DEVELOPMENT AND CHARACTERIZATION OF MAGNETIC ABRASIVE PARTICLE USING SOLID PHASE SINTERING

Submitted by

MONIKA

(Roll No. : **2K13/PIE/13**)

Mechanical Engineering Department

In partial fulfillment of the requirement of the degree of

MASTERS OF TECHNOLOGY

In

Production Engineering

Under the guidance of

Mr. M. S. NIRANJAN

Assistant Professor

Department of Mechanical Engineering

DELHI TECHNOLOGICAL UNIVERSITY

(Formerly Delhi College of Engineering)

Bawana Road, New Delhi

JULY 2015

CERTIFICATE

This is to certify that report entitled **"DEVELOPMENT AND CHARACTERIZATION OF MAGNETIC ABRASIVE PARTICLE USING SOLID PHASE SINTERING"** by **MONIKA,** is the requirement of the partial fulfillment for the award of Degree of **Master of Technology (M. Tech.)** in **Production Engineering** at **Delhi Technological University**. This work was completed under our supervision and guidance. He has completed his work with utmost sincerity and diligence. The work embodied in this project has not been submitted for the award of any other degree to the best of my knowledge.

(Guide)
M.S. NIRANJAN

(ASSISTANT PROFESSOR)

(Co-Guide)
Dr. QASIM MURTAZA

(ASSOCIATE PROFESSOR)

DEPARTMENT OF MECHANICAL ENGINEERING

DELHI TECHNOLOGICAL UNIVERSITY

DELHI

ACKNOWLDGEMENT

To achieve success in any work, guidance plays an important role. It makes us put right amount of energy in the right direction and at right time to obtain the desired result. I express my sincere gratitude to my supervisors, **Mr. M. S. NIRANJAN, Assistant Professor**, and **DR. QASIM MURTAZA, Associate Professor** Mechanical Engineering Department for giving valuable guidance during the course of this work, for their ever encouraging and timely moral support. Their enormous knowledge always helped me unconditionally to solve various problems. I am greatly thankful to **Dr. R.S MISHRA**, Professor and Head, Mechanical Engineering Department, Delhi Technological University, for his encouragement and inspiration for execution of the this work. I express my feelings of thanks to the entire faculty and staff, Department of Mechanical Engineering, Delhi Technological University, and Delhi for their help, inspiration and moral support, which went a long way in the successful completion of my report work.

MONIKA

2K13/PIE/13

ABSTRACT

Magnetic abrasive particles (MAPs) have been developed using solid phase sintering method. Carbonyl iron powder of 20 volume% and silicon carbide abrasives of 3000 mesh size with 25 volume% have been uniformly mixed in ball mill. After mixing the powder, the pellets of 5 gram each have been prepared at 8 ton pressure using cylindrical die of 7 mm diameter and 50 mm length. The pellets have been sintered at 1000°C in inert atmosphere of argon using appropriate sintering cycle. After sintering, the furnace cooling was done in inert atmosphere of argon upto environment temperature. After sintering, the sintered pellets have been crushed in ball mill to obtain the required size of the magnetic abrasive particles. The morphology and elemental composition as well as particle size of magnetic abrasive particles have been studied with scanning electron microscope (SEM) and energy dispersive spectrometer (EDS). The different phases of magnetic abrasive particles have been studied using X-ray diffraction (XRD). The microstructure of magnetic abrasive particles has also been studied with optical microscope.

Keywords: Sintering, Carbonyl iron, Magnetic Abrasive particles, SEM, EDS, XRD.

CONTENTS

LIST OF FIGURES

LIST OF TABLES

NOMENCLATURE

PM	Powder Metallurgy
VOL	Volume
SiC	Silicon carbide
CIP	Carbonyl iron powder
°C	Degree Celsius
i.e.	That is
%	Percent
e.g.	Example
Wt	Weight
D	Diameter
Kgf	kilogram force
Mm	Micrometers
Mm	Millimetre
MA	Magnetic abrasive
XRD	X-ray diffraction
N	Newton
CRH	Constant rate heating
In	Inch
Å	Angstrom
ʎ	Wavelength

DMLS	Direct metal laser sintering
LPS	liquid phase sintering

CHAPTER 1
INTRODUCTION

Many technical investigations by materials scientists have been continuously aimed towards improving the performance and characterstics of materials. major improvements in mechanicaland physical properties have been achieved through chemistry variations, mechanical, and thermo mechanical processing methods. [1]. SiC is a chemical compound of silicon and carbon. It shaped by an elevated temperature electrochemical effect of sand and carbon. SiC is a brilliant abrasive is used to in making grinding wheel and is used as abrasive product. It is used in abrasives, ceramics, and many high-performance uses. These materials are used in electrical conductor and have uses in resistance heating, as ignitersof flame and electronic elements. Silicon Carbide Properties have low density, high strength, low thermal expansion, high hardness, high thermal conductivity and exceptional thermal shock resistance. Silicon carbide is composed of tetrahedral carbon and silicon atoms in crystal . Sic produces tough material. Sic is unaffected by any chemical or alkalis or molten salts up to 800°C. Resistance to any kind chemical reaction at temperature, and strength retention at elevated temperatures has made this material very admired. Sic is used in electrical furnaces due to its high electrical conduction. CIP is a pure form of iron which is prepared by chemical decomposing of penta carbonyl. It is a grey powder having spherical micro particles. Carbonyl iron is used in magnetic cores high spherical particles of CIP are used as a part of radar used in military .CIP has high stability at high temperature. Particles of CIP (20-40%) suspended in a fluid (60-80%) are used as a MRF . Four categories of ceramic matrix composites are classified by Nihara [2]. These nanocomposites show improved properties both at room temperature and at high temperature. The Hybridization of both micro-nano composites is expected to give further improvement. However the synthesis of nonmaterial's for bulk production is difficult due to grain growth of initial fine particles, introduction of processing related process flaws during initial sample preparation and handling of materials on its original dimension till the final microstructure development. Development need of composite materials is very necessary due to improved physical and mechanical properties. Iron has specific saturation magnetization and it is a very soft magnetic material, low coactivity and a high Currie temperature. A very highly pure iron is carbonyl iron powder and it is

manufactured by chemical decomposition of iron penta carbonyl. It usually has the appearance of grey powder. Most of the impurities , oxygen, nitrogen and carbon [3]

1.1 Powder metallurgy

Powder metallurgy technique is used to mix the material which does not form an alloy. The initial step is to compact metal powder into PM product shape at room temperature. Then heating is used to causes the powder particles to mingle together without melting.PM has adequate physical and mechanical properties. The cost of producing a component of given shape and the required dimensional tolerances by PM is generally lower than the cost of casting or making it as a wrought product, because of extremely low scrap and the fewer processing steps..The advantage of cost is the main reason for selecting PM as a process of production for high – volume component which needs to be produced exactly to, or close to final dimensions. Production rate of parts is quite high. PM parts have several industrial applications. These include self – lubricating bearings, porous metal filters, gears, cams, brackets, sprockets, etc.

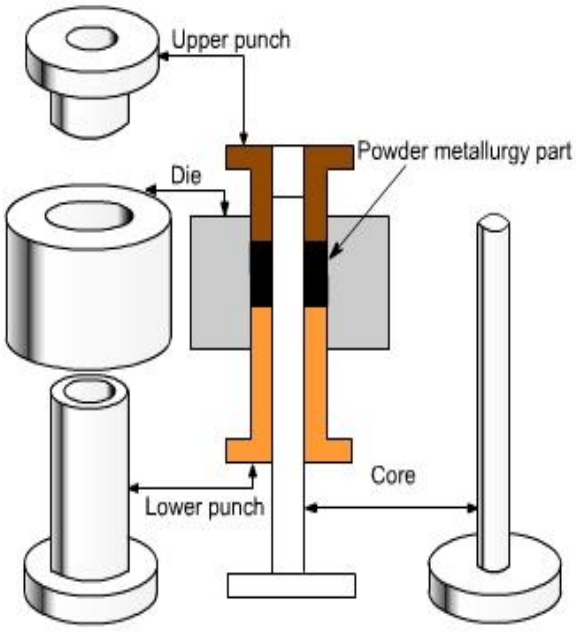


Fig. 1.1 Set of powder metallurgy tools

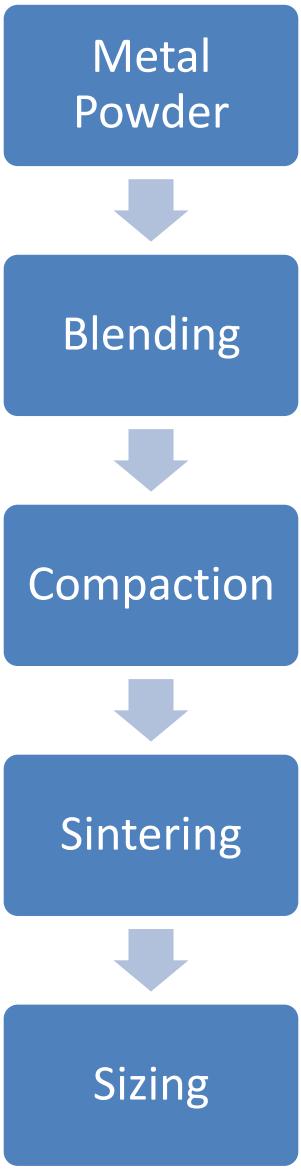


Fig. 1.2 Sequence of powder metallurgy methods

1.1.1. Methods of Making Metal Powders

1.1.1.1. Metal crushing and pulverizing: Brittle materials can be converted into powder by crushing. If the material is not sufficiently brittle, its temperature is decreased.

1.1.1.2. Atomization: ductile materials or the material having low melting point, can be converted into powder by this method. Liquefied material is poured over a high speed rotating disk. Due to the centrifugal action, liquid metal comes down in the form of very fine droplets. The material can also be sprayed by using a plasma torch.

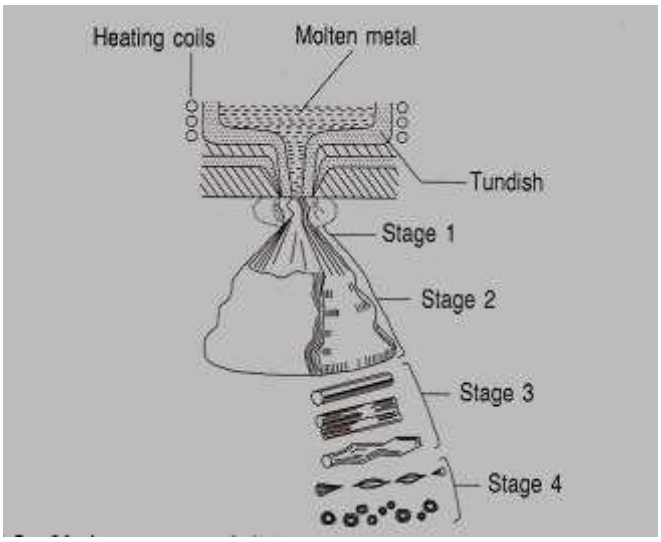


Fig. 1.3 Atomization process

1.1.1.3. Corrosion: when stainless steel is kept in the environment of sulphuric acid and copper sulphate, it dissolves and settles down at the bottom of tank. But this method takes time.

1.1.2. Blending: by mixing the lubricant with powders, a layer of lubricant will be deposited over the particles. This will increase the interaction between them and result of that powder can be given some shape called green compact. This is called as green because it is freshly prepared.

