CHARACTERIZATION OF Fe-P SOFT MAGNETIC ALLOYS

A DISSERTATION

Submitted in partial fulfillment of the
requirements for the award of the degree
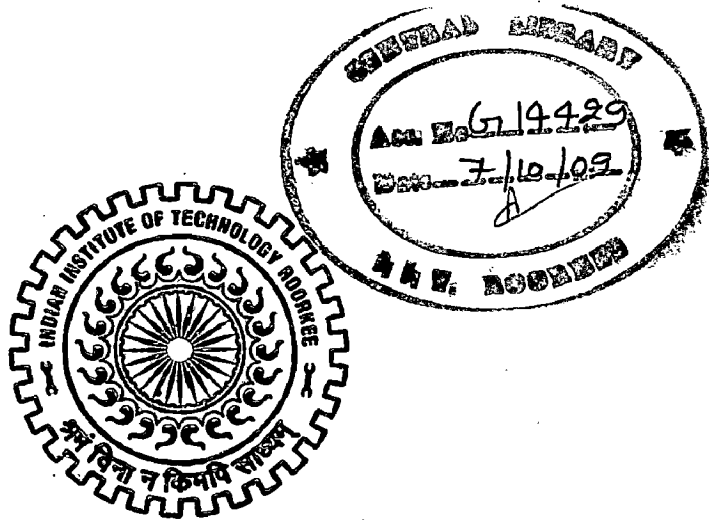*of*

MASTER OF TECHNOLOGY

in

METALLURGICAL AND MATERIALS ENGINEERING
(With Specialization in Industrial Metallurgy)

By

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CANDIDATE'S DECLARATION

I hereby declare that the work presented in this dissertation entitled "CHARACTERIZATION OF Fe-P SOFT MAGNETIC ALLOYS" in partial fulfillment of the requirements for the award of MASTER OF TECHNOLOGY, in Industrial Metallurgy submitted in Department of Metallurgical and Materials Engineering, Indian Institute of Technology,Roorkee, is an authentic record of my own work carried out during the period June 2008 to June 2009, under the supervision of Dr.U.Prakash,Associate Professor, Department of Metallurgical and Materials Engineering, Indian Institute of Technology ,Roorkee.

The matter embodied in this dissertation has not been submitted by me for the award of any other degree.
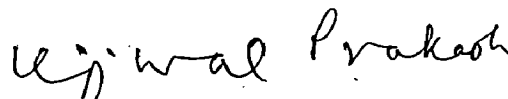
Date: June, 2009

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CERTIFICATE

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

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Place: Roorkee   (Shailesh Kumar Chaurasia)

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III

ABSTRACT

Magnetic materials play a fundamental role in designing electrical and electronic machines, equipments and devices and basically it is the material and secondly the design, which controls the performance and efficiency of a device. Therefore, magnetic material development, which is closely associated with the technology being followed for its manufacturing shall lead to an improvement in the performance of electrical/electronic equipments/devices.

Iron-phosphorous alloys are soft magnetic materials and magnetic properties and toughness both improve with increase of phosphorous content for these alloys as for as phosphorous remains in solid solution form in α-iron, which is about 1.2%.Phosphorous alloying addition improves the resistivity of the iron and decreases the coercivity effectively and thus decreases hysteresis loss of the alloy. It also improves alloys formability. But it is necessary that entire phosphorous remains in solid solution with iron. This is not possible in wrought metallurgy owing to its sensitivity to segregation during solidification. Introduction of phosphorous in iron is therefore only possible with Powder Metallurgical processing.

Phosphorous is an undesirable impurity in wrought steels. It is responsible for cold and hot shortness in wrought steels. This is due to Phosphorous segregation along grain boundaries. In melting and casting route the last liquid to solidify is rich in Phosphorous. However, in conventional powder metallurgy phosphorous iron is to admixed high purity atomized iron powder with ferrophosphorous.Pherrophosphorous powder normally has some degree of carbon, nitrogen, and oxygen besides other contaminants. The presence of these elements is deleterious to structure sensitive magnetic properties.

In the present investigation a process was employed to produce impurity free Fe-P alloy powder by using atomized iron powder and orthophosphoric acid (H3PO4).Iron-phosphorous powder so produced was expected to have a uniform distribution of phosphorous in the preparation of Fe-P alloys. In conventional powder metallurgy, involving compaction and sintering, high phosphorous contents (ranging from 0-0.7%) in Fe based alloys exhibit attractive set of mechanical and magnetic properties. These powder processed alloys suffer from increasing volumetric shrinkage as phosphorous is increased beyond 0.6% during sintering. Consequently these are not processable for intricate shapes by compacting and sintering technique. Thus both casts as well as conventional powder metallurgy routes have their own limitations.

To overcome these difficulties, Hot Powder forging was used in the present investigation for the development of high-density soft magnetic materials of different percentages of phosphorous ranging from 0.30wt%P-0.80wt%P.In this process mild steel encapsulated hot powder (blend of atomized iron powder and master alloy powder) were forged into slabs. Prior to hot powder forging, heating the powder mixture under dry hydrogen atmosphere would ensure reduction of Iron-Phosphate into iron phosphide which on diffusion into iron matrix would disappear resulting in uniform solid solution of phosphorous in iron. Mild steel encapsulation was removed after homogenization of these slabs. The slabs were then hot rolled to get highly densified sheet and wires.Thes highly densified products were annealed to relieve the residual stresses.

It was observed that Phosphorous addition improves the final density of the resulting product. Densification as high as 99.31% has been realized by adopting hot powder forging manufacturing technique. It was further observed that hot forged Iron–Phosphorous alloys have excellent hot/cold workability and could be easily shaped to thin strips (0.5-1.0 mm thick) and wires (0.5-1.0mm dia.).

The powder hot forged alloys were characterized in terms of microstructure, porosity content/densification, hardness and soft magnetic properties such as coercivity, retentivity, saturation magnetization, electrical resistivity and hysteresis losses in order to assess their performance for AC and DC soft magnetic applications. These forged and rolled powder alloys posses high saturation magnetization and low coercivity.The resistivities of the Fe-P alloys were found to increase linearly with phosphorous content. Magnetic properties were performed by two types of the samples like wire and toroid or ring. The results obtained have been discussed from the point of view of microstructures evolved.

The proposed thesis consists of five chapters. **Chapter one** consists of introduction to the subject. **Chapter two** is a critical literature review, which begins with fundamentals used for alloy development and further, selection criteria and application of soft magnetic materials. **Chapter three** consists of formulation of the problem where limitations of existing processing technology i.e. compacted and sintering, which is used for Fe-P alloy production are discussed and possible solutions as attempted in the present investigation are highlighted. **Chapter four** involves detailing of experimental techniques and the procedure followed in the present investigation. **Chapter five** consists of results and discussion in which critical appraisal of forming technology as evolved in the present investigation with regards to forming and characterization of binary Fe-P system. **Chapter six** deals with conclusion and suggestions for future work.

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CHAPTER 1

INTRODUCTION

Soft magnetic materials are known to be easily magnetized or demagnetize. The hysteresis loops of these materials are narrow, which play a fundamental role in designing electrical and electronic machines, equipment and devices. Primarily it is the material and secondly the design, which control the performance and efficiency of a device. Electrical steel, which covers about 60% of the total soft magnetic materials, is used to multiply the magnetic flux in the cores of electromagnetic coils. Among their applications are cores of transformers, electromotors, generators, or electromagnets. In order to make these devices most energy efficient and economical, one needs to find magnetic materials, which have the highest possible, saturation magnetization and permeability. Furthermore, magnetic core materials should be capable of being easily magnetized or demagnetized. In other words, the hysteresis loss or the area within the hysteresis loop (or the coercive force, Hc) should be as small as possible and the amount of core losses should also be low [1].

Powder Metallurgical (P/M) route of manufacturing has its inherent advantages such as the utilization of the material to a great extent, ability to get the product in net shape without subsequent machining or incorporating any other additional forming operation. Further, it makes possible of alloying of an element in a system to a higher amount if so desired, which is otherwise not possible through melting route. Thus P/M processing offers the material and product designer a wide flexible range and a greater scope for material's development. Manufacture of advanced soft magnetic materials involving new compositions (such as P, Si etc.) and complicated parts are only feasible through powder metallurgy processing. Density is by far the most important parameter in this context. As the density of powder metallurgical parts increase, magnetic and mechanical properties improve and at a near full density the properties are comparable with their wrought counterpart [2].

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Near full density pure iron powder metallurgy part can be easily manufactured using conventional powder metallurgical process. However, pure iron powder metallurgy parts have very low resistivity and therefore, suffer from high magnetic losses due to high amount of eddy current generation. To increase its resistivity without sacrificing the flux density Phosphorous is alloyed with it. Phosphorous is normally admixed with atomized iron powder, powder metallurgy technology can take advantage of phosphorous additions to iron. When compacted, the compressibility of the soft iron is realized without loss of green density because the hard, small 10 μm Fe2P or Fe3P particles rearrange themselves among the large iron particle as compaction occurs. When sintered, the phosphorous iron intermetallic compounds melt at a temperature of 1050°C and diffuse into the iron, forming a solid solution of phosphorous in iron. The liquid phase enhances diffusion rates and assists in the rearrangement of the pores and particle boundaries, thereby further densifying the iron. Because magnetic induction is a linear function of the sintered density, the magnetic induction realized from the phosphorous iron alloys is higher than that realized from the pure iron parts [3].

Phosphorous is also known to increase electrical resistivity, permeability, and decrease coercivity and thus improves the soft magnetic performance of pure iron. It also significantly improves ductility and toughness of Fe-P based alloys. However, the drawback to the use of the phosphorous iron parts is that, owing to the liquid phase sintering that occurs, dimensional control is more difficult. If we follow the traditional powder metallurgical process, such as compaction and sintering, for manufacturing of Fe-P alloys, heavy volume shrinkage will be experienced. Consequently phosphorous higher than 0.6 wt% is not normally recommended in conventional powder metallurgy process involving compacting and sintering. Although higher phosphorous up to about 2% continues to improve magnetic properties of these alloys. There are several other densification processes available in the literature. Out of all, hot isostatic

processing is the best so far as density and performance of these powder metallurgy parts are concerned. However, the process is extremely costly.

In the present investigation, the above problems have been encountered by the **hot powder forging technique** with impurity free Fe-P alloy powder by using atomized iron powder and orthophosphoric acid (H_3PO_4). Iron-phosphorous powder so produced was expected to have a uniform distribution of phosphorous in the preparation of Fe-P alloys. Volume shrinkage associated with these alloys is no mare a consideration in hot powder forging. This process renders highest possible densification of these alloys. In this process blended loose powders (Fe-P alloys powder) were encapsulated in a mild steel capsule. A continuous flow of dry H_2 gas through the capsule was maintained in order to create reducing atmosphere for obtaining cleaner P/M part. The encapsulated powders were heated at 1050^0C for 30 minutes in hydrogen atmosphere. Hot encapsulated powders were then forged directly by using 100T capacity friction screw press to produce a slab. These slabs were then homogenized at a temperature of 1100^0C for 1 hour. The outer mild steel skin was removed thereafter. These slabs were then hot rolled to produce wires and sheets.

These products were annealed and characterized in terms of density, microstructure, hardness, resistivity, saturation magnetization, coercivity, retentivity and hysteresis losses. In this investigation densification as high as 99.31% has been achieved. Microstructure of these alloys consisted of single phase ferrite only. Metallographic examination was employed to study the effect of grain coarsening, pore morphology and microstructure on the magnetic characteristics of the material. The hot rolled and annealed samples were mounted with the help of hot mounting and polished for metallographic and SEM examinations. The SEM examination was employed to reveal the porosity/inclusions in the alloy system. All these alloys are capable of hot/cold working to very thin gage of sheet and wire. Alloys developed in the present investigation show very low coercivity values which are less than one and very high saturation magnetization and resistivity.

For characterization of soft magnetic properties, the Hysteresis Loop Tracer HLT-SOFT and Hysteresis Loop Tracer HLT-111 were used. In the first hysteresis loop tracer HLT-SOFT, samples in the form of toroid of dimensions 40mm ID, 50mm OD and less than one mm thickness were utilized. In the second equipment HLT-111, samples in the form of the wire were utilized its dimension like 39mm length X 1.17mm diameter.

CHAPTER 2

LITERATURE SURVEY

Magnetic materials play a fundamental role in designing electrical and electronic machines, equipments and devices and primarily it is the material and secondly the design, which controls the performance and efficiency of a device. Therefore, magnetic material development, which is closely associated with the technology being followed for its manufacturing shall lead to an improvement in the performance of electrical/electronic equipments/devices. Soft and hard magnetic components are produced from large number of ferrous-based material processed through wrought and as well as through powder metallurgy route [1].

Magnetic materials encompass a wide variety of materials, which are used in a diverse range of application. Magnetic materials are classified in terms of their magnetic properties and their uses. If a material is easily magnetized and demagnetized then it is referred to as a soft magnetic material, whereas if it is difficult to demagnetize then it is referred to as a hard (or permanent) magnetic material.

In soft magnetic materials, the hysteresis losses must be kept down to a minimum. When the induction is large for a small applied field, the loop area is small and hysteresis loss is reduced. The key factor in the design of a soft magnet is then to have easily moving domain walls. Soft magnetic materials should be free of impurities and inclusions. The other source of energy loss in soft magnets is the eddy current loss [3].

Consequently, a soft magnetic material must have a high initial permeability and a low coercivity.A material possessing these properties may reach its saturation magnetization with a relatively low applied field(i.e., is easily magnetized and demagnetized) and still has low hysteresis energy losses[4].

The types of applications for soft magnetic materials fall into two main categories: AC and DC.

In DC applications the material is magnetized in order to perform an operation and then demagnetized at the conclusion of the operation, e.g. an electromagnet on a crane at a scrap yard will be switched on to attract the scrap steel and then switched off to drop the steel.

Retains a large fraction of the saturation field when driving field removed

Saturation magnetization

Narrow hysteresis loop implies a small amount of dissipated energy in repeatedly reversing the magnetization.

Magnetization M

M

M

B_0

B_0

B_0

Applied Magnetic Field

Desirable for permanent magnets and magnetic recording and memory devices.

The area of the hysteresis loop is related to the amount of energy dissipation upon reversal of the field.

Desirable for transformer and motor cores to minimize the energy dissipation with the alternating fields associated with AC electrical applications.

Figure.2.1: Variations in Hysteresis Curves [5]

In AC applications the material will be continuously cycled from being magnetized in one direction to the other, throughout the period of operation, e.g. a power supply transformer.

The various methods/processes used for making the soft magnetic materials include Powder Metallurgy and Ingot Metallurgy.

Powder metallurgy is a forming and fabrication technique consisting of three major processing stages. First, the primary material is physically powdered, divided into many small individual particles. Next, the powder is injected into a

mould or passed through a die to produce a weakly cohesive structure very near the dimensions of the object ultimately to be manufactured. Finally, the end part is formed by applying pressure, high temperature, long setting time, or any combination thereof.

Powder metallurgy has got three basic advantages over the conventional manufacturing processes [6].

- It produces Net/Near net component directly without going for complex machining sequences and thereby reduces cost, material loss and increases productivity.
- It has tremendous potentiality to form complicated assemblies consisting of various components without going for complex joining processes.

Properties of the powder metallurgy part can be precisely controlled by modifying process parameters, alloy design, impurity control and density variation.

Ingot castings can be prepared directly from solidifying molten metal. Sometimes the ingot is remelted under an electro conductive slag, the remelted ingots being converted into strips after hot rolling then cold rolling, the strips then being subjected to a treatment for developing the magnetic characteristics.

