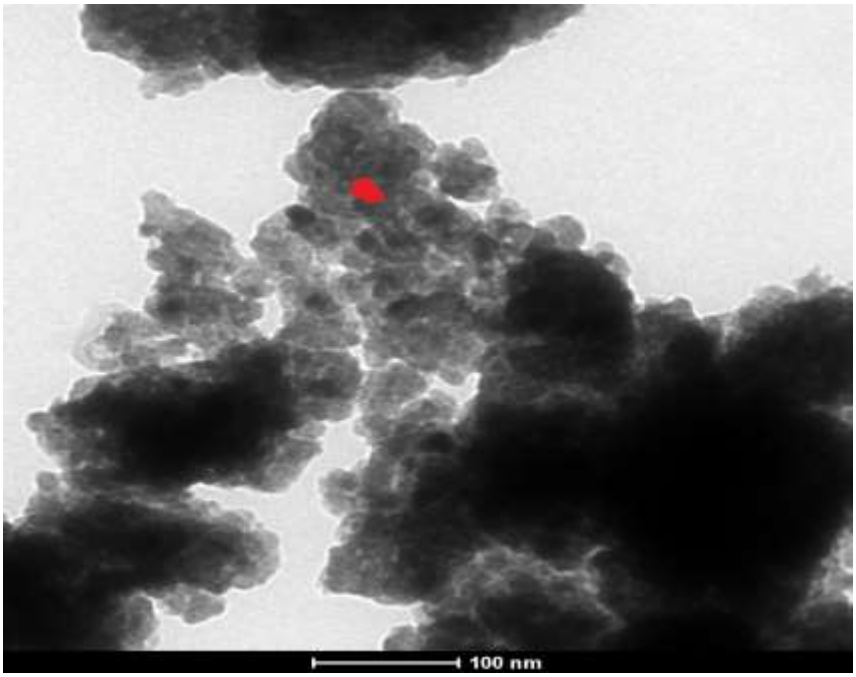


Fig 42 TEM micrograph of sample 1 compacted at 5 ton pressure,

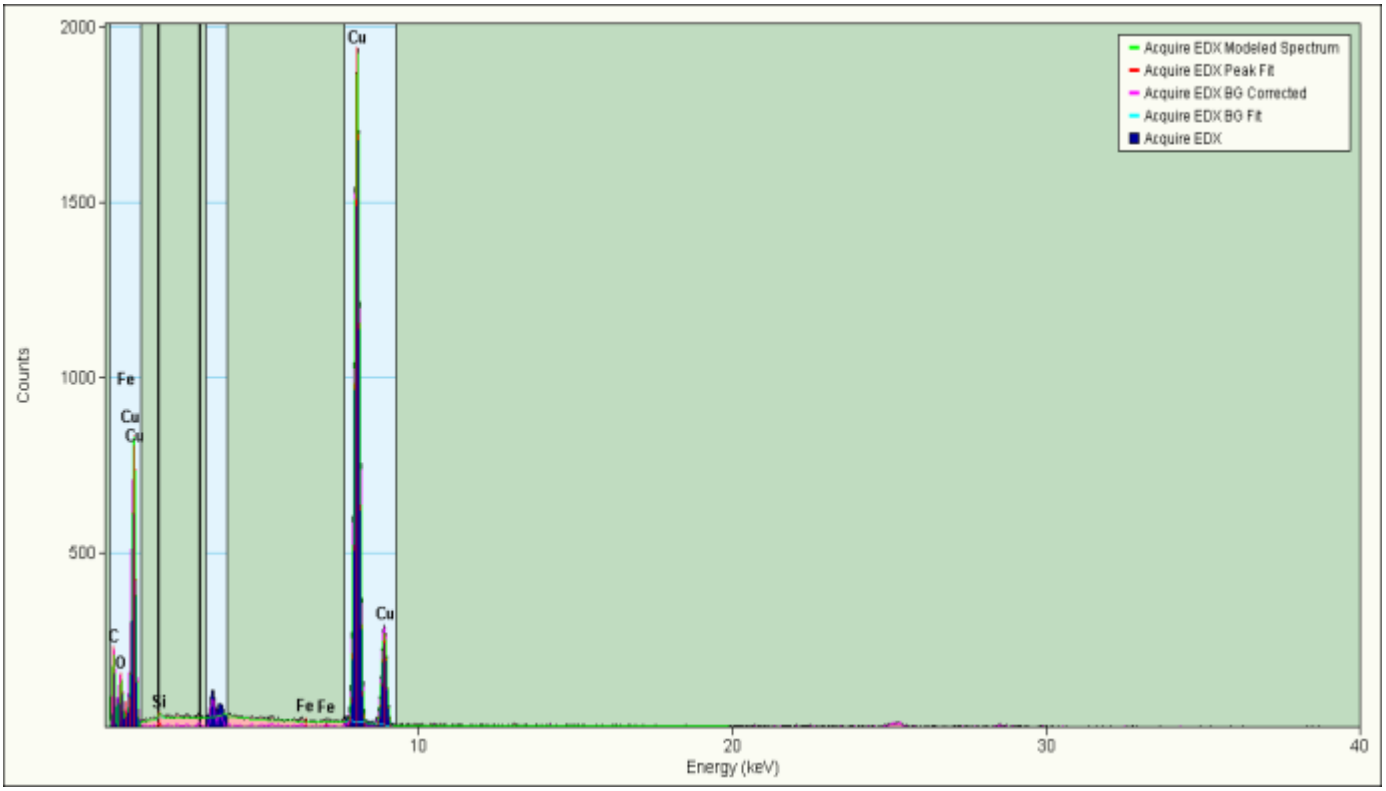


Fig 43:EDS spectrum graph of sample

Table10 composition table from EDS

Peak	Integrated intensity	Uncertainty
C-K	2020.771	413.427
0-K	1400.299	418.276
Si-K	129.504	74.466
Si-L	0	100
Si-M	0	100
Fe-K	0	100
Fe-L	365.006	208.376
Fe-M	0	100
Cu-K	33882.605	1787.940

SAMPLE 2

The TEM micrograph shown below representing the specimen compacted at 7 ton pressure ,The EDS graphs shows the composition at the grains. The composition shows the contents of C,O,Si,Fe and Cu respectively ,since the value of copper is very high as compare to 5 ton specimen .since the electron volt is very high. The quantum numbers for C has only K unitary shell,O also has K,Si has K and L moves to two shells ,Fe has K,L, and M has three shells ,while Cu has K and L shells.