1.1.3 .Compaction it is also called as iso-static pressing .to give initial strength to the green compact powder is pressed on a press .variation in the properties of compact will be more when the compaction is done on a single or a double action press .but on a double action press properties will be more uniform .smaller is size of particle,better will be under diffusion and the Compact will be stronger .if the particle is smaller, the strength will be better because more are will be under diffusion .

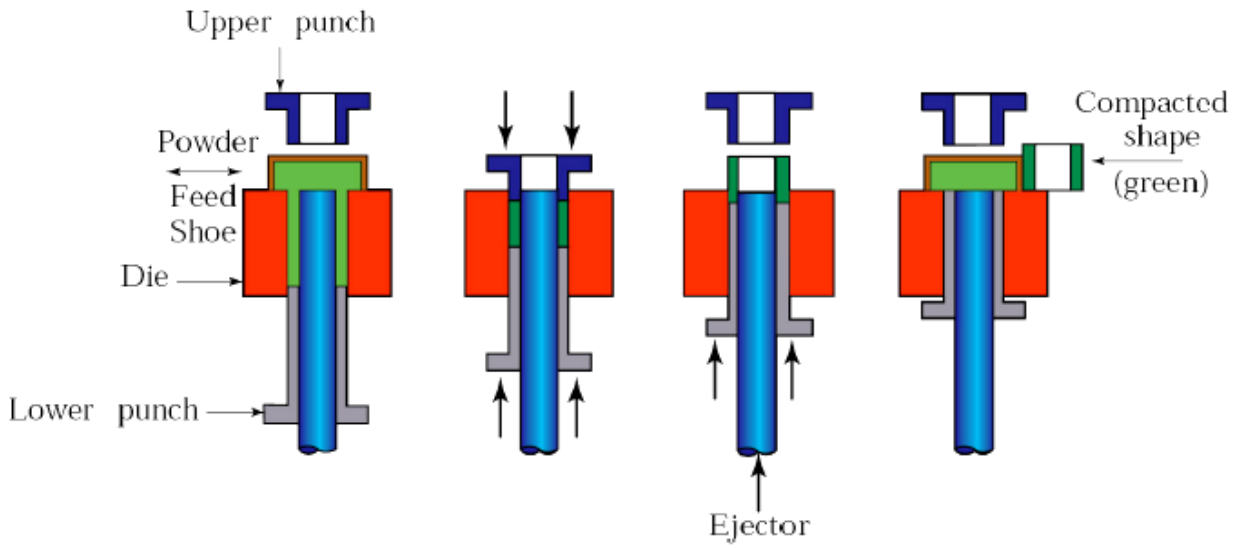


Fig. 1.4 Compaction of metal powder

1.1.4.1 Pre-sintering: when heating is done along with compaction it is called Hot iso-static pressing (HIP).compact is heated and due to that lubricant on the surface of particle will burn. Characteristic of lubricant should be such that after burning, it should not leave any residue. This provides the localized heating which increases the extent of diffusion between the particles .When pre-sintering is clubbed compaction, it is called Hot iso-static pressing. Rough particles give better strength as interlocking among the particle is better.

1.1.4.2. Sintering: In the sintering stage, compact is heated to60-70% of the melting point of the base metal. This intensify the extent of diffusion between the particles. Those powder which could not form interlocking and whose melting point are below this temperature, they will be liquefied and will fill up the the voids. When the compact is a mixture of large numbers of powders having large difference in their melting point two or three stage melting process is preformed. After sintering, product appears to be very hard and brittle, so normally no machining is advisable but to give some simple shape to the part sizing is performed. Powder metallurgy can only be applied for mass production because of expensive tooling arrangement.

Whenever needed, additional operations are carried out on sintered PM parts in order to further improve their properties or to impart special characteristics. Some significant operations are as under.

1.1.5.1. Coining and sizing. These are high compacting process. It imparts bigger dimensional accuracy to the sintered part, greater strength and better surface.

1.1.5.2. Forging. The sintered PM parts may be hot or cold forged to produce shape, better Surface finish, a uniform grain and good dimension

1.1.5.3. Impregnation. The inbuilt porosity of PM parts is used by impregnating them with fluid which may be oil or grease. It is mainly for sintered bearings that are internally lubricated with up to 30% oil by volume by immersing them in warm oil. Impregnated components have a unremitting supply of oil by capillary action, during their use.

1.1.5.4. Infiltration. The pores of sintered part are filled with some low melting point metal with the result that part's hardness and tensile strength are improved. Sintered components are kept close to a slug of metal to be impregnated and they are together heated to slug melting point .

1.1.5.5. Heat Treatment. Sintered PM products may be heat treated to get greater hardnessin them.

1.1.5.6. Machining. The sintered parts are machined by turning, milling, drilling, threading, grinding, etc. to get various geometric characterstics.

Applications of the powder metallurgy process are:

1. Filament of a bulb, tube.
2. Cutting tool and grinding wheel.
3. Nozzles for abrasive jet machining.
4. Porous bearing or self lubricating bearing.
5. Filter used in casting process.
6. Friction material in anti-lock braking in anti-lock braking system (ABS).
7. Atomic Energy Application.
8. Refectory Parts.
9. Use of parts in military and defence system.
10. Products of complex shapes.

Fig. 1.5 Complex shapes manufactured by PM

Fig. 1.6 Automobile parts manufactured by PM

Fig. 1.7 Bearing production using PM

CHAPTER 2
LITERATURE REVIEW

In this chapter description of research papers studied for the present work is given. Some researchers have done remarkable work in the field of sintered magnetic abrasive particles, MAF, MAMF. They investigated the effects of process parameters like pressure sintering temperature, abrasive concentration and grain size on the output response namely surface finish the material removal. The details of the research paper studied are given below.

Palwinder singh and lakvirsingh [4] focused on internal finishing of cylindrical pipes using sintered magnetic abrasive. This research work shows the feasibility of using Al_2O_3 based sintered magnetic abrasive particles for the internal finishing of cylindrical brass pipes and gained an understanding of the mechanism involved. The XRD analysis shows that reduction in the heights of peaks indicates improvement in the finishing of the surface using sintered abrasives.

Upadhayaya and Nekatibeb [5] shows the effect of heating mode on sinter ability of carbonyl iron compacts. He investigated the effects of alloy addition and mode of heating on densification response, micro structural evolution and mechanical property response of Fe, Fe–2Cu and Fe–2Cu. As compared to conventional furnace heating, the sintering cycle time in a microwave furnace was reduced by 60%.

Ulrich Degenhardt et al [6] have studied sintered silicon nitride/nano-silicon carbide materials based on preceramic polymers and ceramic powder. In their research work they concluded that when Si_3N_4 powder particles were coated homogeneously with a poly-carbosilazane by industrial applicable processes like fluidized bed granulation. The resulting granulate was used for the manufacturing of complex shaped components with industrial equipment.

Chen Lung Chih et al [7] aimed to use MOCVI conducted in fluidized bed employed for the preparation of nano-sized composites of ceramic. The Cr-species infiltrated into Al_2O_3 particle by the pyrolysis of chromium carbonyl (Cr (CO) at 300–450°C. The granulated powder is

pressure less sintered to accomplish high density. The outcomes show that the dominant factors influencing the Cr-carbide stage formation, either Cr_3C_2 or Cr_7C_3, in the composite powders during the sintering procedure were the temperature and oxygen partial pressure in the heating arrangement. The coated Cr-phase either in agglomerated or dispersive condition was restricted by applying colloidal dispersion. The microstructures showed that fine (20 – 600 nm) grains positioned at Al_2O_3 grain boundaries hardly retard the densification of Al_2O_3 matrix in sintering. The tests on toughness, strength, and hardness, appeared that the composites with the inclusions of (Cr_3C_2) had gained the compensation over those by the mixture rule. 8 vol. % ultrafine inclusions have really enhanced the mechanical property. The strengthening and toughening mechanisms of the composites were due to grain size decrease, homogenous distribution of inflexible inclusions, and crack deflection.

Olevsky Eugene et al [8] presented theoretical sintering concept formerly based upon ideas of the distinct quality of particulate media. However, the real sintering kinetics of particulate are determined not only by the properties of the particles themselves and the nature of their local interface with each other, but also by macroscopic factors. Among them are externally imposed forces, constraints *(*e.g. adhesion of the sample's end face and furnace surface*)*, and properties of homogeneity of volume under inspection deficient treatment of the querys enumerated above was individual of the basic reasons restricting the use of theory of sintering. A skilled approach is related with continuum, which has been fruitfully applied to the study of compaction of spongy bodies. This advance is based upon the plastic theory and non-linear-viscous deformation of porous products. Analogous ideas have just been embodied in the theory of continuum of sintering. The chief results of the role of this theory for the explanation of certain scientific troubles of sintering are introduced together with their thermo mechanical aspects.

Park, H. H. et al. [9] quantitatively describes models for liquid stream into isolated pores during LPS. The grains are assumed to keep an equilibrium shape determined by surface tension force and capillary pressure (negative) in the liquid due to menisci at the surface of specimen and the pore. With an raise of grain dimension, the grain sphering force decreases while the diameter of liquid menisci increases to continue the force balance. When growth of grain reaches a critical point, the liquid menisci around a pore become spherical and the force of driving for filling the

pore increases rapidly as liquid tends to flows into it. The critical grain size necessary for filling a pore increases linearly with pore bulk Experimentally, satisfying of isolated pores has been investigated in Fe-Cu powder mixture after LPS treatment and after dipping into a melted matrix .The perceived pour filling behaviors agree with the qualitative prediction based on the models. In Fe-Cu alloy, pour filling is ended by gas bubbles shaped in liquid pockets. By focusing on the development of pores during LPS, it was confirmed that pores are filled by liquid current after a critical incubation time during sintering. Theoretical analysis shows that the critical condition for pour filling is fulfilled when the grains develop to definite size. The theory of the critical grain size established upon the equilibrium between the grains and the liquid menisci. In a fully densified specimen with a limited liquid content, the grains are in contact flattened shape by the liquid menisci at the surface of specimen which act in opposition to the affinity of the grains to become spherical. As the grains size increase, the sphering force will tends to decrease, and the menisci radius will enlarge until it becomes equal to the radius of a pore which may be nearby. An explanation of the pour filling procedure in a system which contains many pores of diverse size will be more multifarious. In this study the grain figure was assumed to be always at the equilibrium state indomitable by the liquid volume fraction. If the grain shape deviates from the local equilibrium state, unevenness between the sphering force and the liquid pressure will come into view and induce a grain figure change. The kinetics of this grain shape change is a separate problem.

Rameshc. S and Srinivasc. k [10] describe DMLS which is popular rapid prototype (R.P technology to produce metal prototypes and intricate shapes in short time. However, processing of metal matrix composites (MMCs) by laser sintering is still in infant stage. Mainly two problems are reported in MMCs these are thermal cracks and de-bonding of reinforcements while processing MMCs by laser sintering process. These problems can overcome by using metallic coated ceramic reinforcements .This paper is aimed at using nickel-coated SiC to develop iron composites by DMLS technique and to characterize its abrasive wear behaviour. Microstructure, micro -hardness and abrasive wear tests were carried out on both DMLS iron and its composites sintered at a laser scan speed of 100 mm/s. Pin –on-disc type machine is used for Abrasion wear tests SiC abrasive papers of grit size 60, 80, and 150 having an average particle size of 268, 192, and 93 m respectively, used in this paper. Load was variation from 5 and 25N

in five steps, with sliding distance and sliding velocity of 540m and 2.5 m/s, respectively was used for all the tests. Scanning electron micrograph and surface roughness observation of worn surfaces have been used. When sic content increase micro hardness also increase and decrease in density of the laser sintered iron–Sic composites. Content of sic in iron matrix increase the abrasive wear resistance of composites. Iron–Sic composites shows excellent abrasive resistance for a given grit size of Sic at all load condition.