The magnetic properties of soft magnetic alloys, such as for example the permeability, the coercive field and the losses, depend greatly on the degree of inclusion pureness of the alloys. The goal of purity has always been pursued by all available technical means. Mention may be made for this purpose, for example of the choice of starting materials, production of the alloy under vacuum, the treatment of the liquid alloys by slags etc.

The inclusion density plays a very important role in the subsequent development of the magnetic properties of the products made from the strip.

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On the one hand, the inclusions slow the movement of the Bloch walls during the magnetization processes and thus lead to an increase in the coercive field and a decrease in the permeabilities On the other hand, the inclusions impair the growth of the grain, and thus lead to a poorer magnetic quality on the finished products.

All materials can be classified in terms of their magnetic behavior falling in to one of five categories depending on their bulk magnetic susceptibility. The two most common types of magnetism are diamagnetism and paramagnetism which account for the magnetic properties of most of the periodic table of elements at room temperature. Types of magnetic materials are listed in Table 2.1.

Paramagnetic Materials

These have positive susceptibilities lying between 10^{-3} and 10^{-6}. These materials are slightly attracted by a magnetic field and the material does not retain the magnetic properties when the external field is removed. Paramagnetic properties are due to the presence of some unpaired electrons, and from the realignment of the electron orbits caused by the external magnetic field. Paramagnetic materials include most gases, many salts in the iron group, the alkali metals and also ferromagnetic and ferrimagnetic materials at temperature above the Curie temperature, magnesium, molybdenum, lithium, and tantalum [7].

Diamagnetic materials

These have a very weak and negative susceptibility of the order of 10^{-6}, which is independent of temperature. Diamagnetic materials are slightly repelled by a magnetic field and the material does not retain the magnetic properties when the external field is removed. Diamagnetic materials are solids with all paired electron resulting in no permanent net magnetic moment per atom. Diamagnetic properties arise from the realignment of the electron orbits under the influence of an external magnetic field. Most elements in the periodic table,

including the inert gases, hydrogen, many metals, most non-metals, and many organic compounds copper, silver, and gold, are diamagnetic.

Ferrimagnetic Materials

These include most ferrites with the general formula $MO.Fe_2O_3$, where M stands for some bivalent ion (iron, nickel, manganese, copper, magnesium), Recently a further group of ferromagnetic materials has become known, namely compounds of iron oxide with oxides of the rare earths having the general formula 5 $Fe_2O_3.3M_2O_3$.here M stands for yttrium or one of the trivalent elements such as gadolinium, terbium, holmium, erbium etc [7].

Ferromagnetic Materials

Ferromagnetic materials have a large and positive susceptibility to an external magnetic field. They exhibit a strong attraction to magnetic fields and are able to retain their magnetic properties after the external field has been removed. Ferromagnetic materials have some unpaired electrons so their atoms have a net magnetic moment. They get their strong magnetic properties due to the presence of magnetic domains. In these domains, large numbers of atom's moments (10^{12} to 10^{15}) are aligned parallel so that the magnetic force within the domain is strong. When a ferromagnetic material is in the unmagnitized state, the domains are nearly randomly organized and the net magnetic field for the part as a whole is zero. When a magnetizing force is applied, the domains become aligned to produce a strong magnetic field within the part. Iron, nickel, and cobalt are examples of ferromagnetic materials. Components with these materials are commonly inspected using the magnetic particle method.
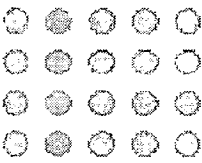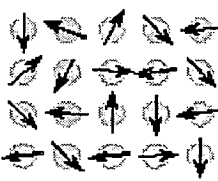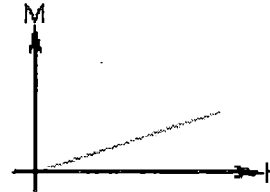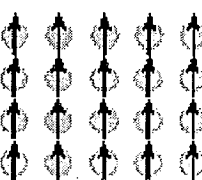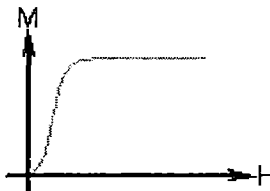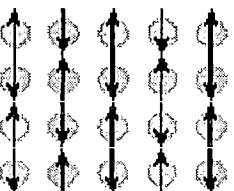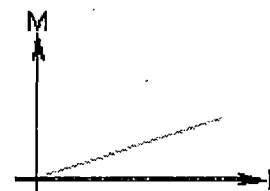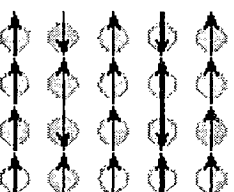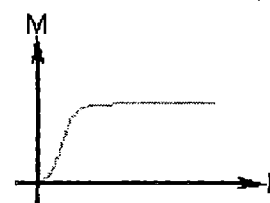
Type of Magnetism	Susceptibility	Atomic / Magnetic Behavior		Example / Susceptibility
Diamagnetism	Small & negative.	Atoms have no magnetic moment		-2.74×10^{-6} -0.77×10^{-6}
Paramagnetism	Small & positive.	Atoms have randomly oriented magnetic moments		0.19×10^{-6} Sn 21.04×10^{-6} 66.10×10^{-6}
Ferromagnetism	Large & positive, function of applied field, microstructure dependent.	Atoms have parallel aligned magnetic moments		~100,000
Antiferromagnetism	Small & positive.	Atoms have mixed parallel and anti-parallel aligned magnetic moments		3.6×10^{-6}
Ferrimagnetisms	Large & positive, function of applied field, microstructure dependent	Atoms have anti-parallel aligned magnetic moments		te ~3

Table 2.1: Types of magnetic materials [8]

Antiferromagnetic materials

These are a particular subgroup of paramagnetic substances. Their susceptibilities are of the order of 10^{-3}, but they increase with the heating, up to a critical temperature, and then fall off again [6]. Antiferromagnetic materials are very similar to ferromagnetic materials but the exchange interaction between neighboring atoms leads to the anti-parallel alignment of the atomic magnetic moments. Therefore, the magnetic field cancels out and the material appears to behave in the same way as a paramagnetic material. Like ferromagnetic materials these materials become paramagnetic above a transition temperature, known as the Néel temperature, T_N. (Cr: T_N =37°C). Examples: manganese monoxide, iron oxide, nickel oxide, iron chloride.

Soft and hard magnetic components are produced from large number of ferrous-based materials processed through wrought and as well as through powder metallurgy route. However, Iron-Phosphorous alloy system for the production of a magnetic materials is not known in wrought processing route in spite of the fact that phosphorous as an alloying elements has all favorable characteristics to enhance magnetic properties of iron [9, 10]. This is because of the fact that in wrought route it is not possible to bring phosphorous in homogeneous solid solution with iron due to its segregation tendency during solidification of the melt. This renders the alloy completely useless not only from magnetic point of view but also from mechanical properties as well.

Powder metallurgical processing has been very successful in exploiting potential of Fe-P alloy system for magnetic as well as mechanical applications [9].

The different soft magnetic materials have been listed in Table 2.2:

Category		Bs (T)	ρ(μΩ-m)	μmax	Typical core losses(W/Kg) (f/Bmax,Hz/T)	Applications, Notes
Steels	Laminated(low C)	2.1-2.2	0.4		2.0(60/1.0)	Inexpensive fractional hp motors
	Non-oriented(2% Si)	2.0-2.1	0.35		2.7(60/1.0)	High efficiency motors
	Convent grain oriented	2.0	0.48	5000	0.9(60/1.5)	50/60Hz distribution transformers
	High grain oriented	2.0	0.45		1.2(60/1.7)	50/60HzDT'S High design Bmax
Fe(Ni,Co)	40-50Ni	1.6	0.48	150,000	110(50K/0.2)	
	77-80Ni square perm alloy	1.1	0.55	150,000	40(50K/0.2)	High μ,used as thin ribbon
	79Ni-4Mo(4-79 Mo permalloy,supermalloy)	0.8	0.58	106	33(50K/0.2)	Highest μ/lowest core losses of any metallic materials
	49Co-2V(Permendur,supermendur)	2.3	0.35	50,000	2.2(60/2.0)	Highest Bs of commercial soft magnetic materials
Ferrites	MnZn	0.5	2*106	6000	35(50K/0.2)	Power supply inductors,transformers
	NiZn	0.35	1010	4000		mHz applications

Table 2.2: Typical soft magnetic Materials [11].

Powder metallurgy (P/M) parts have been used in magnetic applications for many years. Magnetic applications utilizing P/M offer both economical benefits and design flexibility. A wide range of magnetic performance requirements can be met via P/M through the proper choice of materials and the appropriate processing of those materials. Available P/M materials for magnetic applications include pure iron, iron phosphorus alloys, iron-silicon alloys, prealloys of nickel and iron and ferritic stainless steels [9].

Magnetic performance of components produced via powder metallurgy is highly dependent upon the density achieved. Saturation induction is directly proportional to the density of a ferromagnetic material. Density and material structure influence the permeability of a material [12].

In order to achieve optimum mechanical as well as soft magnetic properties of powder metallurgy part at reduced cost, we have to focus on to aspects.

1. Acquire knowledge about how the processing parameters, alloy composition, density, porosity and impurities affect the mechanical and soft magnetic properties of the P/M part.

2. Based on this knowledge find a suitable and economic powder metallurgical technique, where we can control the above factors in best possible way [2].

All magnetic properties depend upon composition of the material i.e., the contents of alloying elements and impurities in it, and also on temperature.Further,these properties may be classified in to two groups viz. (1) Structure sensitive, and (2) Structure insensitive. Induction, permeability, hysteresis loop and associated energy loss, coercive force and remanence are structures are structure sensitive properties. Saturation magnetization and Curie temperature are the two structure insensitive properties [13].

Structure sensitive properties are also sensitive to the metallurgical processing of the material such as (A) the size, shape and orientation of grains; (B) the concentration and distribution of crystal imperfections; and (C) impurities, residual stresses and the heat treatment of the material. Improved magnetic properties are obtained by increasing the grain size of the product, decreasing the number of pores by creating larger pores from several small ones, spheroidising the pore structure and enhancing the homogeneity of chemical composition [14].

2.1 OVERVIEW OF MAGNETIC PARAMETERS

A magnetic material can be characterized by its hysteresis loop, an example of which is illustrated in following Figure 2.2[9].
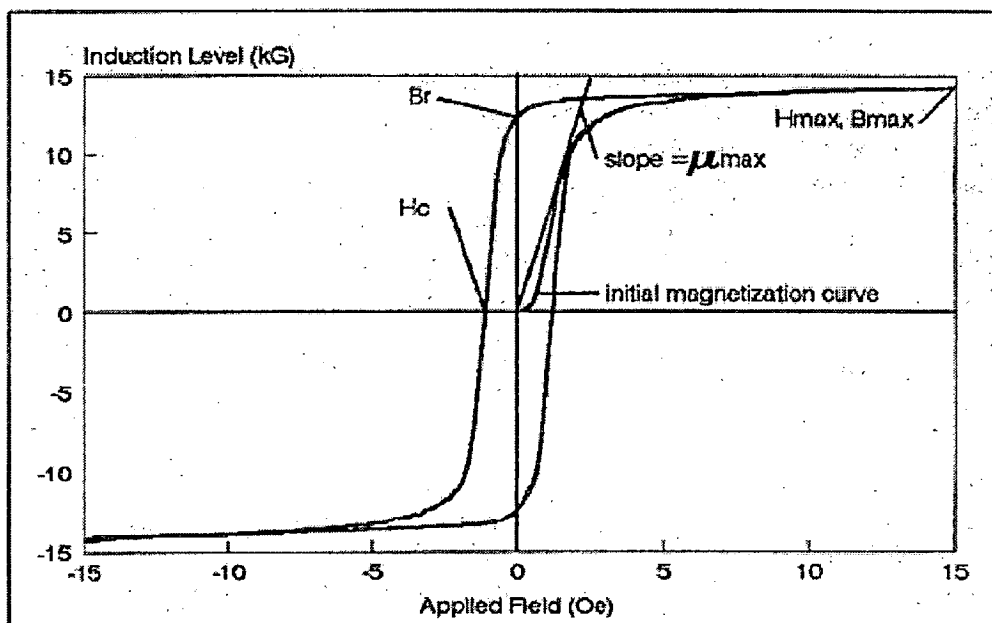
Figure.2.2: Typical Hysteresis Loop

A hysteresis loop relates the response of the magnetic induction (B) or flux density of a magnetic material to an applied magnetic field (H). Applied magnetic field is expressed in oersteds (Oe) and magnetic induction is measured in gauss (G).

Saturation induction is that induction level where further increases in applied field result in no further increase in induction.

The coercive force (Hc) of a material is the reverse magnetic field needed to bring the magnetic induction of a material to zero after the material has been exposed to a forward magnetic field.

The residual induction (B_r) is the induction that remains in a material after the applied field has been removed (H =0). When the material has been exposed to a magnetic field sufficient to magnetize the material to saturation, the coercive force and the residual induction are referred to as the coercivity and the retentivity, respectively.

The permeability (μ) is a measure of how easily a material may be magnetized and is defined as the ratio of the flux density to the applied field (B/H). The highest ratio of B/H along the initial magnetization curve is referred to as the maximum permeability (μ_{max}). Permeability is expressed relative to the permeability of free space (μ_o =1) and is unitless although occasionally permeability is reported in units of G/Oe.

A soft magnetic material is characterized by a low coercivity while a hard magnetic material or permanent magnet possesses a very high coercivity.Materials with Hc values less than 5 Oe are considered soft, and those Hc values greater than 100 Oe are leveled hard [14].

In general, properties that are characteristic of a good soft magnetic material are high permeability, high saturation induction, and low coercivity. Permeability and coercivity are strongly affected by changes in the processing of the material as well as different alloying schemes. Saturation induction is less sensitive to processing variables and is primarily influenced by the amount and type of alloy and the density of the finished part.

High electrical resistivity is important in materials for soft magnetic applications that are exposed to alternating electric fields. A varying magnetic field induces a voltage difference within the material which in turn yields a current. This current, commonly referred to as the eddy current, flows in the surface of the material in a direction that sets up a magnetic field in the direction opposing the original field, lowering the effective permeability of the material. Additionally, this current tends to generate heat within the material, reducing the operating efficiency of the device. The current can be reduced by the use of a higher resistance material. Alloying of the material has the primary influence on resistivity.

2.2 FACTORS AFFECTING THE MAGNETIC PROPERTIES [15]

Porosity and all kinds of impurities in the form of inclusions generate the same magnetic interactions as small-size air gaps inside ferromagnetic materials. Residual stresses may also degrade the magnetization characteristic of the sintered part.

2.2.1. Effect of porosity

Most magnetic parts that are manufactured contain some degree of porosity. The magnetic induction and the remanant magnetization decrease linearly as the amount of porosity increases.

Maximum flux densities of Fe–P-based alloys are found to be linearly dependent on the porosity content of the alloys.

The major impurities in magnetic materials that degrade magnetic properties include carbon, nitrogen, oxygen and sulfur.

2.2.2. Aging Effects

Both carbon and nitrogen have an effect on magnetic properties after aging. Sometimes a part can behave like a high-purity part after sintering

because, at elevated temperatures such as those selected to sinter iron or phosphorus iron parts, large concentrations of carbon or nitrogen can enter in to solid solution in the austenite and form carbides or nitrides as the material cools.

2.2.3. Effect of Residual Stresses

Secondary operations include any machining operation, sizing and repressing. If any of these operations are required, mechanical stresses are induced into the alloy. Mechanical stresses degrade structure-sensitive magnetic properties.

2.2.4. Effect of Surface Treatment

Most ferrous magnetic parts are normally coated for corrosion resistance prior to the service. Normally electroless nickel is used because nickel does not degrade magnetic properties. Other elements can be used for plating as well, such as Zink. Because the plate is thin, magnetic properties are not degraded significantly.