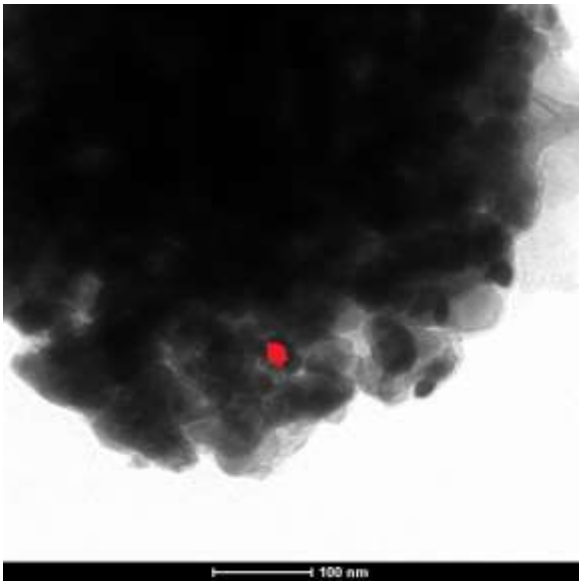


Fig 44: TEM micrograph of sample 2

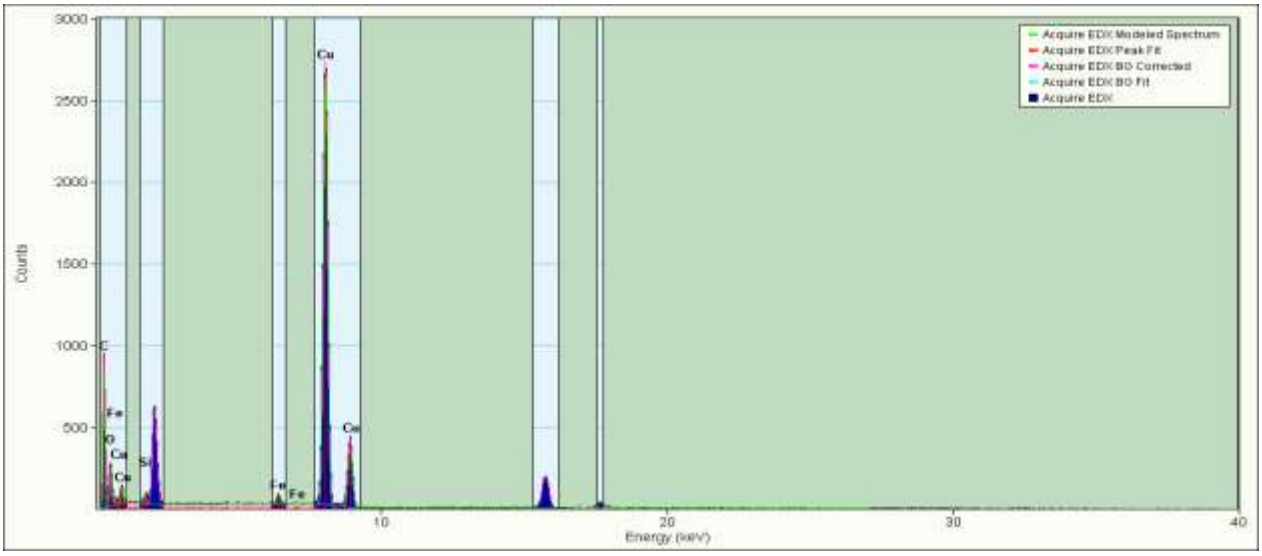


Fig 45:EDS spectrum of sample 2

Table 11:Composition table

Peak	Integrated intensity	Uncertainity
C-K	5915.500	809.338
O-K	1922.890	495.763
Si-K	570.264	255.205
Si-L	0	100
Si-M	0	100
Fe-K	1006.820	266.669
Fe-L	333.338	186.230
Fe-M	0	100
Cu-K	43434.500	2098.130
Cu-L	714.895	280.680

SAMPLE 3

The TEM micrograph sintered abrasive magnetic specimen compacted at the pressure of 9 ton is shown below .The red point representing the area for the EDS spectrum. The result obtain from the EDA spectrum is the composition sintered abrasive contains is C,O,Si,Fe, and Cu.Cu composition is maximum. Since the C has only unitary subshell K, while O also has unitary subshell K,Si also has unitary subshell, while Fe has K and L shell both while Cu has K and L both.