Rahimian. M. et al. [11] investigated the effect of sintering temperature, alumina particle size and sintering time on the properties of Al–Al_2O_3 composite. The average particle size of alumina was 3, 12 and 48μm. Sintering temperature and time were in the range of 500–600°C for 30–90 min. A correlation was established between the microstructure and mechanical properties. The investigated properties include density, hardness, microstructure, yield strength, and compressive strength. It was concluded that as the particle size of Al_2O_3 is reduced, the density is enhanced followed by a reduction in density. In addition, at small particle size, the hardness and yield strength and compressive strength and elongation to fracture were advanced, related to coarse particles size of alumina. The deviations in properties of Al- Al_2O_3 composite are dependent on both sintering temperature and time. Prolonged sintering times had an hostile effect on the strength of the composite. Result demonstration the relative density of Al–Al_2O_3 was higher in samples containing fine particle sizes. The highest relative density of 99.95% was observed in specimens sintered at 600°C. The grain size of samples having fine Al_2O_3 particles are lesser and increasing the sintering time to 90min leads to grain coarsening. The highest hardness was 76HB in specimens containing average particle size of 3μm sintered at 600 ◦C for 45 min. Further rise in sintering time to 90min results in reduction in hardness to 59 HB. The finer the particle size of alumina, bigger the compressive strength and elongation. The highest strength was 318MPa, for the composite containing an average particle size of 3μm and sintered at 600 ◦C for 45 min. Further increase in sintering time has an adverse effect on the strength. Extended sintering times and also the use offline alumina in Al–Al_2O_3 composite results in higher elongations. Maximum elongation was observed to be 61.8% in samples containing the average particle size of 3μm.

Shamsuddin et al [12] focused on fabricating and Characterizing composites of iron-chromium alloy reinforced with 5–25 wt. % of alumina Particles fabricated using powder metallurgy. The

diffraction patterns of X-Ray diffraction (XRD) reveal the influence of varying weight % of alumina. Evaluations on the mechanical properties are also being made on the unreinforced iron matrix. The compatibility between matrix and reinforcement was indicated from the microstructure10examination showing homogeneous distribution of alumina particles in the alloy matrix. Micro-hardness was measured using micro-Vickers hardness apparatus.

2.1 Research Gap

As thoroughly study of many research paper and journal summarized in literature review, I found that the research is continuously on progress in the field of development and characterization of sintered magnetic abrasive particles. Sintered magnetic abrasives are used at hogh tool rotationall speed to get high level of surface finish in magneto rheological finishing. Various types of techniques are being done to develop magnetic abrasives to get better surface roughness. The research papers have their own experimental set up and procedure to conduct the experimental analysis and characterize the abrasives. There is large difference between bonded and unbonded magnetic abrasives. Diffusion of particles take place at high temperature during sintering. Research has been done on aluminum based abrasives with CIP as magnetic particle.

2.2 Research objectives

2.2.1. Development of magnetic abrasive particles (MAPs) using solid phase sintering method.

2.2.2. Characterization of magnetic abrasive particles (MAPs) using scanning electron microscope (SEM) and energy dispersive spectrometer (EDS) and X-ray diffraction (XRD).

CHAPTER 3

EXPERIMENTAL WORK

Magnetic abrasive particles (MAPs) have been developed using solid phase sintering method. Carbonyl iron powder of 20 volume% and SiC of 3000 mesh size with 25 volume% have been uniformly mixed in ball mill. The morphology and particle size as well as elemental composition of magnetic abrasive particles have been studied with SEM and EDS. The different phases of magnetic abrasive particles have been studied using XRD. The microstructure of magnetic abrasive particles has also been studied with optical microscope.

Following steps have been followed in present experimental work.

3.1 Selection of powder

25 % volume fraction of SiC with 3000 mesh size and 20% volume fraction of carbonyl iron powder (CIP) of CS grade has been used to make magnetic abrasive powder.

3.2 Powder preparation (mixing)

Mixing has been done in ball mill having 1 liter capacity of stainless jar. A ball mill is category of grinder which is used to blend and grind the material, paints ceramics etc. It works on the principle of mechanical impact. Reduction in size is done as balls drop from the top of shell. Carbonyl iron powder of 20 volume% and silicon carbide abrasives of 3000 mesh size with 25 volume% have been uniformly mixed in ball mill. Stainless steel jar has been used in ball mill with 1:5 Wight ratio of powder and ball.

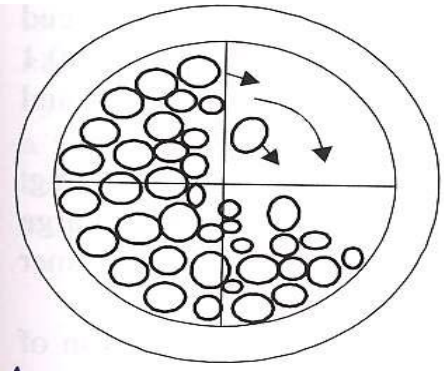


Fig 3.1 Ball mill mechanism

3.2.1. Calculation used for powder preparation

Weight of CS grade of CIP =Volume percentage *100*density of CIP

Weight of CS grade of CIP =0.2*100* 7.87=157.48 gm

Weight of SiC 3000 mesh size = volume percentage*density of Sic

Weight of SiC 3000 mesh size = 0.25*100*3.22=80.5 gm

Total weight of powder =237.9 gram.

Fig.3.2 Ball mill used in present work

3.3 compacting

The pellets of prepared magnetic powder have been prepared with the help of die and hydraulic jack machine by applying 8 ton pressure and this pressure has been held for 25 minutes for each pellet for getting uniform compaction. Hydraulic jack in present used a incompressible fluid (oil or water) for force transmission from one place to another within the fluid system. Hydraulic jack works on Pascal principle which states that pressure intensity is transmitted equally in all directions with the help of fluid at rest.

Pascal principle is stated mathematically as:

$\Delta p = \rho g \Delta h$

Δp =hydrostatic pressure or difference in pressure at two different regions within fluid system .

ρ = fluid density

Δh = difference in elevation between two points in fluid system.

Fig. 3.3 Hydraulic jack machine used for pellets preparation

Specificaton of die used in pellet preparation

Shape - cylindrical

Material - Die steel

Diemeter- 7 mm

Length - 50 mm

Fig. 3.4 Die used in pellet formation

The pellets of 5 gram each have been prepared at 8 ton pressure using cylindrical die before solid phase sintering of magnetic abrasive particles. These pellets have been placed in alumina trays as shown in figure 3.5.

Fig.3.5 Pellets before sintering

3.4 solid phase sintering of pellets

3.4.1Sintering

Sintering refers to the heating of green compact in an oven .The heat is supply to unite the various grains into a single pass, thus developing the strength. Sintering is done to achieve all possible final strength and hardness needed in finished product. Sintering is done by heating the compact product upto 70 to 80% of melting temperature in an inert or reducing atmosphere. Various types of sintering processes are shown in fig. 3.6.

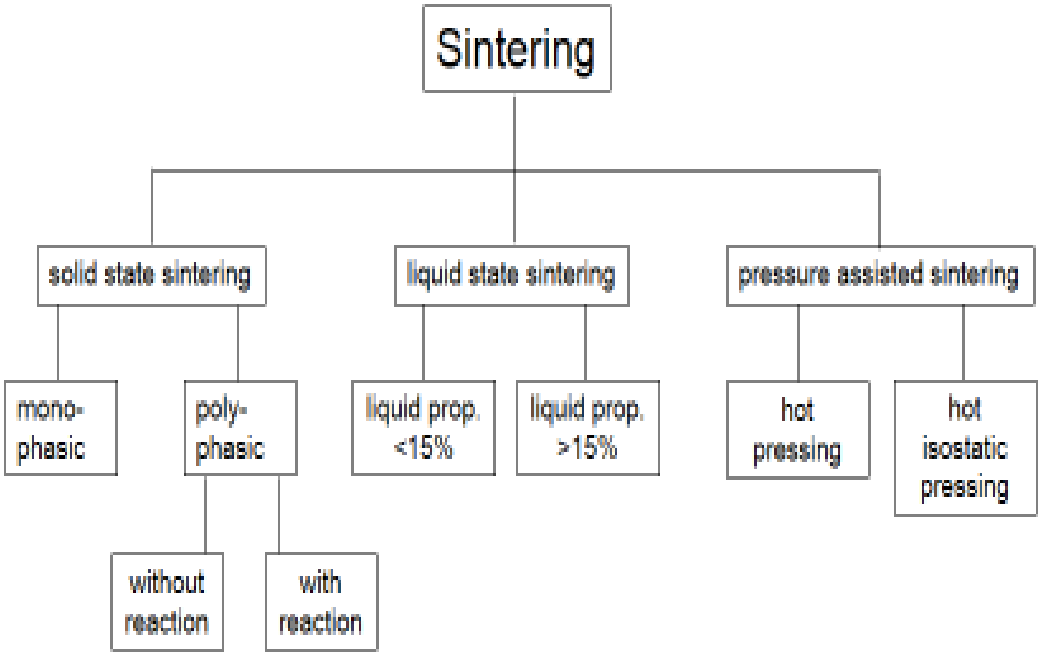


Fig.3.6 Classification of sintering process

Sintering mechanisms

Sintering is mainly categorized under two types: Solid-state and liquid phase sintering (LPS). In the former, individual powder particles bond through an array of solid-state diffusion mechanisms. In the liquid phase sintering, a secondary, liquid phase is produced that typically intensifies densification through the combined action of capillary forces and enhanced kinetics of diffusion.

3.4.1.1 Solid state sintering

This type of sintering inherently involves only solid state diffusion mechanisms and is therefore relatively slow when compared to LPS. The means by which antiparticle bonding and densification occurs includes four mass transport mechanisms: evaporation-condensation, surface diffusion, volume diffusion and grain boundary diffusion. These concepts can be illustrated using the classical "two-sphere model" wherein two particles are in contact with a joining neck "X" formed between them as shown in fig. 3.7 [13].

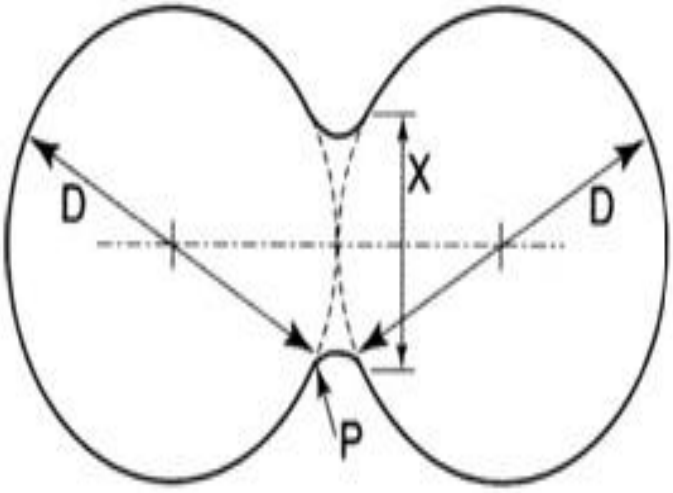


Fig.3.7 Two-sphere model illustrating the terminology used to describe the solid state sintering of two spherical particles [13].