2.2.5. Effect of metallurgical factors

Soft magnetic properties are assessed in terms of coercivity, permeability ,retentivity, saturation magnetization, resistivity etc.These properties can be tailored either by alloying addition or by controlling processing parameters.Whereas,structure insensitive magnetic properties like saturation magnetization or flux density and remanance depend only on chemical composition and density, structure sensitive magnetic properties like coercivity and permeability depend on microstructure thus on cold working, heat treatment, grain size, pore size and distribution as well as impurities content like C,N2,O2 and S.

2.3 SELECTION AND APPLICATION OF P/M SOFT MAGNETIC MATERIALS

The magnetic properties of P/M soft magnetic components not only depend on the alloy system and its composition as in the case of wrought

materials. However, most of them also depend on processing parameters and technique. The selection of a material for a particular application will depend upon its ability to meet the required performance, and also its availability and cost. Thus, following key questions may be asked when deciding about the materials for a magnetic device [9].

1. What are the critical magnetic property requirements for the device?

Depending on application, high permeability, low coercive force, high resistivity, high saturation induction or other specific magnetic requirements may be the key consideration.

2. What are the cost constraints for the final component?

Raw material costs, fabrication costs, secondary operation costs and finished part assembly costs are included in this economic consideration.

3. What environmental situations will the device be required to withstand?

This may include increased corrosion resistance or structural requirements.

2.3.1. Pure Iron

Water atomized pure iron powders are the most frequently specified for sintered DC magnetic materials. These materials can be single pressed and sintered to densities up to 7.3 g/cm$_3$ with minimal dimensional changes following sintering. In general, the magnetic properties for pure iron can be improved as the overall level of impurities is reduced.

Figures 2.3 and 2.4 illustrate that increases in the purity of the base iron result in improved magnetic properties at a given density level. These improvements in magnetic performance allow flexibility in specifying materials and processing parameters.

Available Powder metallurgy materials for magnetic applications include pure iron, iron-phosphorous alloys, iron-silicon alloys, iron-nickel alloys and stainless steels. Table 2.3 summarizes these materials according to relative raw material cost, typical part densities, and the resulting magnetic performance.

TABLE 2.3:Typical Properties of P/M Magnetic Materials [16]

Alloy System	Typical Density (g/cm3)	Approx. Relative Cost	μ_{max}	H_c (Oe)	B_{max} @15 Oe (kG)	Resist ivity (μ_-cm)
Fe	6.8/7.2	1	1800/3500	1.5/2.5	10/13	10
Fe-P	6.7/7.4	1.2	2500/6000	1.2/2.0	10/14	30
Fe-Si	6.8	1.4	2000/5000	0.8/1.2	8/11	60
400SS	5.9/6.5	3.5	500/1000	2.0/4.0	6/8	50
50Ni/ 50Fe	7.2/7.6	10	5000/15000	0.2/0.5	9/14	45
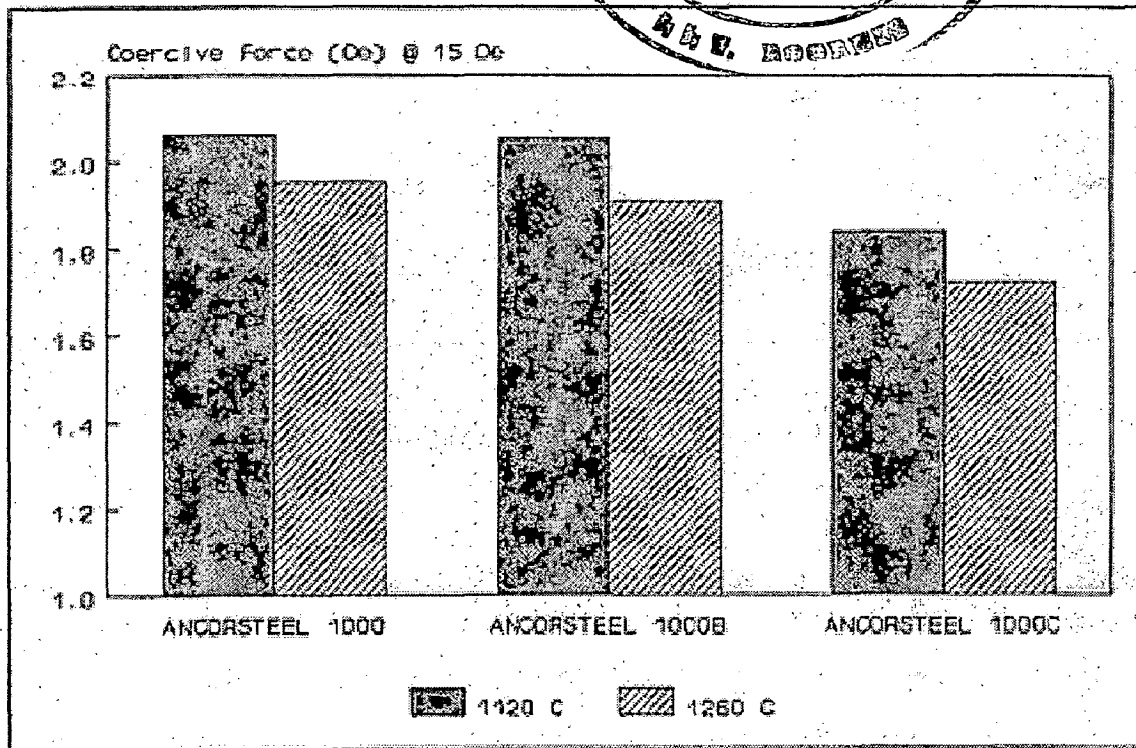
Figure.2.3: coercive Force of Several Pure Irons [9]
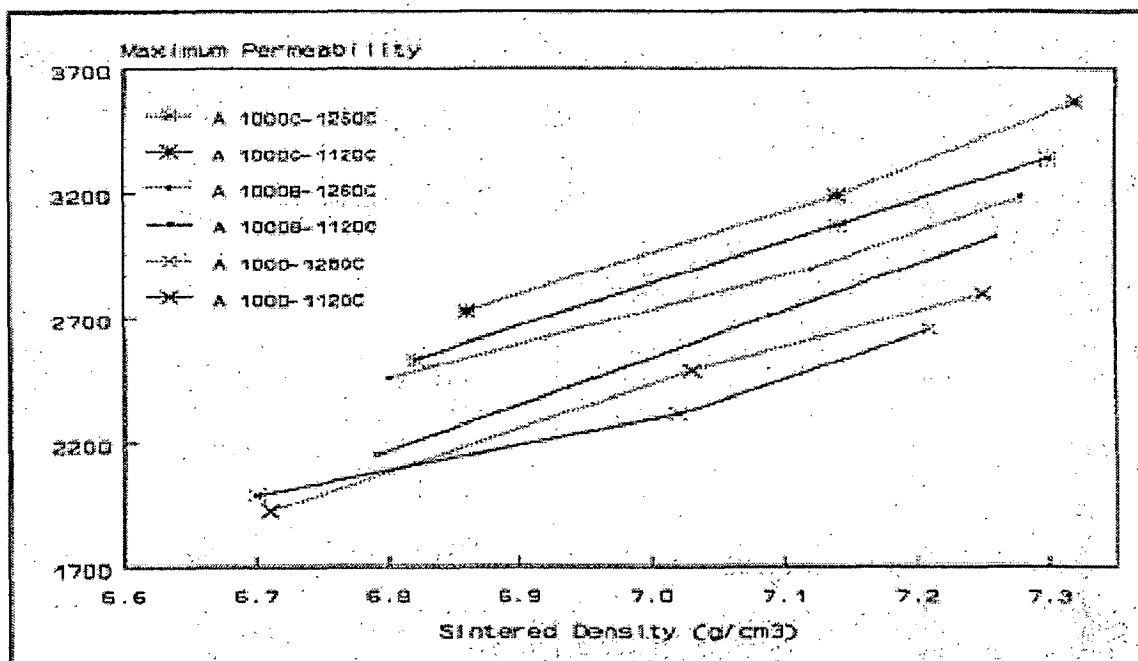
Figure.2.4: Maximum Permeability of Pure Irons [9]

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2.3.2. Iron-phosphorous alloys

The main advantages of phosphorous as an alloying element in powder metallurgy are [17]:

- Its ability to form with metals eutectics of relatively low melting points

- Characterized by good fluidity and adhesion to metals and many refractory compounds.

- High diffusional mobility of its atoms in metals.

- Its ability to precipitation-harden metals and comparatively low cost.

Iron-phosphorus alloys are the second most specified P/M soft magnetic material after pure iron. The 0.45% P/Fe alloy represents the dominant material in this family.

During sintering, the iron phosphorus intermetallic melts and diffuses into the iron, increasing the density of the sintered component while enhancing the resistivity and improving the magnetic properties of the P/M component. Figure 2.5 and 2.6 show the improved magnetic performance of 0.45% P/Fe and 0.80% P/Fe alloys compacted at 30, 40 and 50 tsi and sintered at 1120°C and 1260°C for 30 minutes in dissociated ammonia.

One of the most potent modifiers of structural P/M part performance is density. Magnetic performance of P/M parts is also strongly influenced by density. For a given alloy system, saturation induction is directly related to density. Saturation induction is important in permanent magnet flux return paths where sufficient field is available to saturate most materials. Decreasing density simply decreases the saturation flux density as indicated in Figure 2.7. The higher the density of the part, the higher the saturation flux density and thus the smaller a part can be made in order to contain the flux produced by a permanent magnet.

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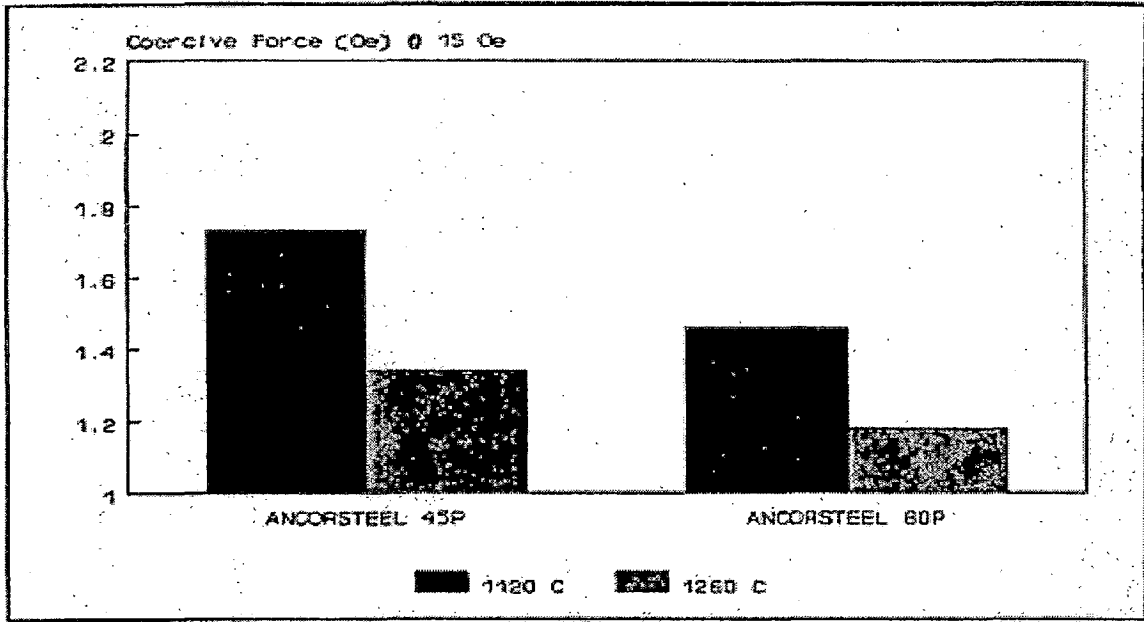
Figure.2.5: Coercive Force of Iron-Phosphorus Alloys [9]
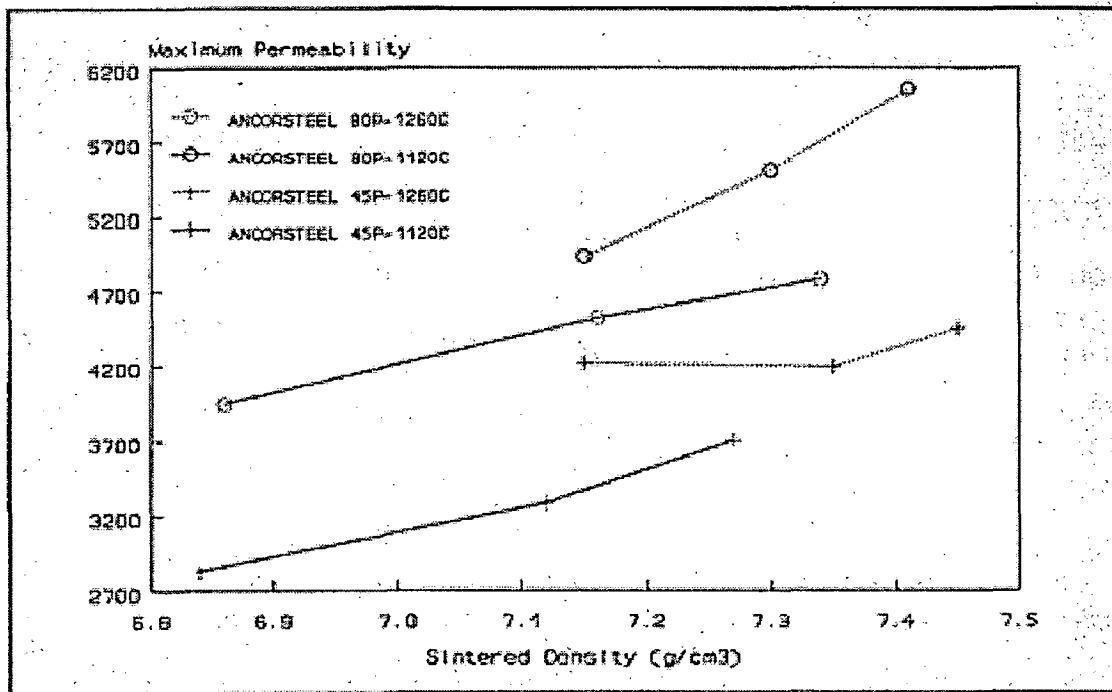
Figure.2.6: Maximum Permeability of Iron-Phosphorus Alloys [9]

Iron-Phosphorous alloys are P/M soft magnetic materials. Phosphorous, added in ferrous powder metallurgy alloys at level up to 0.8%, are an effective solid-solution hardener in iron, and are used primarily in structural P/M parts [18].
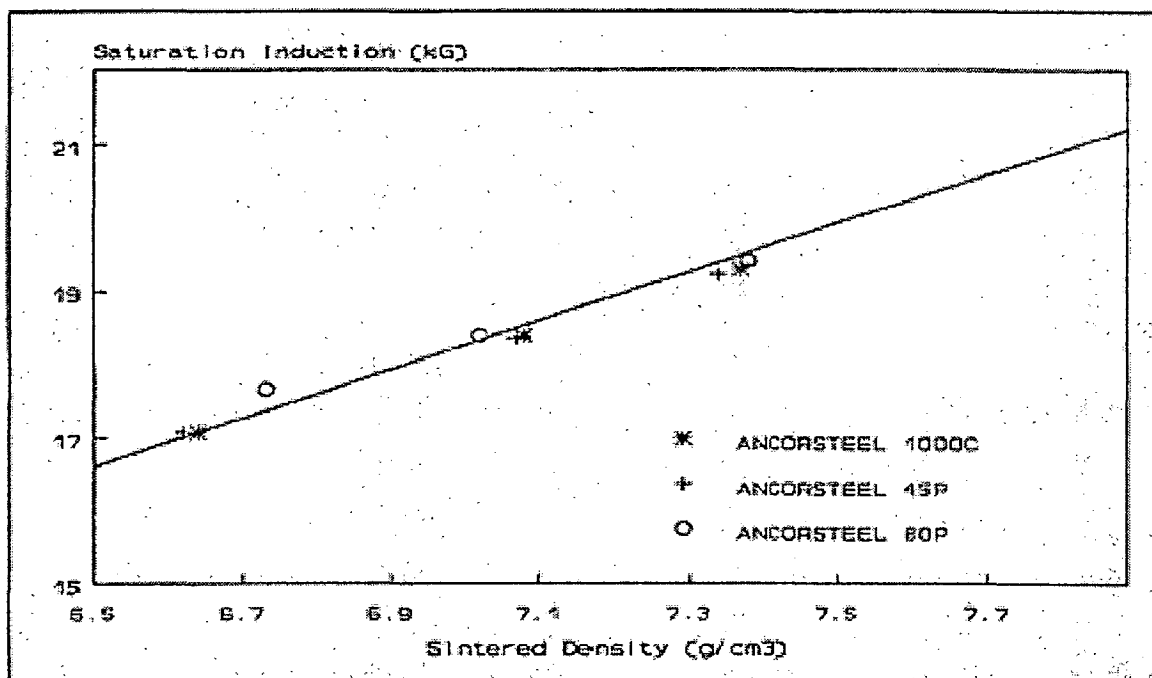
Figure.2.7: Saturation Induction versus Density for Several Alloys [9]

In general, two factors determine the kinetics of grain-boundary adsorption of phosphorus: the equilibrium concentration of phosphorus on a grain boundary with prescribed volume concentration and diffusion mobility. Alloying additives contained in steel strongly affect both factors. The concentration of phosphorus and other elements, which determine the kinetics and thermodynamics of the grain-boundary segregation of phosphorus, in steel used for fabricating reactor vessels, as a rule, is low: the phosphorus and nickel concentrations do not exceed 0.05% and 3%, respectively [19].