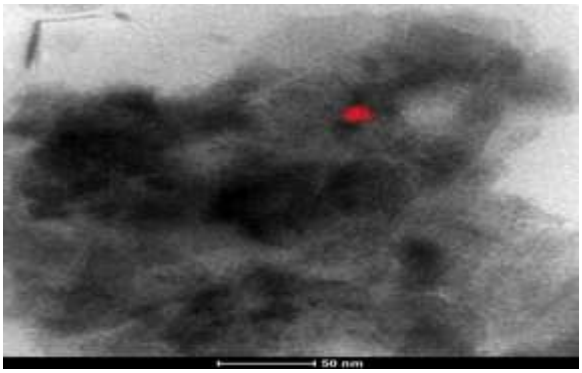


Fig 46 TEM micrograph of sample 3

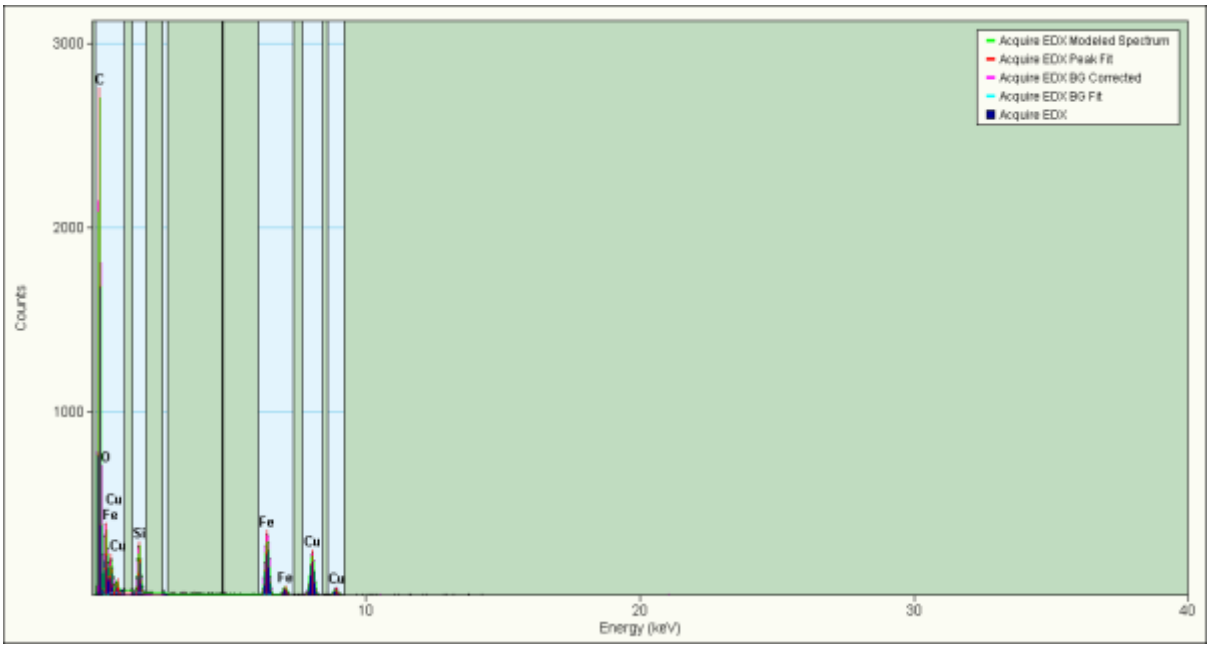


Fig 47 EDS spectrum graph

Table 12: composition and peak values

Peak	Integrated intensity	Uncertainity
C-K	19651.462	1437.160
O-K	2781.465	614.958
Si-K	2524.164	559.294
Fe-K	4497.754	698.499
Fe-L	1288.691	364.336
Cu-K	3605.289	584.100
Cu-L	505.402	249.972

7.7 SELECTED AREA PATTERN(SAD)

Selected area diffraction (SAD) is a TEM technique to obtain diffraction patterns that result from the electron beam scattered by the sample lattice. Obeying the bragg's law, the electrons are scattered elastically by the lattice, therefore, we can index the diffraction spots in the pattern and identify the phases in the sample and study their structures. Typically, the area size of the sample been selected by SAD aperture is 0.5-1 μm, so the microstructure of the sample could be fine selected and tested.