The neck between the two particles manifests itself as a concave face, while the particle is convex. This curvature differentiation develops a little vapour pressure difference between these two surfaces (high at convex and low at concave) and a localized departure from equilibrium. The corresponding pressure gradient causes vapour to migrate from the convex face to the concave region of particle contact where it then condenses and deposits new atoms so

as to increase the size of the neck [14]. This evaporation-condensation action merely forms the neck between the particles and causes little densification. Surface diffusion is similar to evaporation-condensation, in that the curvature of the particles is again the driving force. Here, a locally high vacancy concentration exists at the concave neck surface whereas much lower concentration prevails within the convex area. The compact attempts to eradicate this gradient through the diffusion of vacancies away from neck. This is always accompanied by a counter-current flux of mass into the neck between the particles cause it to grow. Both mechanisms are functions of the powder and neck curvatures and become sluggish as the neck grows and concavity is diminished. Upon heating to higher temperatures, bulk transport mechanisms denoted as volume diffusion and grain boundary diffusion become operative. These are responsible for the majority of shrinkage and densification that occurs during solid state sintering. Volume diffusion is similar to surface diffusion, but surface atoms exchanging with vacancies in neck, the migrating atoms diffuse within the mass of the material instead. Vacancies are transported to, and in the end annihilated, at the grain boundary between the particles. Similarly, grain boundary diffusion involves the movement of atoms along the particle grain boundary. Vacancies move inward from the concave neck surface, while a concurrent flow of atoms occurs towards the neck surface. The four solid-state sintering mechanisms are conceptually shown in fig. 3.8. The arrows represent the flow of materials during the appropriate mechanisms.

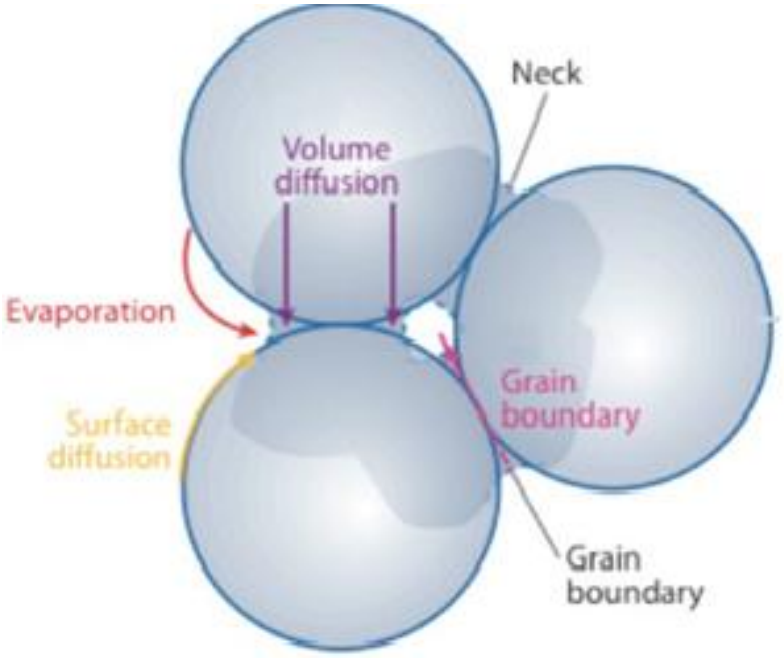


Fig. 3.8 Four solid-state sintering mechanism

In the present work, solid phase sintering has been done in the furnace with argon as inert gas. Set up consist of furnace having programming software to set the sintering cycle and attachment of argon cylinder. solid phase sintering is based upon a sintering cycle. sintering cycle has been explained with the help of a line graph drawn between tempertature and time as shown in fig. 3.9. At initial stage of cycle, heating is done at the rate of 200/h upto 800^0C temperature. After this, constant temperature heating has been done for 1 hour. Now the temperature is increased at the same rate upto 1000^0C temperature and held at this temperature for four hours for getting sintered product completely. The furnace cooling is done at the same rate in the inert atmosphere of argon till the room temperature of 30^0C achieved.

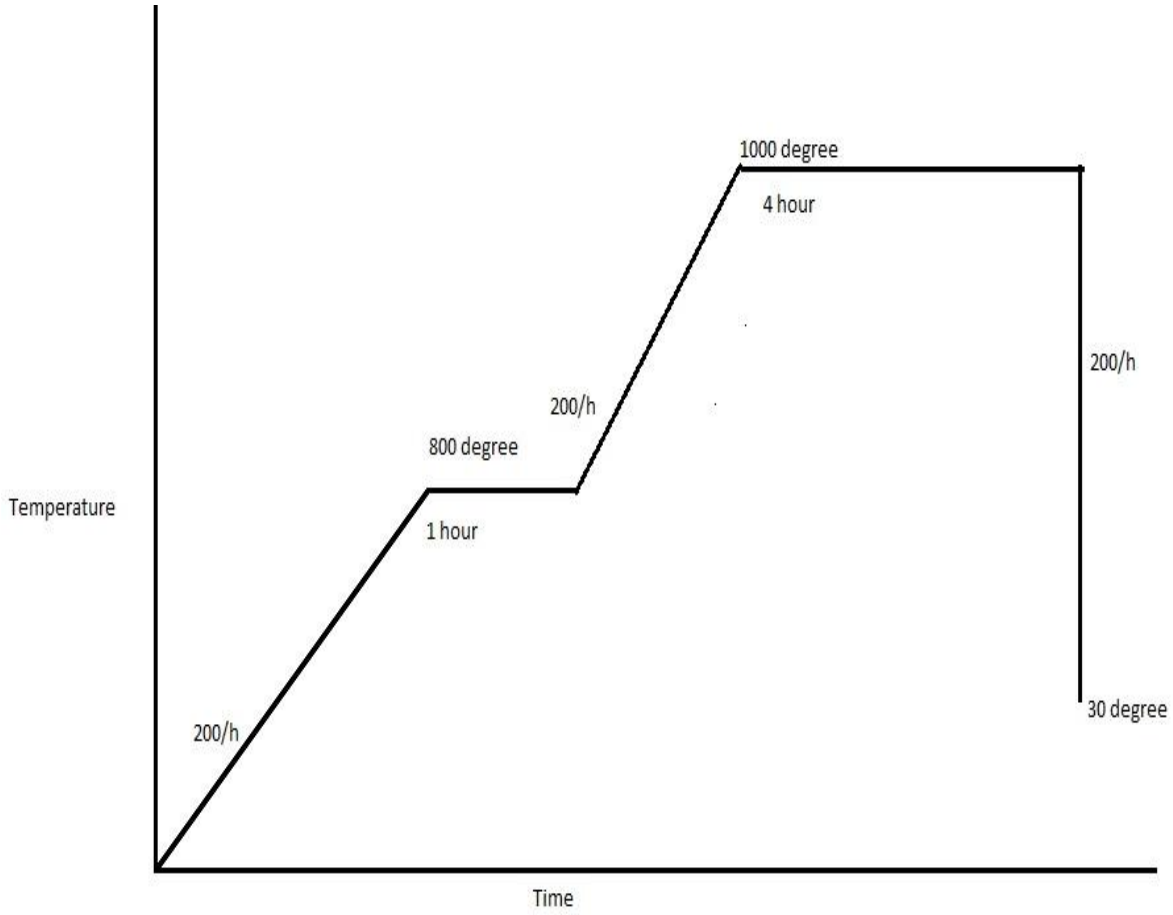


Fig. 3.9 Sintering cycle used in present work

Programme Mode used in solid phase sintering

Step-1: Press enter twice, display show$\frac{3}{prog}$.

Step-2: Choose programs by up and down arrow.

Step-3: To enter program press up and enter simultaneously.

$\frac{prog\ ,cont\ ,end}{set}$ (shown on display)

Step-4: When show program then press enter.

Step-5: Hold dwell (d) - put rate value ZERO then press enter.

Step-6: Enter cycle.

Step-7: To end program put value -1 or value less than zero

OR

Press down arrow which automatically show end.

Step-8: $\frac{End}{06E}$ and then press enter.

Step-9:$\frac{1}{cycle}$ and then press enter.

Step-10: $\frac{min\ /hours}{base}$ (change by up and down arrow). And then press enter.

Step-11: $\frac{!!!!!!}{E-01}$ (don't disturb or ignore it) and press enter.

Step-12: $\frac{0}{loc}$ exit.

Step-13: press up and enter simultaneously.

$\frac{prog\ ,cont\ ,end}{set}$ press when show end then press enter.

Step-14 press run twice to start program(two digital red dots is on it means program run).

Step-15: put sample inside furnance and start MCB.

Step-16: press run at least 7 to 8 second to end the program.

Fig.3.10 High temperature tube furnace

There is change in colour of pellets after sloid phase sintering. Figure 3.11 shows the condition of sintered pellets. Soft pellets become hard after sintering process .

Fig.3.11 Pellets after sintering

3.4.1.2 Liquid-phase sintering

Where some liquid that is present at sintering temperatures aids compaction. Grain rearrangement occurs in the first stage followed by a solution-precipitation stage. Usually, the liquid amount is not enough to fill the green-state porosity in normal liquid-assisted ceramic sintering. In many instances, apparently Solid State Sintering proceeds in the presence of previously undetected (or transient) small amounts of liquid (perhaps introduced as impurities during the powder preparation stage, such as silicates in oxide ceramics) [15].

3.5. Crushing of sintered pellets in ball mall.

Sintered pellets have been crushed in ball mill as shown in fig. 3.12. There is decrease in size of pellets after crushing in ball mill with the help of stainless steel balls.