Phosphorus is usually considered a contaminating element in conventional steels, because of its tendency for segregation at grain boundaries causing brittleness and its low diffusion in iron. Nevertheless, phosphorus acts as a strong enhancer of the sintering process, enabling the time or temperature to

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be reduced. Among the substitutive alloying elements in iron, phosphorus has the strongest solid solution hardening effect on ferrite [20].

Iron-phosphorous alloys are produced through conventional melting and solidification route are notorious for embitterment problem. This embitterment is caused by segregation of phosphides as well as by coarsening of ferrite grain size since phosphorous is a ferrite stabilizer.Jiten Das et al [21] have also reported that different metallurgists followed different routes for making Fe-P P/M alloy to mitigate embitterment problem.

Assuming that the powder compact consists of mixture of particles of iron and Fe$_3$P with an overall phosphorous content of 0.6% solid state diffusion of phosphorous into ferrite will start as the temperature is increased. When the compact reaches 1050^{0}C a liquid phase is produced by the eutectic reaction [10]:

$$Fe_3P \quad + \quad \alpha \quad \Longrightarrow \quad L$$
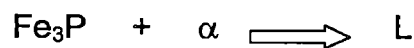
The formation of liquid phase brings about a quick distribution of phosphorous throughout the iron skeleton by capillary action[20].If we consider that the sintering operation is maintained at 1120^{0}C melt will now rapidly be depleted of phosphorous and leaving only phosphorous-rich ferrite and austenite low phosphorous content.

When the first stage is over, the melt has been absorbed, and the temperature has been brought up to the final sintering temperature (1120^{0}C), the material consists of austenite (in area which less than 0.3% P) and ferrite (in areas with more than 0.65%).At intermediate phosphorous contents both phases will co-exist. As volume diffusion is about two orders of magnitude faster in ferrite than in austenite, the sintering process is accelerated [22].
The high rate of sintering leads to rounded pores and is accompanied by a substantial shrinkage of the compacts.P.Lindskog et al [9] have reported that homogenization also proceeds very rapidly as the diffusivity of phosphorous in

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ferrite is very high, 10^{-8} $_{cm}{}^2$/s at 1120°c, as compared e.g. to the diffusivity of nickel in austenite at the same temperature, $5x\ 10^{-12} cm^2/s$.
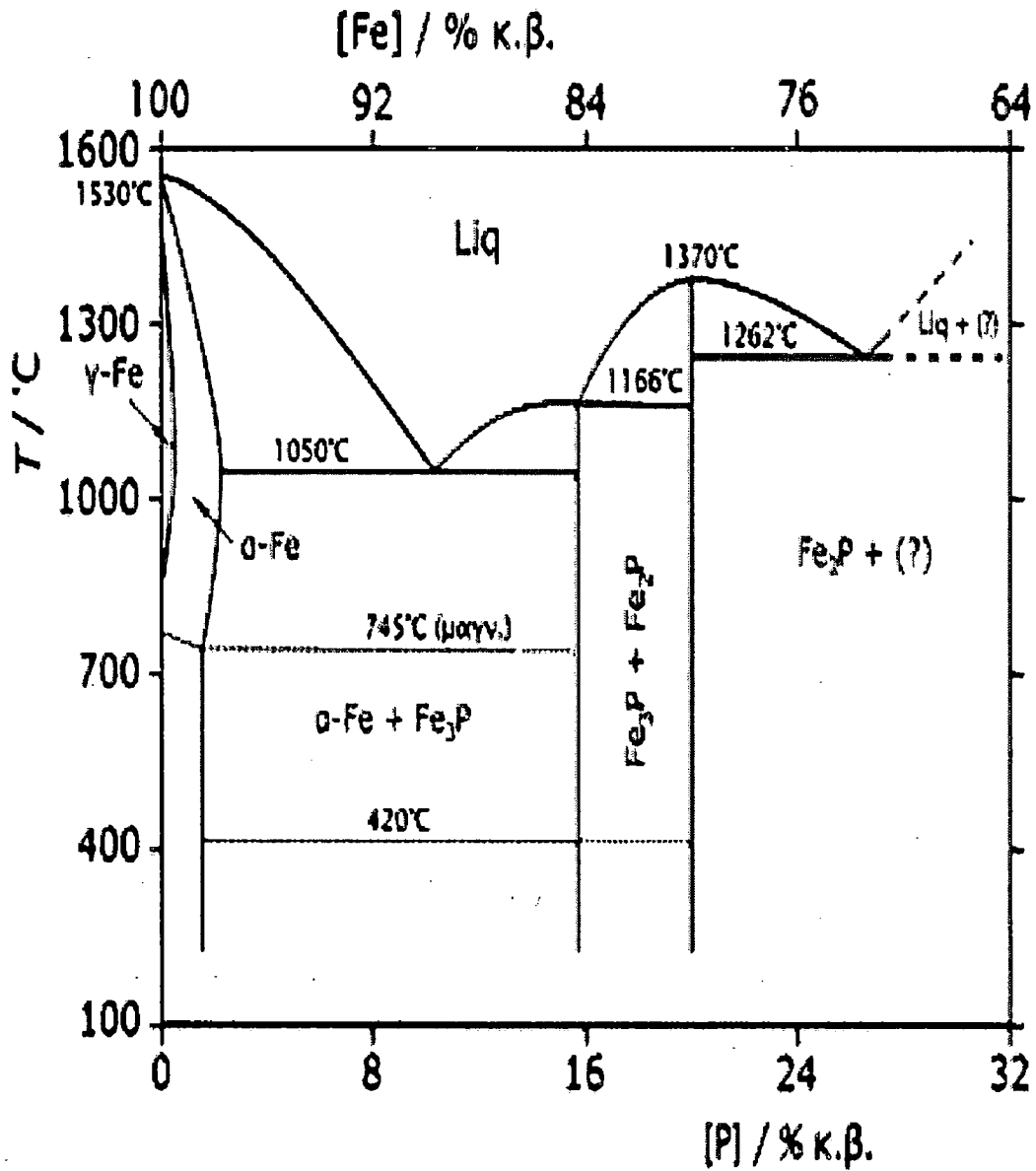
Figure.2.8: Fe-P phase Diagram [9]

Pure Iron Powders

Premix Operation ← Lubricants, Alloys–P,Si

Optional ANCORDENSE Process

Compaction → Optional DPDS Process

Pre-Sintering

Warm Compaction

Re-Press

Sintering

Secondary Operations Machine, Size, etc.

Finished Component
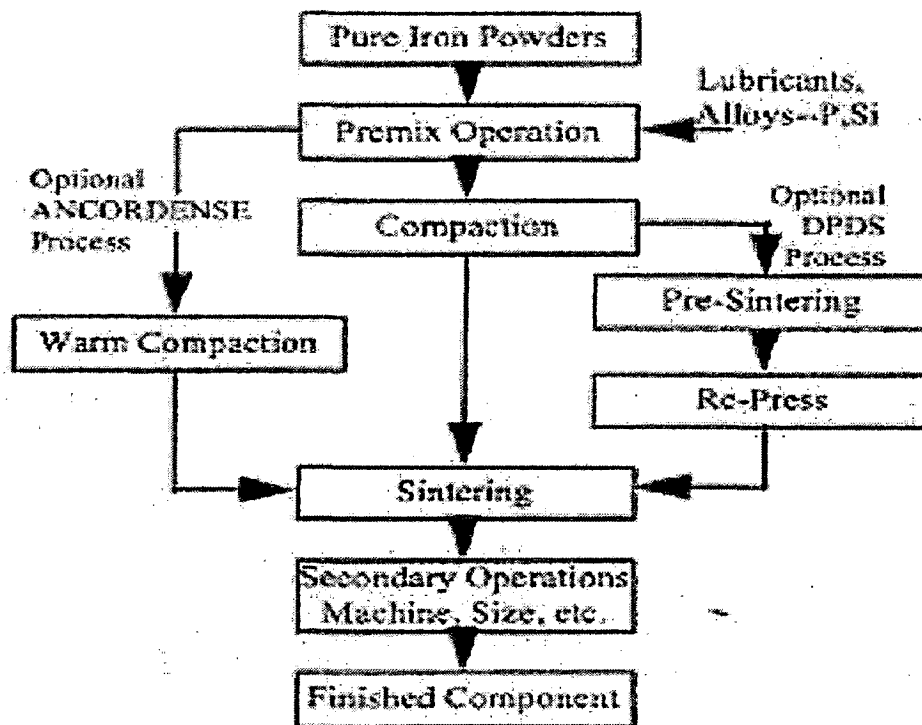
Figure.2.9: Various P/M Manufacturing Routes [23]

Phosphorous is found have a pronounced effect in weakening the grain boundaries of iron. When iron phosphate is added to iron in the hydrogen furnace, complete reduction occurred with substantially no loss of phosphorous [24].

Drawback:

- It has been observed that the incidence of brittle fracture increased with the phosphorous content. Whereas brittle fracture is never observed at 0.3% P, it started to occur at 0.45% P and incidence increased at 0.6% P.

- The use of Fe-P parts is that, owing to the liquid phase sintering that occurs, dimensional control is more difficult.[3]

Small additions of carbon to the system bring about a dramatic reduction of the brittleness problem. A maximum is obtained at about 0.02-0.03% carbon [22]. It has also been shown that it becomes possible to work with even higher phosphorous content up to 1.2%, without encountering brittleness, provided some carbon is added. The optimum carbon addition varies with the phosphorous content. It is generally between 0.1 and 0.4%.

Hartmut Hanse et al [25] have reported that the displacement of phosphorus by carbon as the following reaction:

C (dissolved) + P (segregated) = C (segregated) + P (dissolved)
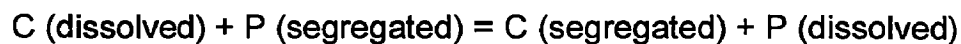
The amount of dissolved carbon rather than the total carbon content determines the carbon segregation. After exceeding the solubility of carbon in equilibrium with Fe_3C or carbide a further increase of carbon content has no effect on the phosphorus segregation. In a Fe-Cr-C-P alloy the carbon solubility is decreased and accordingly the grain boundary segregation of P is increased in comparison to a Fe-C-P alloy. Phosphorus is one of the principal embrittling elements in low alloy steels. The interactions of segregated phosphorus with other metalloid impurities are therefore of particular interest. Studies on Fe-P-C alloys revealed evidence of a beneficial, deembrittling effect of carbon on phosphorus segregation.

2.3.2.1. Effect of phosphorous on Magnetic and Electrical Properties

The powder metallurgy process lends itself well for economical mass production of complicated soft magnetic components. Magnetic induction is a linear function of the density [26]. In order to obtain the best possible magnetic properties it is therefore necessary to use an iron powder which can be compacted to very high density. A low content of impurities in the iron is important to obtain good magnetic properties. The higher magnetic inductions

and permeabilities can be achieved by adding phosphorous to iron powder [9]. He has also shown that the resistivity is increased with phosphorous additions as a result lowers the magnetical losses.

Tanaka.I et al has reported that core loss of steel decreases with reducing thickness and magnetic induction also decreases with increasing cold-rolling reduction. However, magnetic induction rose with phosphorous addition. Furthermore, magnetic induction of 0.1P steel only slightly decreases with increasing cold- rolling reduction [27].

If 0.45 wt% P is alloyed, resistivity is approximately doubled. If 0.8 wt % P is alloyed, the resistivity is approximately 24 mΩ.cm.Higher resistivity is required to minimize eddy current losses at low level.P.Lindskog et al [10] have reported that coercive force decreases with phosphorous contents up to 0.6 %.For contents above 0.6% no further reduction has been observed. The losses in AC-application mainly consist of two parts: hysteresis loss and eddy current loss.Hysteresis losses are proportional to the area of the hysteresis loop.
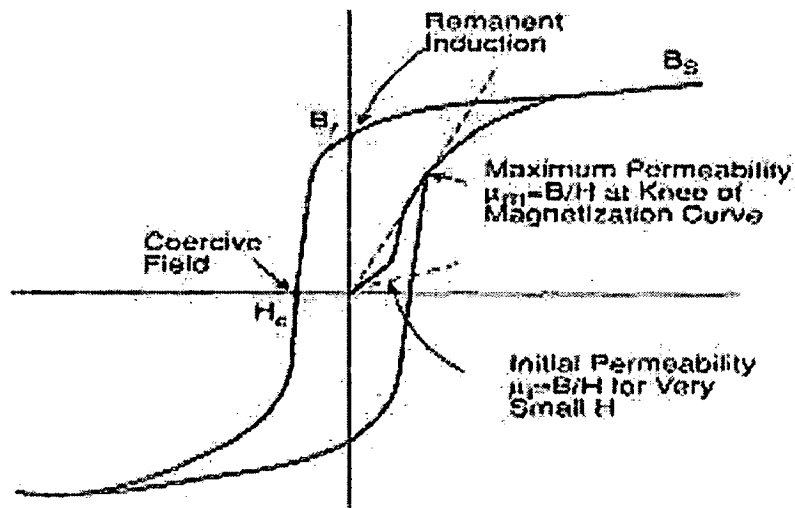
Figure 2.10: Hysteresis loop

Table 2.4 shows the variations of saturation induction with density for material processed at room temperature and material forged at an elevated temperature [28].

Coercivity of the Fe-based alloys fall as we add alloying elements such as P and Si to Fe. The higher the Si, P content, the lower is the coercivity values observed. In spite of higher percentage of porosity level, Fe–0.7P–0.7Cr alloy showed lower coercivity.Furthermore, P alloying also reduces the coercivity of Fe significantly.