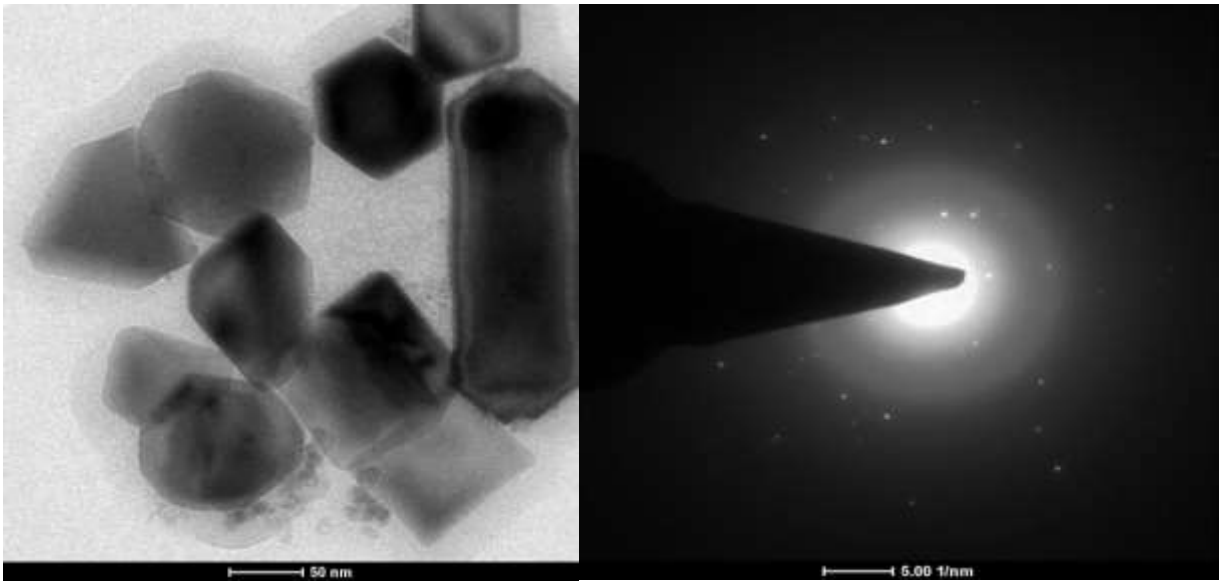


Fig 48TEM micrograph and SAD pattern of sample 1

The TEM micrograph of sintered abrasive magnetic sample compacted at 5 ton, The SAD diagram shows the crystalline structure of the grains , since there are small white dots also present ,so there is possibility of presence of particle with very small size, which can be seen in TEM micrograph also. The composition test confirms the presence of Sic and CIP particle .The particles with very small size represent CIP particles. while the bright circle represents there are particles with nano size or very much small in size. As results optimized form the SAD pattern the presence of Sic and CIP which was expected.