Fig. 3.12 Ball mill used for crushing of sintered pellets

3.6 Characterization of Sintered product

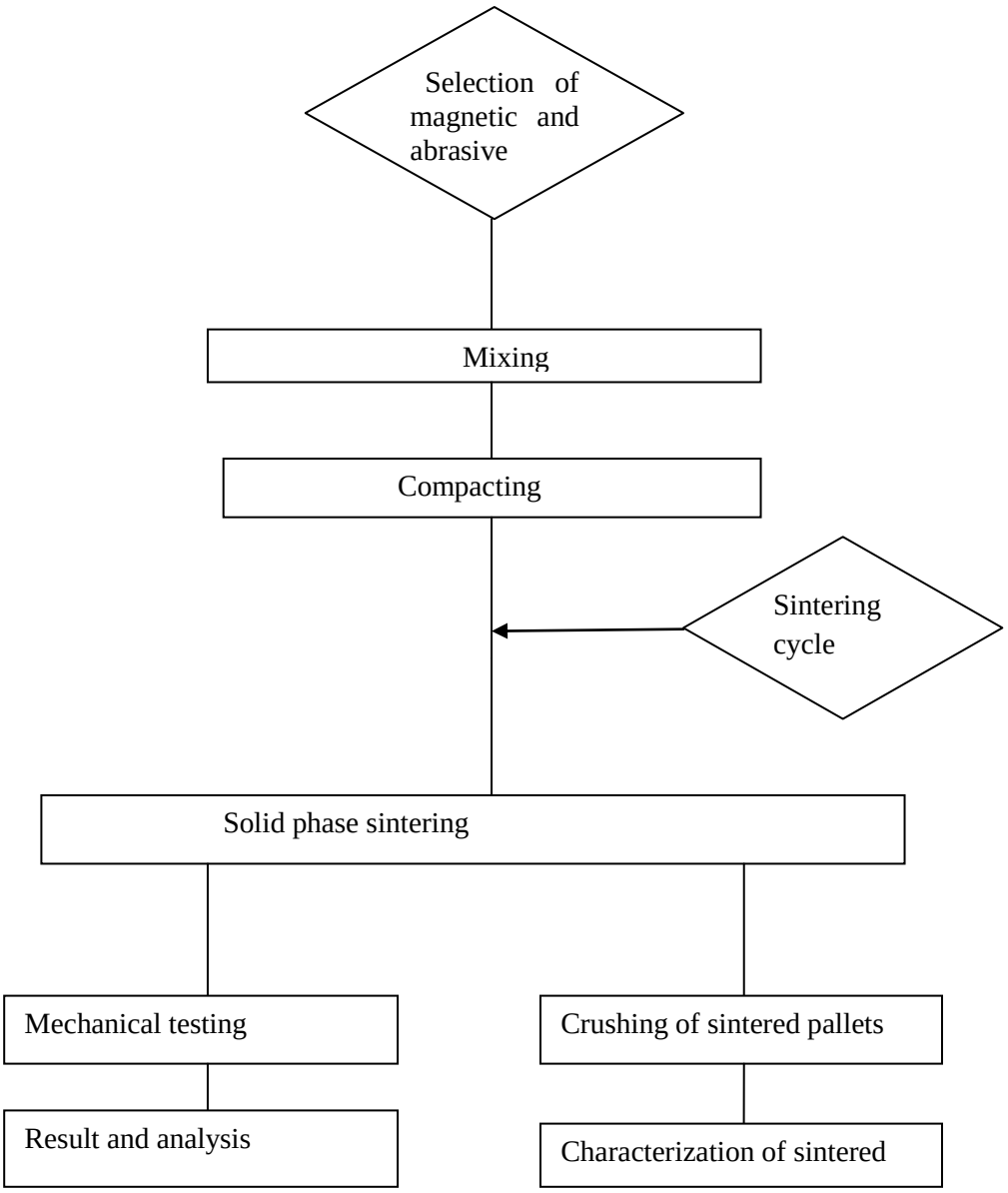
After development of magnetic abrasive particles (MAPs) by sintering method, the particle characterization has been done on SEM and X-ray diffraction to study their entire morphology, shape, size, surface area, phase constitution and microstructure. Additionally, one could also characterize the change behavior of the powders on heat treatments. The measurement of crystal size and lattice strain in the powders is very important since the phase structure and transformation characteristics appear to be significantly dependent on them. In principle, the general techniques used to characterize conventional powders can also be used to characterize the mechanically alloyed powders. The size and shape of the particles may be determined accurately using direct methods of either scanning electron microscope (SEM) for relatively coarse powders or transmission electron microscope (TEM) for fine powders. A powder particle may consist of several individual particles. Further, an individual powder particle may contain a number of crystallites defined as coherently diffracting domains.

Microscopic examination normally gives the particle size (or even grain size if sufficient resolution is available), whereas diffraction techniques (e.g., X-ray) give the crystallite size. It will be shown later that MA and MM have produced amorphous phases in several alloy systems. Differentiation between a "truly" amorphous (i.e., without translational symmetry as in a liquid) and microcrystalline structure (i.e., an assembly of randomly oriented fragments of a bulk crystalline phase) has not been easy on the basis of diffraction studies; considerable confusion exists in the literature. In diffraction experiment of an "amorphous" structure, the intensity but not the phase of the scattered radiation is measured. Fourier inversion of these data can yield only the radial distribution function of the structure that cannot uniquely specify the atomic positions. To determine the structure, the experimentally determined radial distribution function must be compared with the radial distribution functions calculated from the structural models being considered. The occurrence of an amorphous phase is generally inferred by observing the presence of broad peaks in the X-ray diffraction patterns. It should be noted, however, that X-ray diffraction patterns present only an average picture. Thus, by observing the broad X-ray peaks alone, it is not possible to distinguish amongst materials which are (a) truly amorphous, (b)

extremely fine grained, or (c) a material in which very small crystals are embedded in an amorphous matrix. Hence, in recent years, it has been the practice to recognize such observations as “X-ray amorphous”, suggesting that the identification was done only by X-ray diffraction methods. There have been several examples of observation of a phase produced as “amorphous” on the basis of X-ray diffraction studies alone; but, based on supplementary investigations by neutron diffraction and transmission electron microscopy, it could be unambiguously confirmed that the phase produced was not truly amorphous and . Neutron diffraction techniques have the Mechanical alloying and milling advantage that one can detect light atoms in the presence of heavy atoms. Additionally, the technique of neutron diffraction has the ability to distinguish between neighboring elements in the periodic table. X-ray diffraction techniques will be unsuitable for this because neighboring elements will have the atomic scattering factors very close to each other and so their difference will be very small to detect any reasonable amount of scattering. Thus, it is desirable that the X-ray diffraction observations are confirmed by other techniques as well. For example, transmission electron microscopic studies can confirm the lack of contrast in the micrographs for a truly amorphous phase. The appearance of a glass transition temperature during differential thermal analysis (DTA) studies is a clear and unambiguous indication of the presence of an amorphous phase. But, a glass transition may be obscured by the onset of crystallization (since the glass transition and crystallization temperatures are very close to each other in many metallic systems) and hence may not be observed in all cases. However, DTA studies can show the presence of an exothermic peak on heating the sample indicating that crystallization of the amorphous phase has occurred. If the material is extremely fine grained (nano crystalline, and not amorphous), then only grain growth, driven by the decrease in grain boundary free energy, can occur in these alloys on heating them to high temperatures. In this case, a monotonically decreasing isothermal signal will be observed in the DTA studies. Other indications of grain growth are scanning peaks that are low and wide with a long high temperature tail; further, these peaks shift to higher temperatures after pre annealing and thus one should be able to differentiate between amorphous and microcrystalline (fine grained) samples processed by MA techniques by using a combination of techniques such as microscopy, diffraction, and thermal analysis. The sintered samples were studied for phase analysis of Sic and CIP, shape and size has been determined during characterisation process .The different phases have been studied for prepared samples using X-ray diffraction (XRD) .The morphology and

elemental composition as well as particle size has been studied using scanning electron microscope (SEM). Elemental composition has been studied using Energy dispersive spectrometer (EDS). Microstructure of samples has been studied using optical microscop[16].

FLOW CHART- In flow chart whole process is shown in systematic manner. In other word flow chart is a mirror image of whole process.



3.13 Flow chart of whole process

3.6.1 SCANNING ELECTRON MICROSCOPY

SEM has been done to study the morphology of sintered and unbonded magnetic abrasives. The scanning electron microscope has a resolution of about 10 nm (100 Å) and is capable of very low magnification (about 10X) up to about 50,000X. Therefore, it can be used to count particles ranging in size variation from 1 mm to 0.1μm. Particles less than 0.1μm usually have too low a contrast with the background to be counted efficiently. The scanning electron microscope has about 300X the depth of field of an optical microscope. The image in the scanning electron microscope is usually obtained by using the secondary electron output of the sample as it is scanned by a very narrow electron beam. The contrast of the image depends more on the topography of the sample than on differences in atomic number. Therefore, prepared powders must not be embedded in films, but dispersed on a smooth substrate. Any smooth surface can be used as a substrate. However, if energy dispersive x-ray analysis (EDXA) is to be performed for particle identification, a carbon or polystyrene surface is preferred. An excellent substrate can be made by placing a polystyrene pellet on a glass slide and heating it on a hot plate until it softens. A second glass slide is then placed over this slide and pressed until the pellet forms a thin disk. The slides are removed from the hot plate and pressed together until the polystyrene sets. The disk thus formed is as smooth as the glass and contains no elements that may hinder EDXA. For sample preparations using aqueous suspensions, polystyrene surfaces can be rendered hydrophilic by a brief treatment in oxygen Asher at low power (5 to 10 W for 5s). While the substrates for SEM do not have to be as thin as those used for TEM, they must be conductive. Consequently, if glass or plastic surfaces are used, they should coated with a metal which evaporates (or carbon, for EDXA) film. This coating is usually applied after the particles have been dispersed on the surface. Many of the dispersing techniques used for TEM can be applied to SEM. Particle dusting, drying from liquid suspensions, and mulling in liquids that can be sublimed in a vacuum are suitable dispersing methods, depending on the powder. If the technique of mulling in parlodion and amyl acetate is used, parlodion can be removed in an oxygen Asher, thus leaving the particles on the substrate. Suitable substrates include glass or metal, because they are not affected by the aching. The prepared sample should always be placed in the scanning electron microscope with the surfaces normal to the electron beam so that the

magnification, which changes with working distance, will be the same on all areas of the viewing screen.

Fig. 3.14 Scanning Electron Microscopy (SEM)

3.6.2 X-RAY POWDER DIFFRACTION (XRPD)

(XRPD) techniques are used to characterize samples in the form of aggregates of finely divided matter. Various investigations are covered by these techniques, including qualitative and quantitative phase identification and analysis, determination of crystalline, micro identification, lattice parameter determinations, high temperature studies, thin film analysis and in crystal structure analysis. The powder method, as it is referred to, is perhaps best known for its use as a phase characterization tool partly because it can routinely differentiate between phases having the same chemical composition but different crystal structures. Though chemical study can represent that the empirical formula for a given sample is $FeTiO_3$, it cannot determine whether the sample is a mixture of two phases (Feo and one of the three polymorphic forms of TiO_2) or

whether the sample is the single phase mineral $FeTiO_3$ or limonite. The ability of XRD to execute such identifications very easily and routinely than that of other analytical method explains its importance in many industrial applications as well as its wide availability and prevalence. In general, an x-ray powder diffraction characterization of a substance consists of placing a powder sample in a collimated monochromatic beam of x-radiation. The diffraction pattern is recorded on film or using detector method, then analyzed to get x-ray powder information that can be used to solve such problems. In XRD analysis, samples usually exist as finely divided powder (usually less than 44μ m in size) or can be reduced to the form of powder. The particles in a given sample consist of one or more independently diffracting regions that coherently diffract the x-ray beam. These small crystalline regions are called as crystallites. Consolidated bodies, such as ceramic or as-received metal samples will likely have crystallites small enough to be useful for powder diffraction analysis, although they can appear to have considerably larger particle sizes. This occurs because a given grain or particle can consist of several crystallites (independently diffracting regions). Although larger grain sizes can sometimes be used to advantage in XRD, the size limitation is important because most applications of powder diffraction rely on x-ray signals from a statistical sample of crystallites. The angular position θ of the diffracted x-ray beam depends on the spacing's, *d*, between planes of atoms in a crystalline phase and on the x-ray wavelength λ:$N\lambda = 2d\sin\theta$ .A diffraction pattern can be recorded using film, analog, or digital methods. Whether film, analog or digital data collection is used, the final data can be displayed as a graph of intensity, as a function of inter planar distance *d*, or as a function of diffraction angle 2θ. Many modem automated powder diffraction meters can provide further data reduction, including peak finding. A tabular listing of peak intensity versus inters planar spacing, search/match software, and other computer utilities.