Table 2.4: Variation of saturation induction vs. Density [28]
(Fe+0.45%P, Sintered 1260°C, H2 gas, 30 MIN)

Pressing Temp.(°C)	Molding Pressure(Mpa)	Sintered Density(g/Cm^3)	B_{sat}(gauss)
20	413	7.09	18,340
	551	7.31	18,960
	689	7.45	19,340
150	413	7.24	18,740
	551	7.45	19,370
	689	7.59	19,730

The magnetic characteristics of ferromagnetic materials in alternating fields depend on the specimen shape and size as well as on the physical properties of the materials themselves. The level of magnetic characteristics in sintered parts is determined by the depth to which the alternating magnetizing field penetrates them.

The following Figure 2.11 depicts the variation of the electrical resistivity of the material, sintered under the optimum conditions and having a porosity of 5%, with P content. Increases in electrical resistivity are observed at P

contents of up to 1.5%, but above this concentration the electrical resistivity slightly falls.
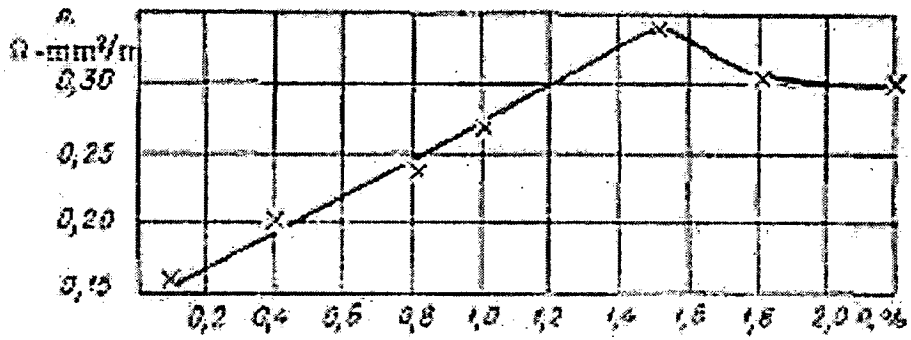
Figure 2.11: Variation of electrical resistivity with phosphorus content [29].
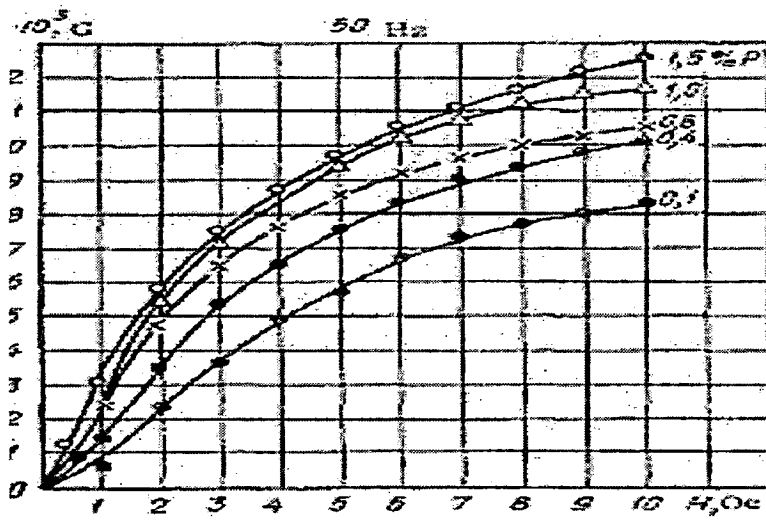
Figure 2.12: Magnetization curves plotted for materials of various phosphorus contents [29]

The following Table 2.5 shows the Coercivity, maximum flux density and Retentivity Values of the different Fe-P alloys system which has been reported [26]:

Table 2.5: Magnetic Properties of Fe-P alloys

Material	Coercivity (Oe)	Max.flux density (G)	Retentivity (G)
Fe–0.35P—0.35Cr	1.50	874.0	510.0
Fe–0.7P–0.7Cr	1.40	787.0	520.5
Fe–0.35P–0.35Cr–1Si	1.10	905.8	338.0
Fe–0.7P–0.7Cr–1Si	1.00	977.5	565.0
Fe–0.35P	2.20	998.0	608.0
Fe–0.7P	1.50	980.0	597.0
Fe–1Si	1.67		
Fe–2Si	1.27		
Fe–3Si	1.10		
Fe–6.5Si	0.30		
Fe–3Si–0.45P	0.98		

The powder metallurgy approach of magnetic cores for AC electrical apparatus and miniature motors offers many technical and economical advantages [9].The following Figure 2.12 shows magnetization curve plotted at a frequency of 50 Hz for Fe-P specimens of various P contents, sintered under the optimum conditions.

2.3.2.2. Effect of alloying elements on mechanical Properties of Fe-P alloys

The hardness is found to increase with P, Cr as well as Si alloying additions. Combined effect of chromium and phosphorous causes more hardening of ferrite than the silicon in solid solution with iron. However, porosity also affected the hardness of these products(Fig.2.13) .Jiten Das et al [23] have

reported that In similar porosity level of two alloys, the improvement in hardness could have been realized due to P and Cr alloying addition, namely Fe–0.35P0.35Cr–1Si alloy and Fe–0.7P–0.7Cr–1Si alloy with similar porosities (4 vol%).
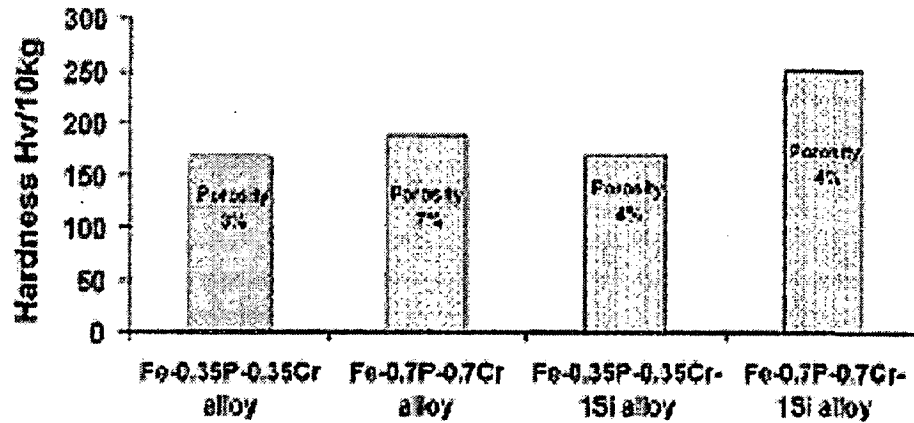
Figure 2.13: Variation of hardness with P, Cr and Si alloying additions in the alloys [21]

Fe–P based alloys containing 0.35 wt%P showed very higher ductility as compared to that containing 0.7 wt%P. However, the Fe–P based alloys containing 0.7 wt%P shows fairly high strength as compared to that containing 0.35 wt%P.

Engineering stress versus engineering strain curves for Fe–P based alloys has been developed for different alloying elements which are shown in Figure 2.14.

Lund.j.A has reported that Sintered ferrous compacts containing up to 0.8% phosphorous have attractive mechanical properties, including an excellent combination of tensile strength and ductility when sintered at relatively low temperatures [30].

Figure 5.13: Stress-strain curves for Fe-P based alloys
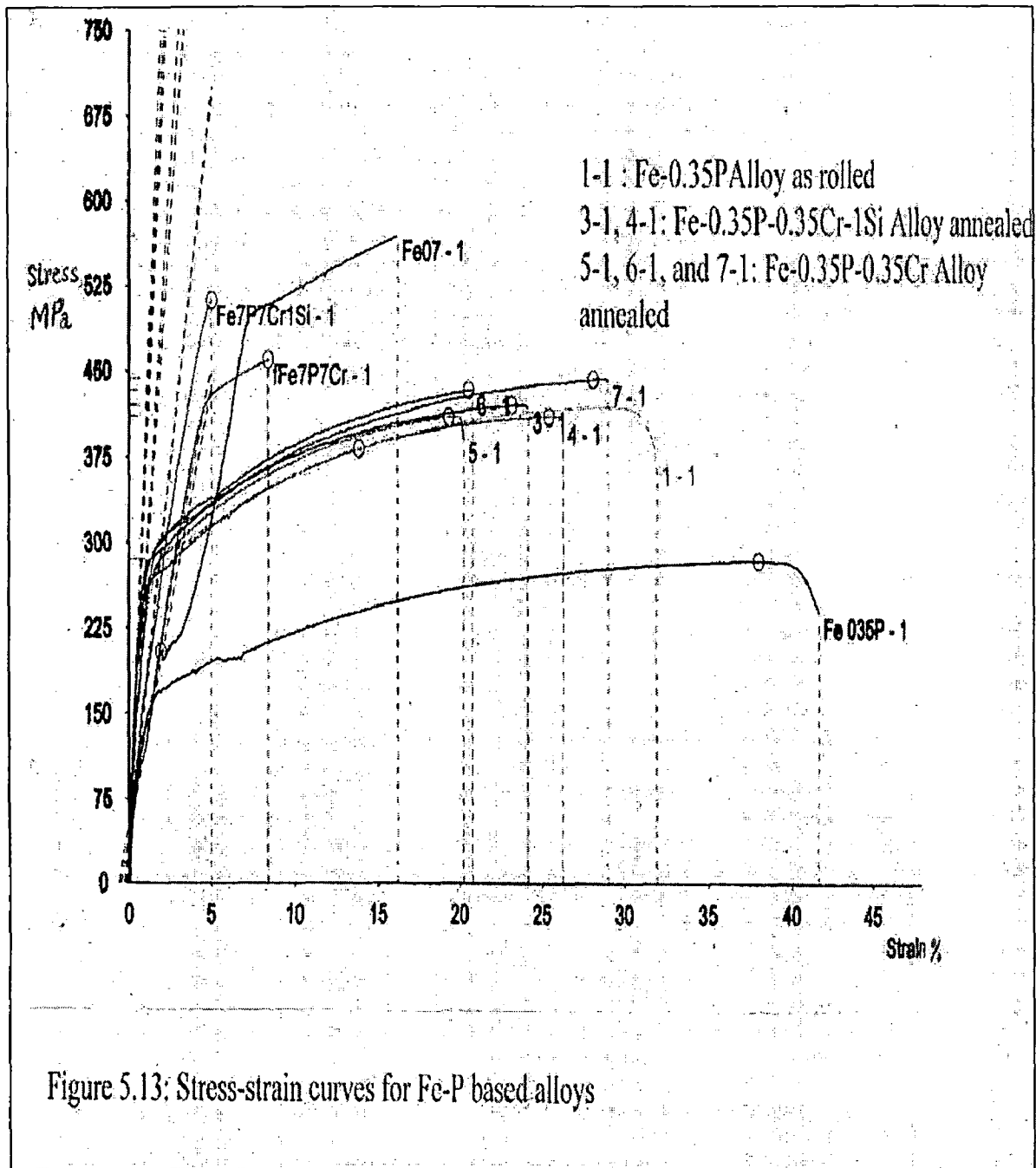
Figure 2.14: Stress-Strain Curve for Fe-P base alloys [2]

Tensile properties, such as yield strength, tensile strength, elongation%, strain hardening exponent and work hardening rate at yield point of Fe-P alloys is reported in Table 2.6. It shows that all the specimens have low

strain hardening exponent and able to form into very thin gage of sheets (0.5 mm) and wires (1 mm).

Table.2.6: Tensile properties of Fe-P alloys [21]

Material	Yield strength (MPa)	UTS (Mpa)	Uniform elongation (%)	Total elongation (%)	Strain hardening exponent (n)	Work hardening rate $(d\sigma/d\varepsilon)$ at $\varepsilon=0..2$
Fe-0.35P annealed	170.0	274.4	38.6	39.6	0.16	14.9
	292.0	410.0	28.7	29.6	0.13	13.6
Fe-0.35P hot rolled	296.3	429.2	21.4	21.5	0.15	16.4
Fe-0.35P-0.35Cr annealed	277.7	416.4	23.3	23.7	0.14	12.9
Fe-0.35P-0.35Cr- 1Si annealed	497.3	557.7	9.0	9.0	0.17	15.8
Fe-o.7P	424.3	460.0	4.0	4.0	0.14	16.3
annealed Fe-0.7P-0.7Cr	460.0	511.7	1.0	1.0	0.49	51.2
annealed Fe-0.7P-0.7Cr- 1Si annealed	260.0	359.0	-	30.0	0.15	-
Commercial hot rolled steel sheet*[1]						

*Composition C<0.1wt%, Mn =0.15-0.35wt%, P<0.040wt%.

P increases the yield strength, ultimate tensile strength and hardness, but decreases both elongation and reduction in area at failure. Very high P contents promote brittle behavior (Stewart *et al* 2000). Phosphorus causes solid

solution strengthening, of the same order as interstitial carbon and nitrogen (Allen 1963).

2.3.2.3 Effect of Phosphorous on corrosion properties

The beneficial effect of phosphorus in iron with respect to iron's superior atmospheric corrosion resistance has been well established, as revealed by the Delhi iron pillar. Therefore, the use of phosphoric irons in applications that require atmospheric corrosion resistance has been propounded. This idea is already being implemented in some grades of weathering steels. The beneficial effect of P on the corrosion resistance of iron could be one of the reasons why phosphoric irons are found well preserved in excavations, because they are presumably less susceptible to corrosion. As phosphorus is a common feature in all these irons, it therefore has an important role to play in their corrosion resistance [31].

The iron produced from the direct reduction processes can be utilized for several large-scale applications. One important application is the production of corrosion-resistant iron. The specific environment in which corrosion resistance needs to be enhanced is atmospheric exposure. Huge investments are being currently made to prevent and control the atmospheric corrosion of iron objects. The Delhi iron pillar reveals that phosphoric irons would offer excellent resistance to atmospheric corrosion. [32]

The study on the rust layers are formed on weathering steels which contain a small amount of Cr, P and Cu has been a principal theme in the field of atmospheric corrosion, since the rust layers give certain beneficial effects for the prevention of degradation of the steels.

2.3.3. Applications

The general area of application can be summarized as follows:

- Materials for medium and high strength components can be manufactured at lower material cost.
- Applications where the combination of high strength and ductility is important, e.g. during assembly.
- Applications where components are subjected to shock loads.
- Two special applications, viz. Fe-P-C materials for bearings and Fe-P alloys for soft magnetic components.

Powder forged connecting rods which are gaining in popularity all over word due to their improvement dimensional accuracy, higher dynamic properties, smoother running in the engine, and significant cost savings.

The following Table.2.7 shows several areas where P/M applications are proven successful along with the material type and performance concerns that dictate the material selection. Insulated iron powders are found a significant automotive application in the area of ignition system.