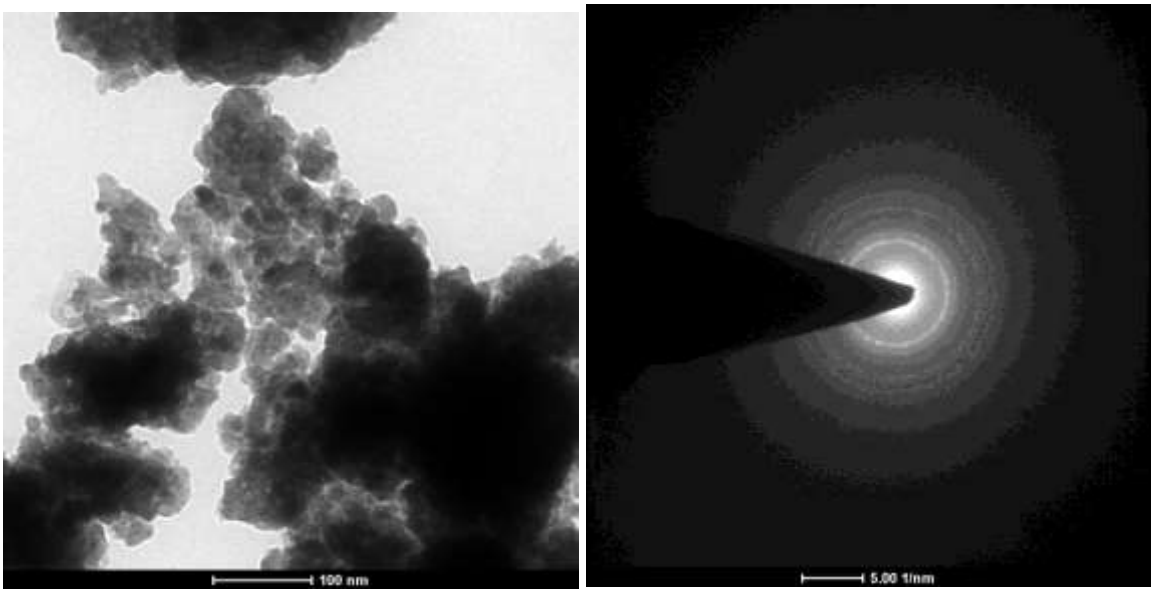


Fig 49 TEM micrograph and SAD pattern of sample 1 on other area

The TEM micrograph of sintered abrasive magnetic sample compacted at 5 ton shows an agglomeration of SiC crystallites. The rings are continuously increasing , which is showing that the grain size is decreasing and some of the rings are not completely formed so there are some particles with smaller grain size. The SAD indicates a polycrystalline material due to the powder rings. Because the crystallites were small, it was not possible to obtain a single crystal diffraction pattern from a crystallite.

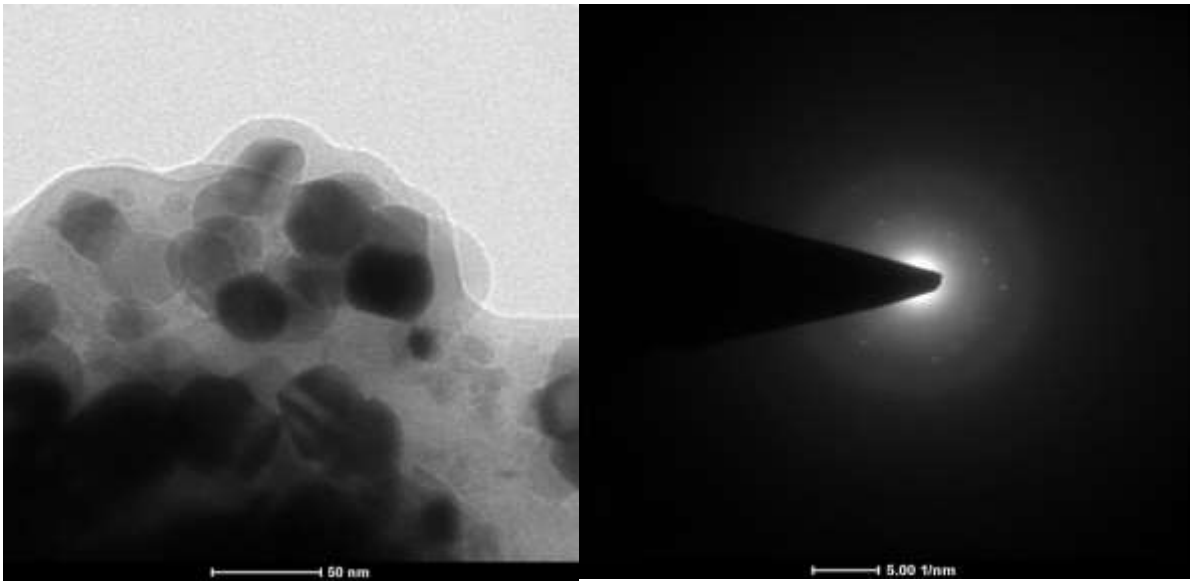


Fig 50 TEM micrograph and SAD pattern of sample 2

The TEM micrograph of sintered abrasive magnetic sample compacted at the pressure 7 ton. There were no rings as the bright field are small, So the particles present which are smaller in size and it shows amorphous structure as the bright circle is more, The reason behind the

structure as it is compacted at high pressure due to which the grains compacted very closely or packed much, And also due to the sintering. The dark field in the microscope represents the grains which are closely packed and the beam of light doesn't pass through it.