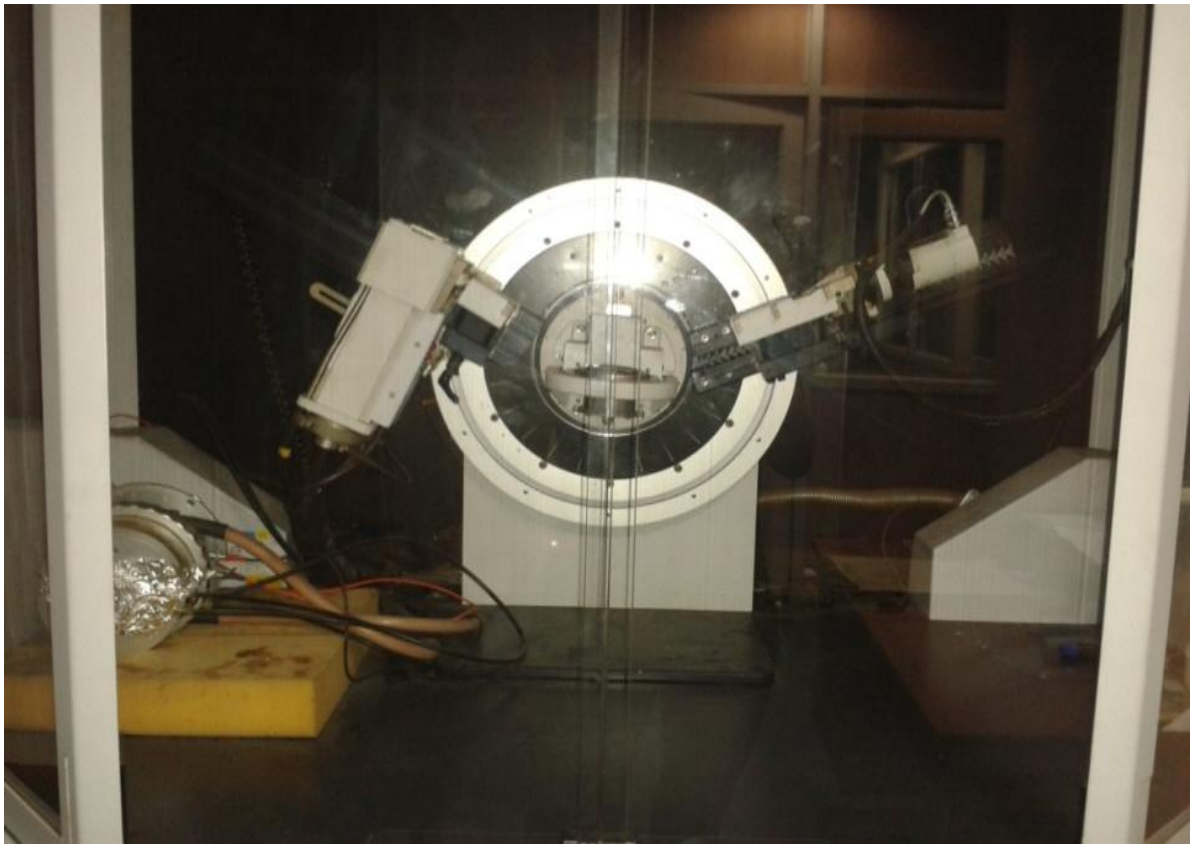


Fig.3.15 X-ray diffracto- meter (Bruker AXS D8 Advance instrument)

3.6.3 MICROSTRUCTURE

Microstructure was observed by Olympus GX 41 Microscope with META-Lite software. In this microstructure analysis the whole process consists of following steps.

- Dry polishing.
- Wet polishing- In wet polishing silicon powder and water was used.

Etching- In this process, solution of HNO_3+alcohol was used. The purpose of etching to remove thin layer on the surface. Secondly, the etchant attacks the surface with preference for those sites with the highest energy, leading to surface relief which allows different crystal orientation, grain boundaries, and defects to be distinguished in reflected light microscopy. Olympus GX 41 microscope has magnification range from 25X to 100X.

Fig. 3.16 Olympus GX 41microscope

CHAPTER 4

RESULT AND DISCUSSION

4.1 SEM analysis

The SEM topography of unbounded magnetic abrasive particles shows that, the carbonyl iron has spherical morphology and silicon carbide has sharp edges. Fig. 4.1 gives diagonally particle size at 10 micron resolution and 3500 X magnification for unbonded magnetic abrasive particles. Magnetic abrasive particle size ranges from 1.4-2.94 μm. The average particle size for unbonded magnetic abrasive particles with respect to diagonal is 2.11μm Unbonded magnetic abrasive particles have no bond between CIP and the abrasive particles.

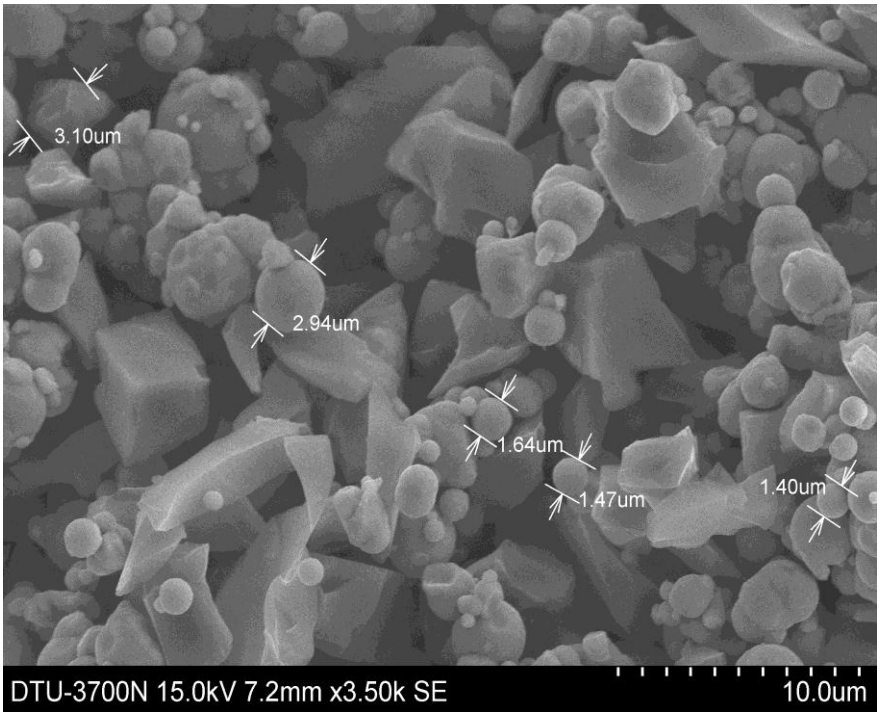


Fig 4.1 SEM micrograph of unbonded magnetic abrasive particles

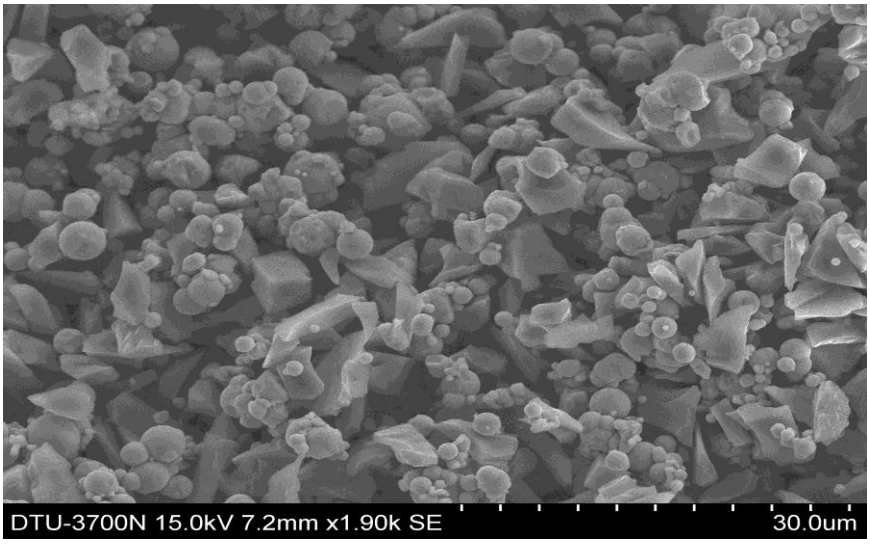


Fig. 4.2 SEM micrograph of unbonded magnetic abrasive particles

SEM analysis of sintered sample

SEM investigates the morphology of bounded magnetic abrasive particles. SEM shows that the sharp edges of magnetic abrasive particles are obtained after crushing the sintered pellets in ball mill. The silicon carbide abrasive (SiC) particles are diffused on the surface of carbonyl iron particles during sintering and make sintered magnetic abrasive particles (MAPs). The particle size varies from 2.94 to 9.23 micron and average particle size is has been found as 6.33micron. SEM shows that the size of the particles is nonuniform. Abrasive and magnetic particles are bonded due to bond formation between grain of SiC and CIP. There exist closeness between two particles due to very high compaction pressure.

Four SEM images of sintered sample have been taken at resolution of 20 micron, 5 micron, 10 micron and 100 micron and 2700X, 6500X, 4000Xand 470 X magnifications respectively. Dimensional analysis of SiC abrasive particles has been done at 20 micron resolution out of four SEM images of sintered sample. Figure 4.3 gives diagonally particle size at 20 micron resolution and 3500X magnification.