Table 2.7. Application of Fe-P alloys [23]

Application Type	Typical Material	Performance criteria
Motor Frames	Fe , Fe-P	Saturation, Density, Bmax.
Pole pieces	Fe , Fe-P , Fe-Si	Saturation , Density , Permeability
Relays and Actuators	Fe-P ,Fe-Si , 50Ni/50Fe	Response time,permeability,coercive force
Other sensors	400 SS,Fe ,Fe-P	Corrosion, Permeability

Examples of different applications are listed in the following Table 2.8:

Table 2.8: Different product made by P/M processes [33]
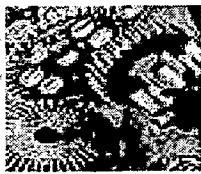
	STRUCTURAL PARTS		POWDER FORGING
	POROUS BEARINGS		HARD METALS
	NON POROUS BEARINGS		DIAMOND TOOLS
	FILTERS		ELECTRIC, MAGNETIC PRODUCTS
	FRICTION MATERIALS		MIM PRODUCTS
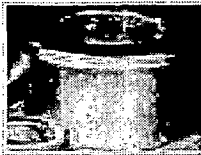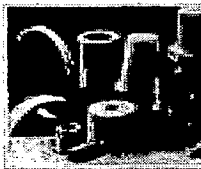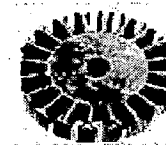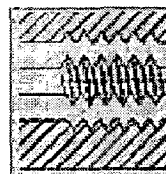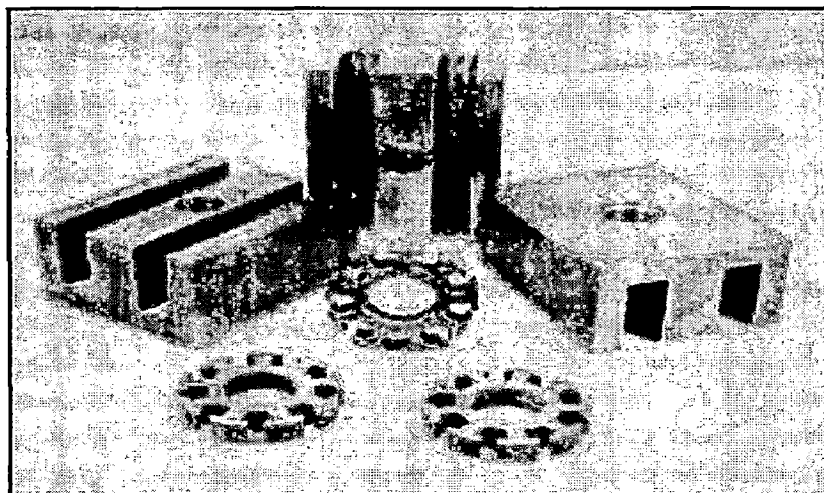
Figure2.15.Typical product of soft magnetic materials [34].

2.4. ADVANCED TECHNIQUE FOR IMPROVED PROPERTIES OF P/M FERROUS SOFT MAGNETIC MATERIALS

Powder forging is a process in which unsintered, presintered, or sintered powder metal performs is hot formed in confined dies. The shape, quantity, and distribution of porosity in Powder metallurgy and Powder forging parts strongly influence their mechanical performance [35].

Powder forging process is a new technology combining the merits of powder metallurgy and hot working. The technique, which involves the hot forging of performs made by P/M methods in fully closed die systems, has been variously called "Sinter Forging", "PM Forging", and "Powder Forging". With Powder forging, however, a fully formed component can usually be produced with one forging blow only, in one set of closed dies, with an attendant reduction in actual forging cost, and greater improvement in press utililization. The application of powder forging to manufacture engine pistons for vehicles, gears, parts for oil pumps, and parts for shock absorbers has been actively achieved [36]. Powder forging is a deformation technology processing technology aimed at increasing the density of powder metallurgy parts and thus their performance characteristics.
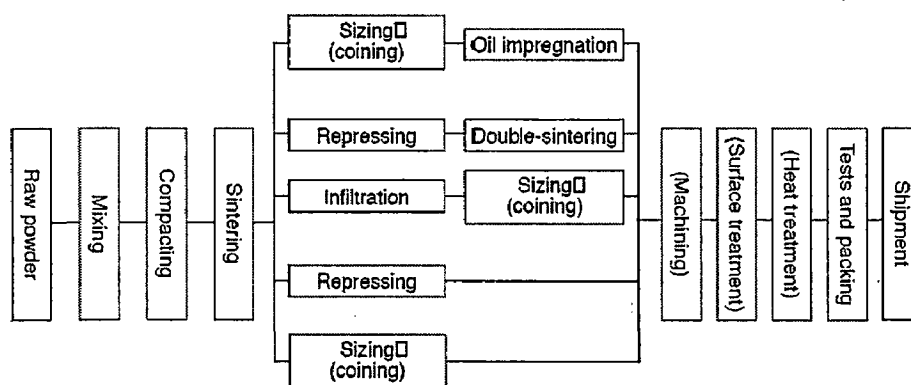
Figure 2.16: Outline of powder metallurgy manufacturing method [34]

The entire powder metallurgy process mainly consists of the following basic stages [9]:

- Production of metal powders
- Mixing or blending of the metal powders in required proportions
- Pressing and compacting the powders into desired shapes and sizes
- Sintering the compacted parts in a controlled furnace atmosphere
- Subjecting the sintered parts to the secondary processing, if required

Density of P/M parts are depends on several parameters which have been shown in [2] as below:

- Powder particle size
- Forging load
- Powder heating temperature
- Percentage reduction of preform
- Forging speed

Powder Particle size:

Powder particle size should be as low as possible because lower is the particle size, higher is the density of P/M part.
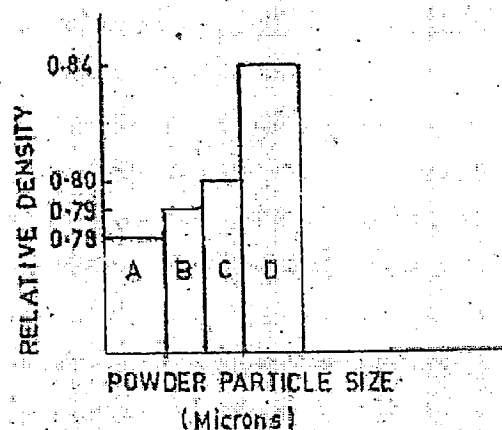
Figure 2.17: Relative density of powder forged parts as a function of powder particle size [2].

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Forging Load:

Higher is the forging load, higher is the density of P/M part.
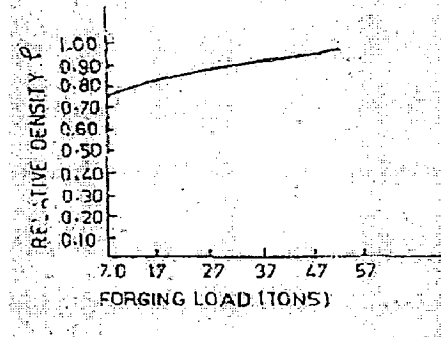
Figure 2.18: Relative density of powder forged parts as a function of forging load [2].

Powder heating temperature:

Heating of the powder causes cleaning of the powder surface. High temperature also reduces the flow stress during deformation and thereby improves workability.

Forging Deformation:

Higher forging pressure causes collapse of pores. Higher forging deformation also causes shearing of the powder and therefore, exposes clean metal.

Forging speed:

The porosity of the surface is caused by the sudden local cooling of the surface of powder material. This rapid local cooling occurs due to the contact between the powder surfaces with the die. Therefore; contact time between

powder preform surface and the die during deformation should be as low as possible.
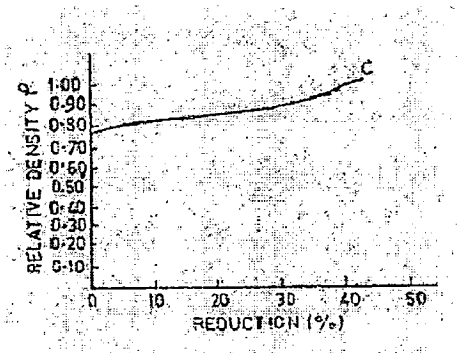
Figure 2.19: Relative density of powder forged part as a function of percentage deformation [2].

In recent years there has been an increasing interest in phosphorous-containing sintered alloys. Phosphorous is added to powder metallurgy materials to improve a number of their characteristics or to reduce the cost of manufacturing them without adversely affecting their properties [22].

Density is a predominant factor in the performance of powder metallurgy components. Powder forging have been employed to provide higher densities than traditional single press and sintered operations. Higher density level can be achieved in parts with complex shapes that are unsuitable for double pressing/double sintering. [37]. Generally, as density is increased almost all properties, including strength and magnetic performance are improved [9]. Fe-P alloys are also promising P/M soft magnetic materials.

Addition of P also has several disadvantages; large phosphorous additions increase the shrinkage during sintering of P/M parts to such a degree that the tolerances of the sintered P/M component may become adversely affected [10].

The feature that distinguished phosphorous from all other strengthening agents is that the improvement in strength is not accompanied the usual drop in

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ductility up to a phosphorous content of 0.6%.At higher P-contents the materials abruptly became brittle[38].

With the identification of applications requiring corrosion resistance as well as mechanical and magnetic properties, including the Anti-lock Brake System tone wheel industry is investigating ferritic stainless steel solutions [39].

Conventional powder metallurgy is hindered by its ineffectiveness for use in alternating current applications. Laminated steels have been used to provide materials with improved frequency performance, but the production of Powder metallurgy laminations are impractical.

The two recent advances in powder metallurgy are as below [28]:

- First process utilizes warm compaction of the powder premix to enable sintered densities of 7.59 g/cm3 or about 96% of the theoretical density for iron containing 0.45% phosphorus.
- The second is a process for applying a thin thermoplastic coating to the iron particles and compacting these coated particles into components with excellent structural properties and high frequency soft magnetic performance.

2.4.1. High density via Warm Compaction

It has been found that elevating the temperature of the iron powder premix and the tooling during the compaction Process yields significant increases in pressed density.
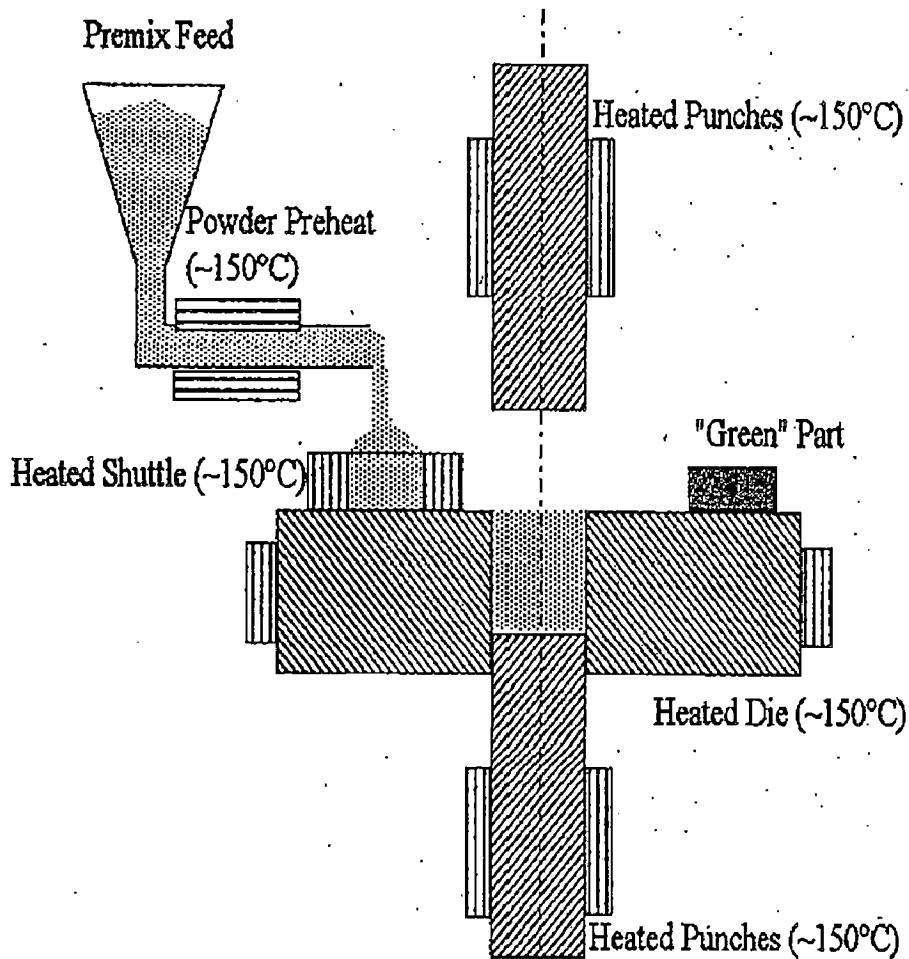
Figure 2.20: Warm compaction Process [28]

The powder is preheated before being introduced into the heated die. The pre-heating of the powder enables production rates of greater than 10 components per minute to be maintained. A specially engineered premix is utilized to provide good powder flow characteristics at the elevated temperature and to provide lubrication during the heated Compaction process. Increased density brings about an improvement in magnetic performance

The following table 2.9 shows density and saturation induction (Bsat) for material processed at room temperature and material compacted at an elevated temperature:

Table 2.9: Properties of P/M Fe-P materials [28]

Pressing Temp. (°C)	Molding Pressure (MPa)	Sintered Density (g/cm³)	Bsat (gauss)
	413	7.09	18,340
20	551	7.31	18,960
	689	7.45	19,340
	413	7.24	18,740
150	551	7.45	19,370
	689	7.59	19,730

2.4.2. Plastic Coated Iron

A newly developed method for applying a high temperature thermo-plastic coating to the iron particles achieves high density compacts with high frequency magnetic properties. A uniformly distributed plastic coating is applied to the iron particles by a fluidized bed coating process. The plastic coated iron powder is then compacted at an elevated temperature similar to that shown in Figure 2.20.

Magnetic performance can be tailored to meet the needs of particular applications by the proper selection of iron and coating. Plastic coated iron powder offers several cost advantages over the use of laminated steels. Plastic coated iron has the high material utilization typically associated with the near net shape capability of the powder metallurgy process. The use of laminated steels often times has low material utilization particularly for complex shapes and high assembly costs [28].

Plastic coated iron powder has found application in the stator assembly for brushless DC motors. Analysis has been performed on stators produced with plastic coated iron compacts versus stators made from 0.15 mm nickel iron laminations.

Generally, as density is increased almost all properties, including strength and magnetic performance, are improved. Traditionally, density has been increased by raising compaction pressures, elevating sintering temperatures, sintering enhancement additions, double press/double sinter processing, copper infiltrating and powder forging. Figure 2.21 indicates the density ranges that may be expected from these various powder metallurgy processes. Table 2.10 shows the strengths and weaknesses associated with the powder metallurgy processes. Figure 2.21 shows that the higher density can be achieved by the powder forging process.

Table 2.10: Effect of P/M processes on performance [37]

Process	Strengths	Weaknesses
Conventional compaction	Inexpensive	LimitedDensity Capability
	Wide range of part shapes possible	
DoublePress/Double Sinter	High Density	Expensive
		Limited Part Shapes Double Processing
Copper Infiltration	Improved Performance	Expensive Double processing
Powder Forging	Near to Full Density	Very Expensive Complicated processing

2.4.3. Iron Powder Polymer Composites [38]

The development of iron powder polymer composites was expected to broaden the application base for P/M into high volume AC magnetic applications. Powder metallurgy because of its inherent shape making capability and high material utilization offers the potential to be an ideal part making technique to produce high volume AC electrical devices.