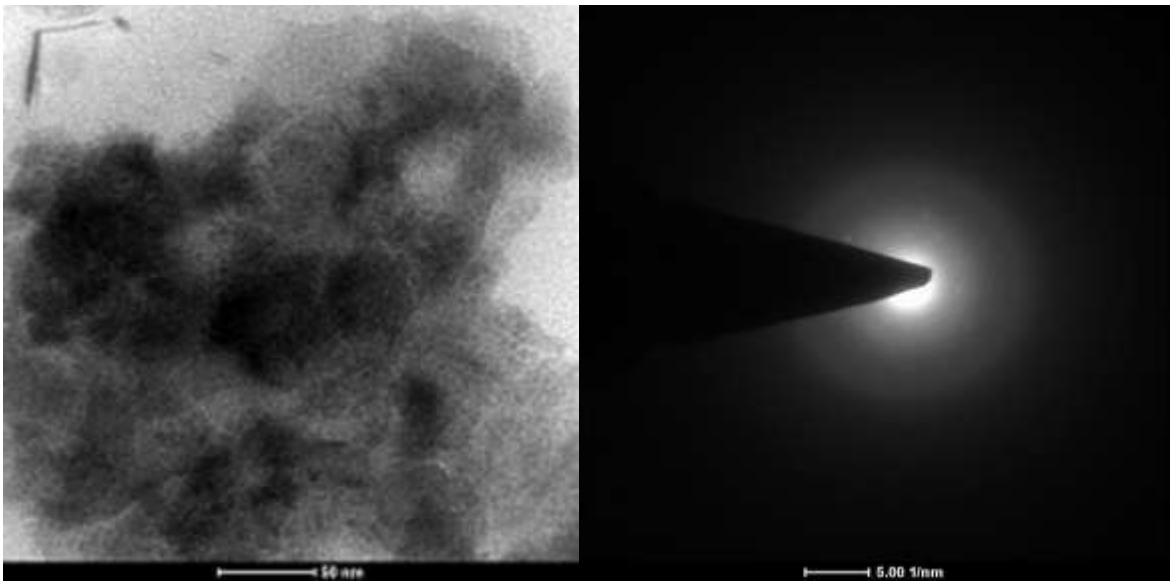


Fig 51 TEM micrograph and SAD pattern sample 3

The TEM micrograph of sintered abrasive magnetic sample compacted at 9 ton pressure. TEM micrograph shows the fully dense SiC and CIP, crystals cannot be seen clearly due to densely packed structure. The agglomeration of Sic can be seen.The SAD diagram shows the small bright field and bright circle which shows the amorphous structure and the particles size is very small.