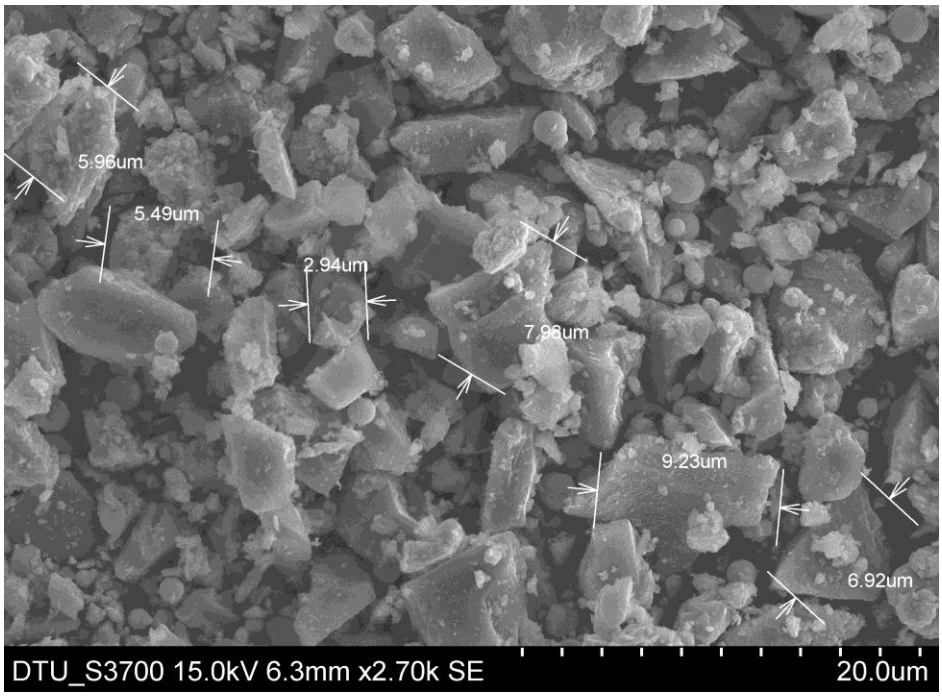


Fig. 4.3 SEM micrograph of sintered magnetic abrasive particles

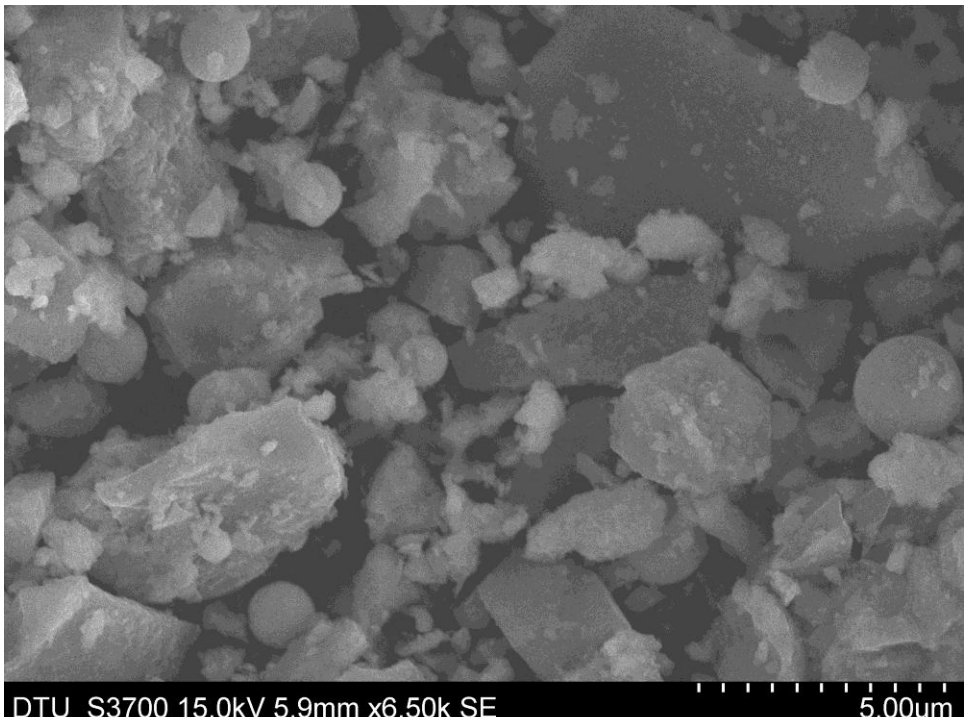


Fig. 4.4 SEM micrograph of sintered magnetic abrasive particles

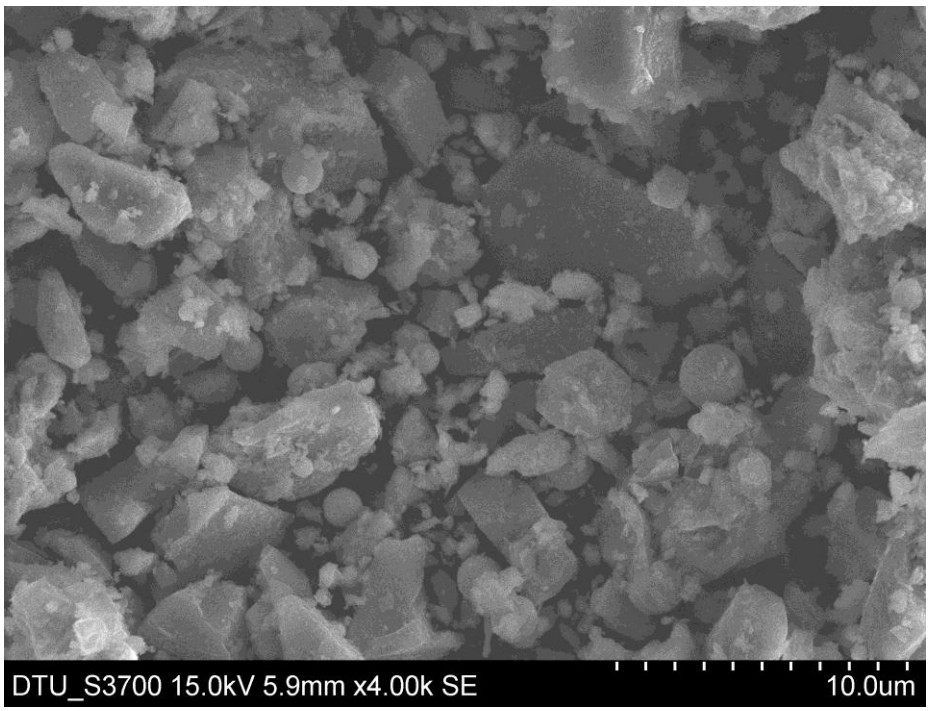


Fig4.5 SEM micro graph of sintered magnetic abrasive particles

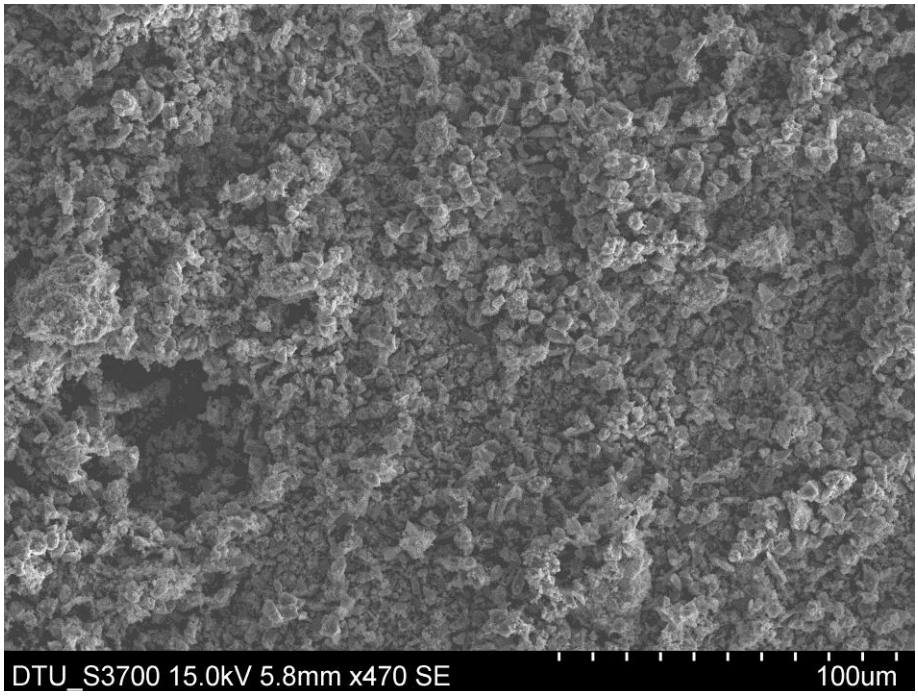


Fig. 4.6 SEM micrograph of sintered magnetic abrasive particles

4.2 EDS Analysis.

EDX analysis has been done on sintered magnetic abrasive particles and shows the presence of different elements present in the powder. It investigates that silicon and iron are major elements in the magnetic abrasive powder. Minor elements detected include oxygen. presence of oxygen shows that some oxidation has been done during sintering process.

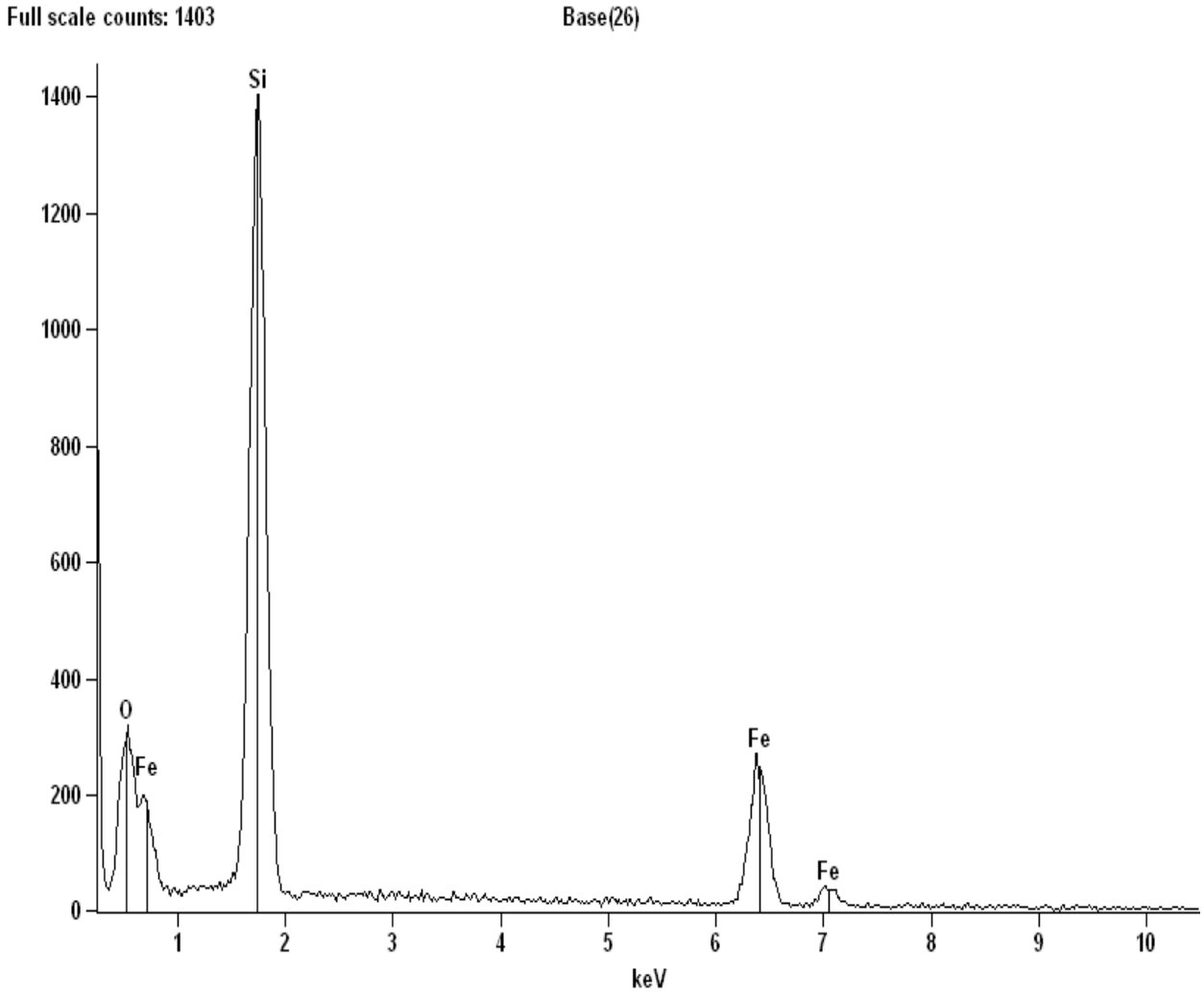


Fig. 4.7 EDS graph of solid sintered sample

Table 4.1 Quantitative results of EDS

Element Line	Net Counts	Int. Cps/nA	Weight %	Weight % Error	Atom %	Atom % Error	Formula	Standard Name
O K	10319	---	27.81	+/- 0.49	48.62	+/- 0.85	O	
Si K	20669	---	30.77	+/- 0.35	30.64	+/- 0.35	Si	
Si L	0	---	---	---	---	---		
Fe K	5131	---	41.42	+/- 1.28	20.74	+/- 0.64	Fe	
Fe L	6552	---	---	---	---	---		
Total			100.00		100.00			

4.3 Phase evolution of sintered particle

XRD Analysis Displayed the crystal structure of the abrasive particles. The diffraction peaks are relatively sharp and the scope of amorphous peaks is relatively small.