Iron powder polymer composites utilize a high strength polymer to both electrically insulate the powder particles and add mechanical strength to the as compacted part. The iron powder polymer composites utilize warm compaction techniques to facilitate the plastic flow of the polymer; thus increasing the green density and giving a significant increase to the green strength of the component.
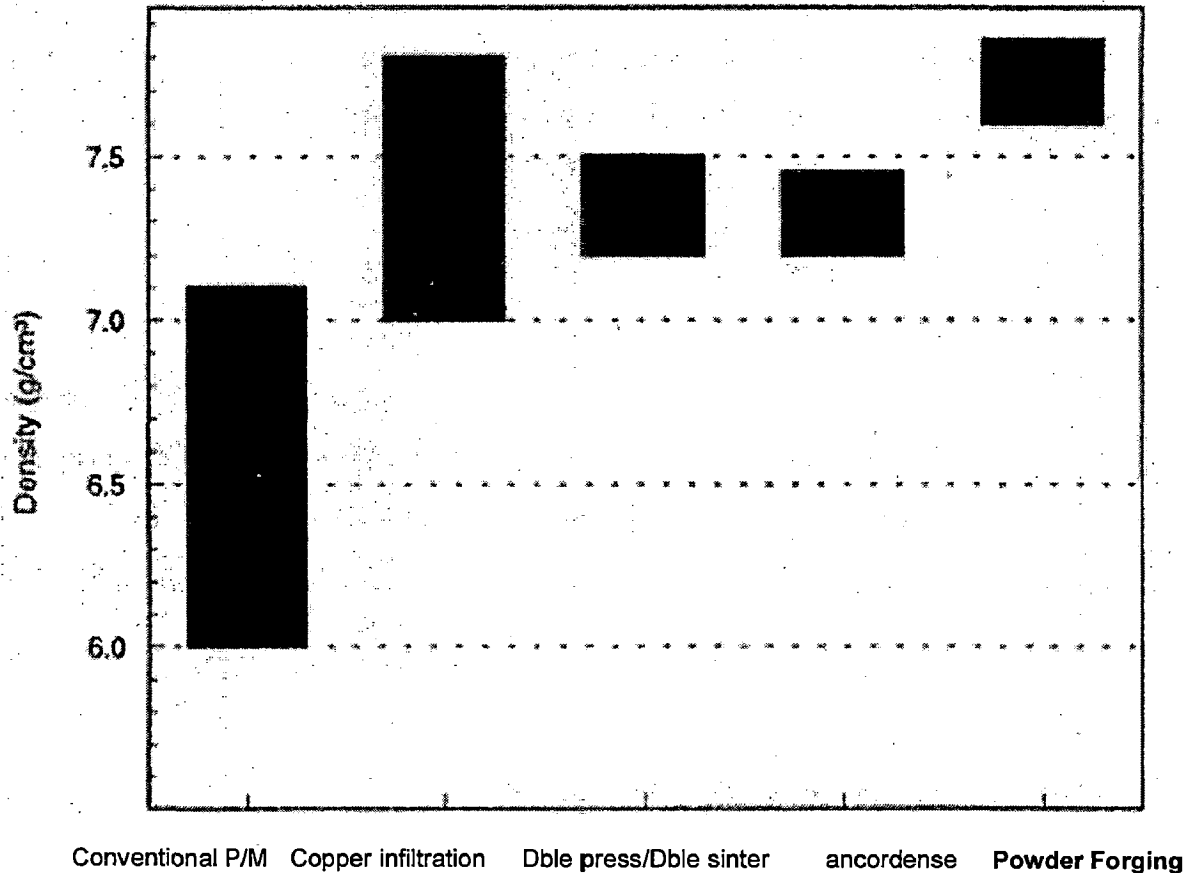
Figure 2.21: Density Ranges for several common Powder Metallurgy Processes [37]

Components made from the iron powder polymer composites are brittle and exhibit no elongation during tensile testing. The measured modulus of elasticity of these materials is approximately 5 X 10s psi.

Magnetic properties of the iron powder polymer composites are dependent upon the amount of polymer, the particle size distribution of the iron powder, and any pretreatment of the iron powder prior to polymer coating.

Ancorsteel SC 120 utilizes a coarser particle size distribution with the lowest polymer content and yields the highest compacted density. Consequently, it exhibits the best DC magnetic properties and has the highest AC magnetic performance at Low frequencies.

Ancorsteel TC 80 utilizes a smaller particle distribution combined with an initial oxide treatment of the powder followed by the polymer coating. This increased level of insulation gives the lowest DC magnetic performance but yields the best high frequency performance, exhibiting constant permeability up to 50,000 Hz.

The advantages of powder metallurgy for producing high volume, high strength automotive components include [40]:

- The ability to form complex shapes in a single forming operation.
- Net or near-net shape capability resulting in minimal finish machining.
- High volume capability.
- Energy efficiency.
- Competitive cost

2.5. POWDER FORGING PROCESSES

The powder forging is an economic method of producing components directly from metal powders. Therefore, it is currently arousing interest in many parts of the world. Advantages of the process are:

- Reduction of number of steps for producing components.
- Reduction of scrap due to elimination of flash and secondary machining operation
- Reasonably high density can be obtained and the mechanical properties P/M parts are comparable to the wrought product [3].

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Forging is the working of metal into a useful shape by hammering or pressing. The two broad categories of forging processes are open-die forging and closed-die forging. Open-die forging is carried out between flat dies or dies of very simple shape. In closed-die forging the work piece is deformed between two die halves which carry the impressions of the desired final shape [41].

Forging denotes a family of processes by which plastic deformation of the work piece is carried out by compressive forces. Forging can be carried out at room temperature, a process called cold working, or at elevated temperatures, a process called warm or hot forging [42].

There are two basic forms of powder forging [42]:

- Hot upsetting
- Hot repressing

Powder forging (P/F) is used to produce components essentially free of internal porosity. The associated properties are equivalent to those developed in conventional forged products made from billets. The Conventional P/F process is performed in four steps with the first two similar to normal PM processing. A preform is pressed as a conventional PM compact. The mass, density, and shape of the preform are controlled closely to ensure consistency in the characteristics of the final forged component. The preform is sintered with particular attention paid to the reduction of nonmetallic inclusions. The sintered preform is then reheated, placed in the forging die, and forged to full density. The tooling shape is designed to be close to that of the finished part with material flow controlled to fill the cavity completely.

The conventional powder forging process consists of four steps:

1. Mixing of the powder
2. Compacting it
3. Sintering it
4. Forging it

The determination of optimal parameters on the powder forging process is very important [43, 44].

Densification Mechanism in powder forging

Heating of powder prior to forging causes cleaning of the powder surface and improves the workability of the porous preform.During forging considerable shear deformation is involved since the preform is in different shape from the forging. Pore closure takes place in the powder forging mainly due to plastic collapse of pores and material flow into the pores.Diffusional flow, particle sliding, rotation, revolution and dislocation motion also contribute to densification.

Advantages of Powder forging Process

The following advantages can result from the use of the powder forging processes:

- Simplified Forging route
- Improved Detail and Surface Finish
- Material Saving
- Machining saving
- Weight Control
- Superior in some respects because of the freedom of the sinter forged part from directionality
- Greater homogeneity as regards composition
- Finer microstructure
- Absence of internal discontinuities [45]
- An additional advantage is the dimensional consistency achievable in consequence of the accurate metering of the quantity of powder used.

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Disadvantages and Limitations of powder forging process

- Production costs are higher than conventional
- Component size is currently limited

CHAPTER 3
FORMULATION OF PROBLEM

In early applications, sponge iron was used to produce magnets. The magnetic properties of these early magnets were not as good as those of current magnets because the sponge iron magnets contained as much as 2 wt% insolubles, generally in the form of Oxides. Besides being impure, the sponge iron powder was also harder(due to impurities associated) and therefore could not be compacted to densities above 6.2 g/cc.If improved magnetic properties are required, either carbonyl iron or electrolytic iron is suitable owing to its high purity levels.

Today, inexpensive water-atomized iron powder is readily available. This powder has less than 1wt% impurities present. Because the powder is pure, the structure-sensitive magnetic properties are greatly enhanced. In addition to water-atomized iron being pure, it is also soft because there are no hard inclusions present [2].

Why water atomized powder is chosen for soft magnetic application?

Water atomized powder has following characteristics over other powder producing methods:

- Free from microporosity.
- Very low impurity level.
- Soft because atomized powder is annealed under H_2 gas atmosphere.
- Compressibility is matching in comparison to other methods.
- Flowability is high.
- Low hydrogen loss value.
- Carbon content is controllable to any low value low.

- Cost effective.

Pure iron was used as soft magnetic material in electrical industry due its high flux density. However, use of pure iron as soft magnetic material is limited only for direct current application. In alternating current application this material suffers from magnetic losses due to high amount of eddy current generation. This eddy current can be minimized by decreasing the thickness of the material. However, the thickness reduction may cause reduction of cross section area for magnetic flux to pass. The other way of reducing eddy current loss is by increasing electrical resistivity of the material. Alloying additions like Ni, Si, as well as P to iron cause increase in the resistivity of iron. However, Ni and Si reduce the flux density to a great extent if heavy amount of alloying is done. Moreover, nickel irons are the most expensive of all the commonly used magnetic alloys [2] while Si is accompanied by embitterment when it exceeds beyond 2wt%.

Phosphorous alloying addition improves the resistivity of the iron and decreases the coercivity effectively and thus decreases hysteresis loss of the alloy. It also improves alloys formability [7].But it is necessary that entire phosphorous remains in solid solution with iron. This is not possible in wrought metallurgy owing to its sensitivity to segregation. Introduction of phosphorous in iron is therefore only possible with powder Metallurgical processing.

Phosphorous is an undesirable impurity in conventional steels. It is responsible for cold and hot shortness in wrought steels. This is due to Phosphorous segregation at grain boundaries. In melting and casting route the last liquid to solidify is rich in Phosphorous. However, In conventional powder metallurgy phosphorous iron is to admix high purity atomized iron powder with ferrophosphorous.Ferrophosphorous powder normally has some degree of carbon, nitrogen, and oxygen besides other contaminants. The presence of these elements is deleterious to structure sensitive magnetic properties.

It is therefore proposed in the present investigation to employ a process to produce impurity free Fe-P alloy powder by using atomized iron powder and orthophosphoric acid (H_3PO_4).Iron-phosphorous powder so produced is expected to have a uniform distribution of phosphorous in the preparation of Fe-P alloys.

In Conventional powder metallurgical process, involving compaction and sintering, high phosphorous contents (ranging from 0-0.7%) in Fe based alloys exhibit attractive set of mechanical and magnetic properties. However, the conventional powder metallurgical processes involve too many steps. It involves filling of powder mixture into a die cavity, compaction of powder mixture, sintering and/or post sintering operation. We need to use lubricants also in order to facilitate compaction. In spite of so many processing steps, conventional powder metallurgical process does not ensure high density P/M parts. These powder processed alloys also suffer from excessive volumetric shrinkage as phosphorous is increased beyond 0.6% during sintering. Consequently these are not processable for intricate shapes by compacting and sintering technique. Thus both casts as well as conventional powder metallurgy routes have their own limitations.

To overcome these difficulties, it is proposed to employ Hot Powder forging of this alloy in such a manner that the liquid phase is already absorbed by matrix phase before subjecting it to hot powder forging. In this process mild steel encapsulated hot powder (blend of atomized iron powder and iron-phosphate powder) were forged into slabs. Prior to hot powder forging, heating the powder mixture under dry hydrogen atmosphere would ensure reduction of Iron-Phosphate into iron phosphide which on diffusion into iron matrix would disappear resulting in uniform solid solution of phosphorous in iron. Mild steel encapsulation was removed after homogenization of these slabs. The slabs were then hot rolled to get highly densified sheet and wires. These highly densified products were annealed to relieve the residual stresses. A study reveals that the hot forging of iron powder with controlled processing parameters results in

substantial improvement in the density level of the product as compared to the material being produced by conventional compacting and pressure sintering [34].

The powder hot forged alloys would be characterized in terms of microstructure, porosity content/densification, hardness and soft magnetic properties such as coercivity, retentivity, saturation magnetization, electrical resistivity and hysteresis losses in order to assess their performance for soft magnetic application. Magnetic properties would be performed by two types of the samples like wire and toroid or ring. The results obtained would be discussed from the point of view of microstructures evolved. Magnetic characteristics would be assessed for both AC and DC applications.

CHAPTER 4

EXPERIMENTAL WORK

4.1 PREPARATION OF Fe-P ALLOY POWDERS

Magnetic materials require strict impurity control.Ferrophosphorous, which is being used to introduce phosphorous in Fe-P sintered alloys, has some inherent impurities. Therefore, it was planned to replace ferrophosphorous by a high purity iron-phosphorous alloy powder in the present investigation.

Accordingly, a process has been developed to prepare impurity free iron-phosphorous alloy (master alloy) powder by using atomized iron powder and orthophosphoric acid, H_3PO_4.The coating of iron phosphate, $Fe_3(PO_4)_2$ took place on keeping the iron powder dipped in orthophosphoric acid as per the desired % of phosphorous in master alloy and adequate amount of distilled water.

The specifications of atomized iron powder and orthophosphoric acid, which was used in the present investigation, are as following:

(A) Specification of atomized powder

Atomized iron powder grade No: AHC100.29

Manufactured by	: M/S HOGANAS AB, Sweden
Upper particle size	: 0.17mm
Apparent density	: 2.59 gm/cc
Flow	: 25-28 second per 50 gm.
Compressibility	: 6.65-6.68 gm/cc
Hydrogen loss percentage	: 0.1-0.2(i.e. reducible oxygen content)
Carbon content	: 0.01-0.02

(B) Specification of orthophosphoric acid

Purity : 83-98%

1cc contains : 1.75 gm H3PO4

For preparation of 1 kg master alloy of 10% phosphorous, 900 gm atomized iron powder was mixed with 200 ml ortho-phosphoric acid. While mixing with iron powder,ortho-phosphoric acid was diluted by slowly mixing it in to adequate amount of distilled water(approximately 100ml H_3PO_4 was diluted by 800 ml distilled water) so that the volume of the diluted orthophosphoric acid was sufficient to completely cover the iron powder, which was laid in a tray. The iron powder, dipped in diluted orthophosphoric acid was left in open air to form iron phosphate and dry. If required it was also put in the oven at low temperature. The ortho-phosphoric acid would react with the surfaces of iron particles and form $Fe_3(PO4)_2$ on the iron particle surface by the reaction

$$3Fe + 2H_3PO_4 \rightarrow Fe_3(PO4)_2 + 3H_2$$
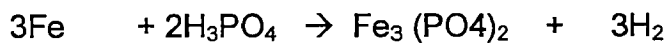
The composite (i.e. coated) powder so produced is decanted and allowed to dry as described. The situation is schematically illustrated in figure.4.1.