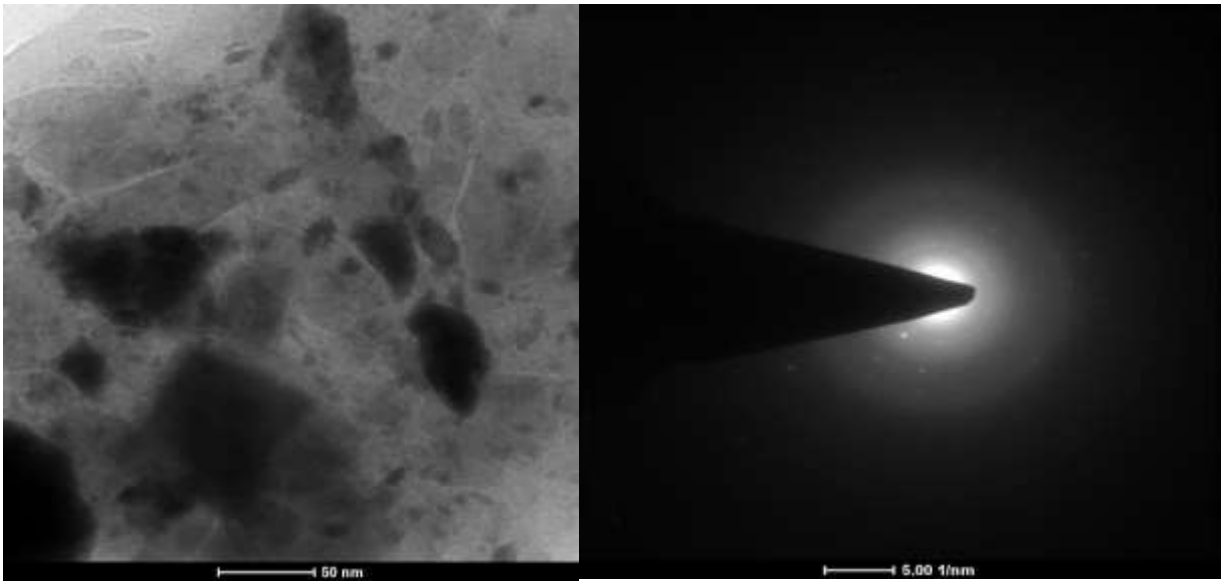


Fig 52 TEM micrograph and SAD pattern of sample 3 at other area

The TEM micrograph of sintered abrasive magnetic powder sample compacted at 9 ton. TEM micrograph shows the phases can be seen and the dark grain are strongly bonded i.e. Dark grains are those with a zone axis aligned with the electron beam creating the diffraction pattern shown in the inset .Structure is very densely packed ,the particle size ranges in average and it have polycrystalline structure due to presence of SiC grains. The densification achieved fully during sintering due to the closely packed structure.

CHAPTER 8

CONCLUSIONS

8.1 OVERVIEW

The main purpose of this research was to develop a sintered abrasive magnetic powder that resulted composed of superior properties than single component. The method was to mix homogenously abrasive and magnetic powder to create a strong bond. In order to accomplish this , sufficient processing of the components was necessary to break up agglomerates and uniformly mix the powders .Sintering of these powders was completed using pressure less sintering i.e. Liquid Phase Sintering (LPS).After processing and sintering, the microstructure of the final samples was investigated. Several methods, including micro hardness, density measurement, scanning electron microscopy, and transmission electron microscopy were conducted. The information gathered from the microstructure characterization explained hoe the processing conditions gave the resulting properties of the sintered abrasive magnetic powder.

8.2 PROCESSING

During processing we learned the important step to remove the compacted powder carefully ,as in this research work there was no additive or binder used ,so it was very difficult to remove green compact from the die ,either it stick on the die ,to avoid sticking grafoil should be placed between powder and die. We observed from ball milling that it bring the size of particles homogenously. The ball milling step was conducted in dry conditions. The ability to freely ball mill the powders was beneficial because after milling the powders were much easier to work with than initial powders. The initial powders were nano-sized, and they tended to airborne and became a hazard to work. The ball milling provides the powders to lose much of their fluffiness, which was attributed agglomeration of particles.

8.3 MICROSTRUCTURE

In this research work three samples were taken sintered in liquid phase sintering technique, All the samples described in this research work exhibited considerable density. The dense microstructure was assumed to be result of complete densification. We concluded that the

temperature duration in sintering was sufficient to fully sinter the samples. By measuring the density in three samples, we concluded that the theoretical density gave the lowest measurement, while the experimental i.e. Archimedes' gave the highest value. As the value of density increases with increasing the compaction pressure. And the samples were stable at high compaction pressure.

The following results concluded from the three samples compacted at three different pressures

1. The highest sample density were obtained from the high compaction pressure sample.

2. Density increases with increasing compaction pressure.

3. The holding time calculated was 14 minutes.

4. The SEM micrograph displayed considerable densities in all samples, and grain were clearly visible in each sample.

5. The SEM micrograph depicted with good homogeneity in microstructure.

6. The TEM micrographs and SAD patterns displayed the evidence of presence of CIP and SiC particles respectively.

7. Hardness increases with increase in compaction load.

CHAPTER 9

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