Sample Name	Sintered Magnetic Abrasive Particles Of SiC and CIP
Crystal system	Hexagonal Lattice
Type	P
Lattice Parameter	a= 4.9168 b= 4.9168 c= 5.4089
Lattice Parameter	Alpha= 90 Beta= 90 Gama=120
Radiation	Cu
Wavelength	1.540598micron
2Theta Start	10
2Theta End	80
d	interplaner distance

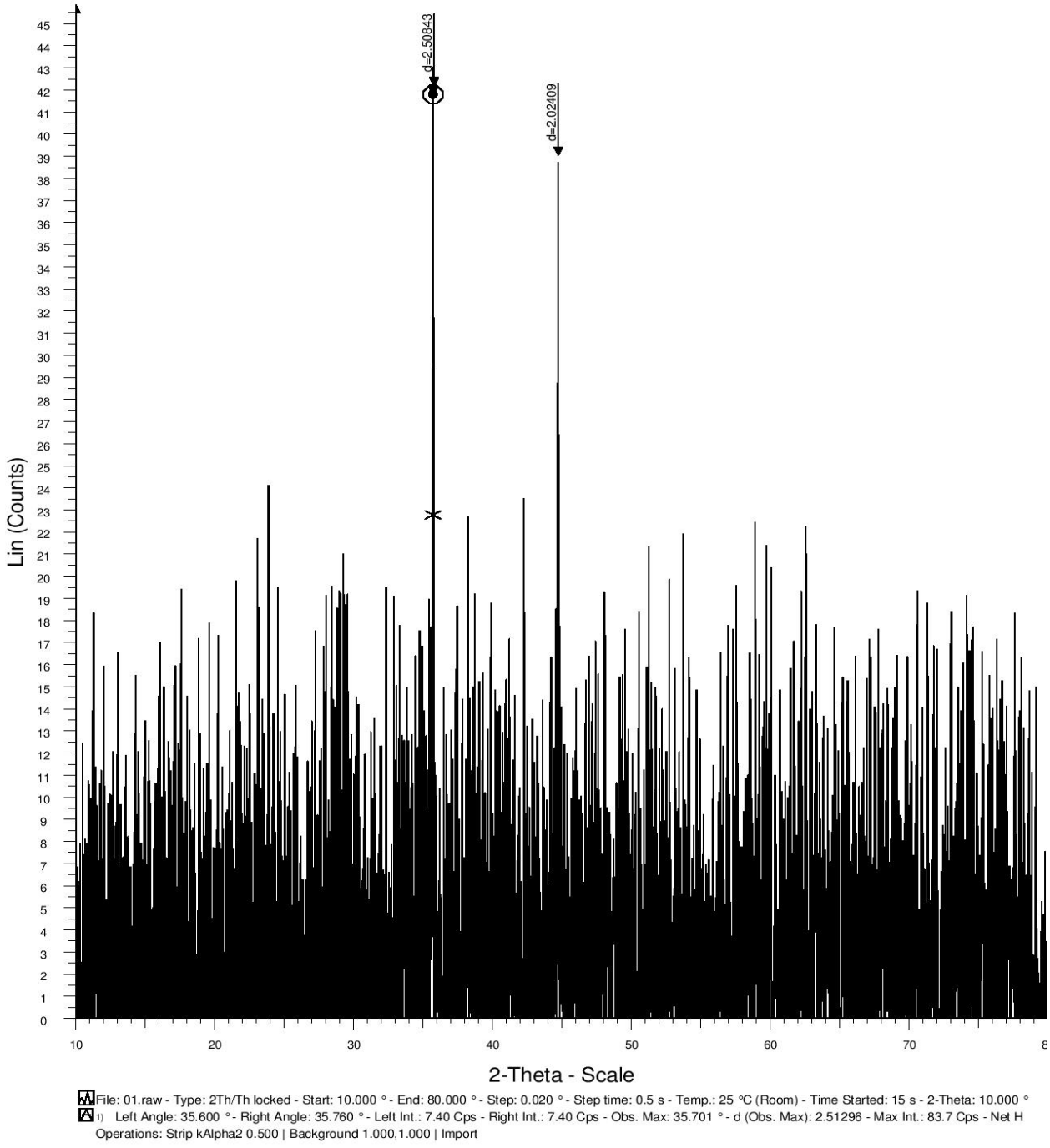


Fig. 4.8 XRD pattern for solid sintered abrasives

Table 4.2 Lines generated during XRD analysis

H	K	L	d	2 theta
0	0	1	5.40890	16.375
1	0	0	4.25807	20.845
1	0	1	3.34573	26.622
0	0	2	2.70445	33.097
1	1	0	2.45840	36.520
1	0	2	2.28291	39.440
1	1	1	2.23807	40.263
2	0	0	2.12904	42.422
2	0	1	1.98109	45.763
1	1	2	1.81913	50.104
0	0	3	1.80297	50.585
2	0	2	1.67286	54.835
1	0	3	1.66027	55.286
2	1	0	1.60940	57.191
2	1	1	1.54256	59.916
1	1	3	1.45388	63.987

The XRD pattern of sintered SiC and CIP is shown in fig.4.8.From these figure first peak comes at 21.54°. This peaks belongs to Fe_2o_3 and has (1, 0, 0) h, k,l value, his plane is simple cubic. Second phase is at 35.767° of SiC has (1, 1, 0) h k l value and his plane is simple cubic. Third peaks is at 44.735 of Fe_3o_4 has (2, 0, 1) h k l values and his plane is FCC. Highest Peak has 2.50843Å interplaner distance.

4.4 Micro structure Analysis

Micro structure analysis was observed by optical microscope with 100x magnification of 8 ton. From fig.4.9 it is clear that there are two phase present in the sample. In the coloured part is Sic and the red portion is iron.

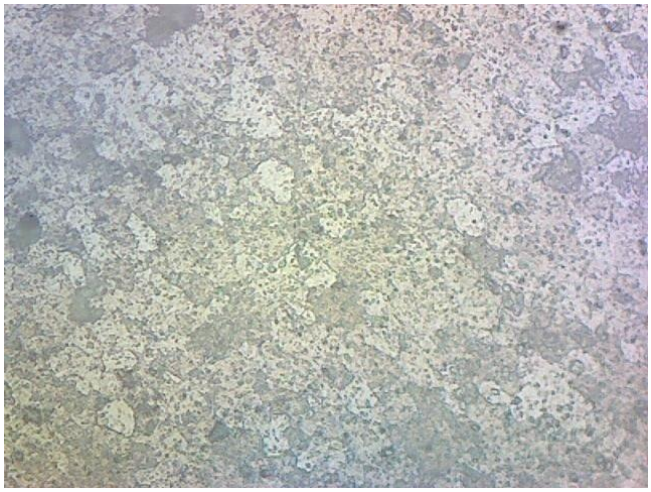


Fig. 4.9 Microstructure of sintered pellet

CHAPTER 6

CONCLUSION

The present work on the powder processing and characterization on SEM, XRD, EDS and mechanical properties of SiC- CIP composite has the following conclusions.

1. The SiC and CIP based sintered magnetic abrasive particles were developed by solid phase sintering method.

2. Scanning electron microscopy (SEM) analysis shows the morphology, shape and size of magnetic abrasive particles (MAPs) after sintering and crushing. It has been observed that the abrasive particles are diffused on the surface of carbonyl iron powder and make magnetic abrasives. The size of particles has been seen diagonally on the micrograph to calculate the average particle size. Maximum size of particle in diagonal is 9.23 micron and average particle size is 6.333 micron.

3. XRD Shows crystalline structure and different phases present in the sample and lattice parameter. XRD study shows simple cubic, face cubic centred, and body cubic centred planes present in the sample.

Future work

The present study reveals that powder processing is an important step for achieving a dense microstructure. The magnetic abrasive can be used for finishing purpose in MAF, MRF and RMAF. The magnetic abrasive will be used in aerospace and electronics industries for finishing of hard material which is not easy to finish by conventional methods.

REFERENCES

[1] suryanarayna.c “Mechanical alloying and milling ”,Progress in Material Science,1-184 (2001).

[2] K. Nihara, “New Design Concept of Structural Ceramics: Ceramic Nano composites”, J. Ceram. Soc, 974-982 (1991).

[3] Lenel F.V “Powder metallurgy principles and applications”, New Jersey, USA: Metal Powder Industries Federation,683-690 (1980).

[4] Palwinder singh and lakvirsingh “internal finishing of cylindrical pipes using sintered magnetic abrasive”, . International Journal of Engineering Science and Technology (IJEST),43-569 (2008).

[5] Ulrich Degenhardt , Frank Stegner c, Christian Liebscher b, Uwe Glatzel b, Karl Berroth c, Walter Krenkel a, Günter Motza “ sintered silicon nitride/nano-silicon carbide materials based on preceramic polymers and ceramic powder ”Journal of the European Ceramic Society1893–1899(2012).

[6]Upadhayaya and Nekatibeb“Effect of heating mode on sinterability of carbonyl iron compacts”89 -92 (2013).

[7] Chen Lung Chih et al. “Sintering behavior and mechanical properties of nano-sized Cr_3C_2/Al_2O_3 composites prepared by MOCVI process” Journal of the European Ceramic Society 2883–2886 (2002).

[8] Olevsky, Eugene A. “Theory of sintering: from discrete to continuum.”Materials Science and Engineering 41–100 (1998).

[9] Park, H.H., Cho, S.J., Yoon, D.N “Pore filling process in liquid phase sintering.”,

Metallurgical transactions 1075-1080 (1984)

.

[10] Ramesh c.s ,srinivasc.k “Abrasive behaviour of laser sintered iron –Sic composite . ”,An international journal on the science and technology of friction lubrication Wear 1777–1783 (2009).

[11]. Rahimian, M. et al. (2009). “The effect of particle size, sintering temperature and sintering time on the properties of Al–Al_2O_3 composites, made by powder metallurgy.” Journal of Materials Processing Technology 5387–5393(2009).

[12].Shamsuddinsaidatulakmar et al “ Characterization of Fe-Cr-Al_2O_3 Composites Fabricated by Powder Metallurgy Method with Varying Weight Percentage of Alumina” Journal of physical science 89-95(2008).

[13] R. M. German, Powder Metallurgy & Particulate Materials Processing, Princeton, New Jersey: Metal Powder Industries Federation,(2005).

[14] J. E. Garay, "Current-activated, pressure-assisted densification of materials," Annual Review of Materials Research 445-468(2010).

[15] G. B. Schaffer, T. B. Sercombe and R. N. Lumley, "Liquid phase sintering of aluminum alloys," Materials Chemistry and Physics 85-91 (2001).

[16] R. W. Cooke, R. L. Hexemer, I. W. Donaldson and D. P. Bishop, "Dispersoid strengthening of Al-Cu-Mg P/M alloy utilising transition metal additions," Powder Metallurgy 191-199(2012).