Figure 4.1: Schematic diagram showing the preparation of iron-phosphate

4.1.1. Blending and heating of powders prior to forging: The powder blends were made by using atomized iron powder and master alloy (iron-phosphate) for

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making different composition of Fe-P alloys. Mixing was done mechanically in a laboratory jar mill for 2-3 hours. The total weight of mixture was taken 1 Kg.The mechanically mixed powder was encapsulated in mild steel cylin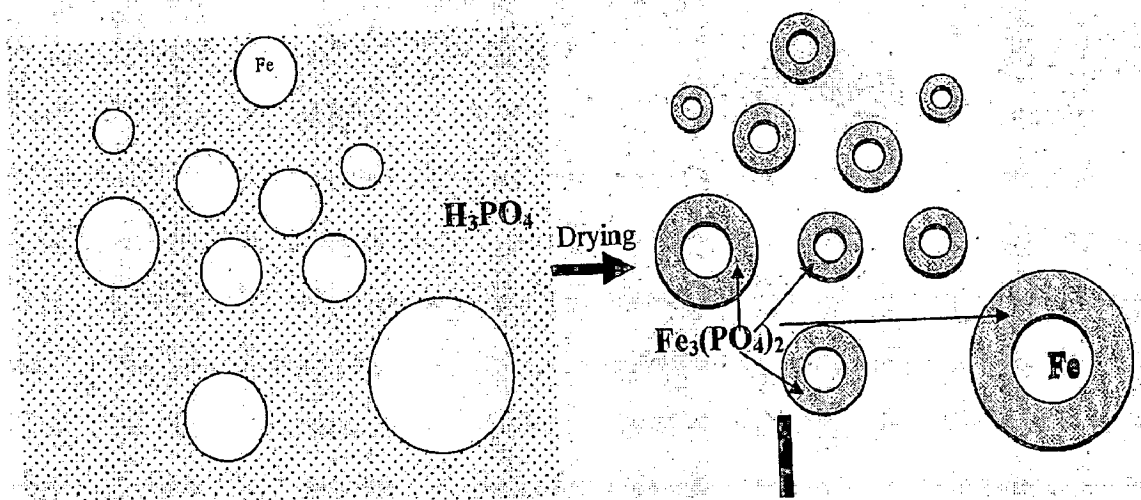drical can (capsule). Dry hydrogen gas was passed through the encapsulated powder mix just before the insertion of mild steel capsule in the furnace chamber and continued till it was taken out and the first blow of the hammer was delivered in the forge press. The burning of H_2 gas exit end of the capsule ensures the continuous maintenance of reducing gas atmosphere in the capsule during heating of powder mix. The encapsulated powders were thus heated in a tubular furnace at 1050^0 C for 30 minutes in dry hydrogen atmosphere as shown in figure 4.2. During heating the encapsulated powders were required to rotate for complete the reduction of powder. The H2 atmosphere would remove the oxide layer from the surfaces of the powders heated at the high temperatures. The product at the iron particle surface i.e. Fe_3 $(PO4)_2$ would be converted to iron-phosphide (Fe_3P), during heating at 1050^0 C for 30 minutes in H_2 atmosphere, whereas the core of the iron particles remained unaffected. The reaction would be as follows

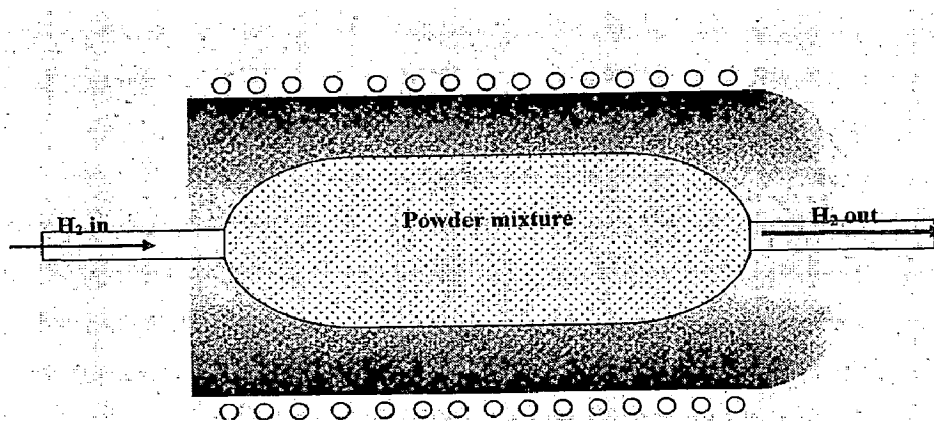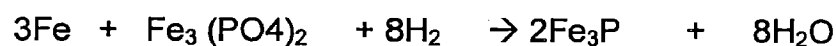$$3Fe \ + \ Fe_3\,(PO4)_2 \ + 8H_2 \ \rightarrow 2Fe_3P \ + \ 8H_2O$$

Figure 4.2: Illustration of heating of mild steel encapsulated powder mixtures inside a tubular furnace in presence of hydrogen gas.

4.1.2. Hot forging of alloy powders, Homogenization and forming into sheet and wire form: The capsule was taken out and hot forged in a 100 metric ton

friction screw driven forge press. After first blow of the hammer the supply of H_2 gas is closed. Heated capsules were then forged in the press to make slabs using a channel die. Only three strokes were required to form a slab of thickness 15mm including the mild steel skin having about 85% of theoretical density.

The forged slabs were then homogenized at 1100^0C for 1 hour in a muffle furnace followed by air cooling to ensure uniform distribution of alloying elements throughout the cross-section of forged component. The additional purpose of homogenization is to eliminate brittle non-equilibrium phases situated along grain/particle boundaries.

After homogenization, the ends of mild steel capsule were cut off. The mild steel encapsulation was also removed by machining. A forged slab with about 7mm thickness was thus obtained.

Samples were prepared from the forged slabs for density measurement, hardness test and microstructural studies; and remaining parts of the forged slabs were rolled to produce thin sheets and wires using wire drawing die.

The forged slabs, after removal of mild steel skin, were hot rolled using flat roll and section rolls at ~1000^0C to make thin sheets(0.5-1mm thickness) and wire(1-1.5mm diameter) respectively. For making thin sheets and wire of said thickness, 25 to 30 roll passes were required. The final two passes were given at room temperature during rolling of each sheet and wire. All the compositions developed in the present investigation had excellent hot/cold rolling response and no failure by way of cracking of any sort was observed. Rolled wires were passed through wire drawing dies to draw the wires of different diameter.

Some samples were also rolled with mild steel skin up to 3-4mm thickness under similar parameters. The main purpose of this to avoid the oxidation of the sample during the rolling at about 1000^0C.In this process, we have got the sheet of 60-70cm long .The mild steel skin was then removed with the help of grinder.

4.1.3. Annealing treatment of sheets, toroids and wires: The rolled sheets, stamped toroid and wires were annealed by keeping them in muffle furnace at 920^0C for 20 minutes and then cooling in the furnace. For annealing, the rolled sheets, toroid and wires were first coated with a special type of ceramic coating which has been developed in the Metallurgical and Materials Engineering Department of IIT Roorkee as proprietary item to prevent the oxidation.Toroid or ring samples were prepared by the die-punch of dimension OD 50mm and ID 40mm.The rust from the surface of the samples were removed and cleaned by gently rubbing with a coarse and finally with a fine grain emery paper. The whole alloy manufacturing process is illustrated through the flow chart Figure 4.3.

4.2 CHARACTERIZATION OF ALLOYS:

4.2.1. Density measurement: Density of the forged and homogenized as well as rolled and annealed samples was measured and it was ensured that it is in the range of 7.4 to 8.2 gm/cc.

4.2.2. Magnetic Properties measurement: The magnetic characteristics were determined using Hysteresis Loop Tracers (Model: HLT-111 and HLT-SOFT).Hysteresis loop tracer (Model: HLT-111) requires wires type samples whereas Hysteresis loop tracer (Model: HLT-SOFT) requires toroid/ring, or Epstein squire samples. The standard dimensions of wires were 1.17mm diameter X 39mm length. In the another method samples in the form of toroid or ring were utilized of dimension 1mm thickness, 50mm OD, 40mm ID. In case of the Ring samples primary and secondary windings are made using Teflon coated multi strand wire (Figure 4.5). The primary winding should be uniform and with as many turns as possible. The primary winding serves as a source of applied field/current and secondary winding as the pick up for induction. In the present investigation AC and DC measurements have been taken. Model HLT-SOFT is capable of giving up to 20 Amperes AC and 10 Amperes DC of source current. Winding must be properly insulated from the test sample. The number of turns in

the secondary would depend largely on sample properties and should be adjusted properly for selected 'secondary flux range' on panel to get good plot on monitor screen. The set up offers measurement of total loss/kg at the frequency of measurement. In the present investigation the losses due to eddy current can not be separated off from the hysteresis losses. The difference of OD and ID of the toroid/ring samples should have minimum to perform the smooth and closely pack windings (primary and secondary).

The following steps were followed for measurement under DC and AC conditions by Hysteresis Loop Tracer (Model: HLT-SOFT) of Toroid samples:

- For measurement on ring sample select toolbar option i.e. Current ranges (10A), Induction ranges (0.2M), Ring sample(R), Measurement units CGS/MKS, shows hysteresis curve etc.

- The primary and secondary winding terminals should be connected to the terminals marked as primary and secondary on the Epstein Square assembly.

- Clicking on parameters option which require the input parameter such as

  Sample Name : Gives the sample name (say Fe-P alloys)

  Mean Path Length : Gives Mean Magnetic Path Length in cms (14.14cm)

  Cross Sectional Area: Gives the cross sectional Area in sq.cms (0.10cm^2)

  Density : Gives Density of sample in gms/cc (7.76gm/cc)

  X : Gives full scale for field in oersteds (250 Oe)

  Y : Gives full scale for induction in Kilo Gauss (30KGs)

  No. of Primary Turns: Enter the no of primary turn wound on the stack (73)

  No. of Secondary turns: Enter the number of secondary turns wound on the Stack (10)

Full scale loss   : Gives full scale loss per Kg in Watts (15).

Full scale permeability   : Gives full scale permeability (50000)

- Clicking on measurement option which requires the input parameters such as:

Frequency range for DC   : 6-50

Frequency range for AC   : 150-300

The noise in the hysteresis loop can be reduced by adjusting the drift closure to zero (say 0.00012).The measurement was carried out by clicking on the scope or measurement option which is shows on the screen.

The following steps were followed for measurement by Hysteresis Loop Tracer (Model: HLT-111) of wire type samples:

- Firstly hysteresis loop tracer was calibrated without sample by adjusting the demagnetization nobe (N) at zero and area ratio nobe (As/Ac) at 0.40 at magnetic field 200 G.

- At particular magnetic field (200G), the values of e_x in mm and e_y in volt (if read by applying on Y input of CRO) were recorded. These terms have been stated in previous chapter.

- The values of ex and e_y were calculated for the area ratio 1(in proportion to area ratio 0.4), to calculate the values of Go (rms) and Go (peak to peak) correspondingly.

- The wire sample was put in the sample holder. By adjusting demagnetization (factor) nobe and area ratio nobe for particular samples as per calculated values, the B − H loop was found on the CRO.Since the

loop was too small so demagnetization and area ratio nobe were adjusted to three times the calculated values. The demagnetization factor (N) was taken from the Table A corresponding to the c/.a (ratio of length of sample to diameter of sample).

- We were recorded the loop width in mm, tip to tip height and intercept in volt of the B-H loop at different magnetic field(Gauss),

- We were ploted the curves (loop width, tip to tip height and intercept vs applied field) for calculating the coercivity, saturation magnetization and retentivity.The following expressions were used for calculating the coercivity,saturation magnetization and retentivity:

(a) Coercivity

$$H = \frac{Go\ e_x}{[As/Ac - N]} \quad in\ Oe$$

Where, e_x =1/2 X loop width (after dividing the multiplying factor 3)

As= Cross sectional area of the sample

Ac= Cross sectional area of the pickup coil

N= Demagnetization factor

(b)Saturation magnetization

$$Bs = \frac{Go\ \mu o\ g_x\ (e_y)_s}{Gy\ [As/Ac - N]} \quad in\ gauss$$

Where, $(e_y)_s$ =1/2 x tip to tip height

Mo =1

$$g_x = 100$$

$$g_y = 1$$

(c) Retentivity

$$Br = \frac{Go \ \mu o \ g_x \ (e_y)_r}{g_y \ [As/Ac - N]} \quad \text{in gauss}$$

Where, $(e_y)_r = 1/2X$ Intercept

4.2.3. Resistivity measurement: The resistivity of the samples was determined by the four probe method. The basic model for the measurements is indicated in Figure.4.6.Four sharp probes are placed on a flate surface of the material to be measured, current is passed through the two outer electrodes, and the floating potential is measured across the inner pair. The probes are collinear and equally spaced. The probes are mounted in a Teflon bush, which ensure a good electrical insulation between the probes. A Teflon spacer near the tips is also provided to keep the probes at equal distance. The probe arrangement is mounted in a suitable stand. The experimental circuit used for measurement is illustrated schematically in Figure 4.7.A nominal value of probe spacing which has been found satisfactory is an equal distance of 2.0mm between adjacent probes. The resistivity of samples was then calculated from the following formula:

$$\rho = \frac{\rho_0}{G_7 \ (W/S)}$$

Where: $\rho_0 = (V/I) \ x2\pi S$

The function G_7 (W/S) may be obtained from Table-4.1 for the appropriate value of (W/S).

W = Thickness of the sample

S = Distance between probes (i.e. 2mm)

I = Current in mA

V = Voltage in mV

The required dimensions of the surface plates, used for resistivity measurement were 1mm thickness (max.) × 5mm breadth (mim.) × 12mm length (mm)

4.2.4. Metallographic and SEM examinations: The metallographic examination was employed to study the effect of grain coarsening, pore morphology and microstructure on the magnetic characteristics of the material. The hot rolled and annealed samples were mounted with the help of hot mounting and polished for metallographic and SEM examinations. The SEM examination was employed to reveal the porosity/inclusions in the alloy system. Conventional polishing techniques, such as grinding on emery papers up to 4/0 grade and polishing on the cloth were used in the preparation of these samples for optical metallographic and SEM examinations. During cloth polishing, the polishing cloth with fine grains and abrasive slurry of 1 to 0.1 micron ferric oxide red (about 85% Fe_2O_3) powder were used. The optical metallographic analysis includes volume percentage of porosity and grain size measurement. The microstructures were taken at the cross section of the rolled and annealed wires. Additionally microstructures were taken also along rolled surfaces of the rolled and annealed wires. The microstructures were studied in uneatched and etched condition. The etching reagent was freshly prepared by 4% nitric acid and 96% methanol which is known as 4%nital.Sometimes, repeated etching and polishing was applied in order to reveal pores and grain boundaries clearly. The volume percentage porosity was measured by the optical microscope.

4.2.5. Hardness measurement: For hardness measurement, the mounted specimens and also the forged specimens both were polished using emery paper up to 4/0 grade. Hardness of the forged and homogenized samples as well as hot rolled and annealed samples were carried on a Brinell hardness testing machine (BHN).The load of 31.25 Kg for forged and homogenized samples and

15.625 Kg for rolled and annealed samples were applied for 20 second. At least four indentations were made on each sample of forged and homogenized as well as rolled and annealed and the average hardness is reported in the figure 5.1-5.2.

TABLE -4.1

The function G7 (W/S) corresponding to W/S (ratio of thickness of the sample to distance between probes):

S.No.	W/S	G7(W/S)
1	0.100	13.863
2	0.141	9.704
3	0.200	6.931
4	0.33	4.159
5	0.500	2.780
6	1.000	1.504
7	1.414	1.223
8	2.000	1.094
9	3.333	1.0228
10	5.000	1.0070
11	10.000	1.00045

```
┌─────────────────────┐                    ┌─────────────────────┐
│ Atomized Iron Powder│                    │ Fe-P Alloy Powder   │
│                     │                    │ (Master alloy)      │
└─────────────────────┘                    └─────────────────────┘
           │                                          │
           │        ┌──────────────────────┐          │
           └───────▶│ Mixing/Blending by ball│◀────────┘
                    │ mill machine for 2-3 hrs│
                    └──────────────────────┘
                                │
                    ┌──────────────────────┐
                    │ Encapsulation in a mild│
                    │ steel capsule          │
                    └──────────────────────┘
                                │
                    ┌──────────────────────┐
                    │ Heating the capsule in │
                    │ H₂ atmosphere          │
                    └──────────────────────┘
                                │
                    ┌──────────────────────┐
                    │ Forging the capsule    │
                    └──────────────────────┘
                                │
                    ┌──────────────────────┐
          ┌─────────│ Homogenization of slab │
          │         └──────────────────────┘
          ▼                     │
┌──────────────┐    ┌──────────────────────┐    ┌──────────────────────┐
│ With mild steel│   │ Removal of mild steel  │──▶│ Samples are prepared   │
│ skin           │   │ skin                   │   │ from forged slabs      │
└──────────────┘    └──────────────────────┘    └──────────────────────┘
          │                     │                             │
          └────────┐┌──────────────────────┐                 │
                   └▶│ Hot rolling            │                │
                    └──────────────────────┘                 │
                                │                             │
          ┌─────────┌──────────────────────┐                 │
          │         │ Cold   rolling         │                │
          ▼         │ (Final two passes)     │                │
┌──────────────┐    └──────────────────────┘                 │
│ Removal of skin│              │                             │
└──────────────┘    ┌──────────────────────┐                 │
          └────────▶│ Samples are prepared   │                │
                    └──────────────────────┘                 │
                                │                             │
                    ┌──────────────────────┐                 │
                    │ Coating the samples    │                │
                    └──────────────────────┘                 │
                                │                             ▼
                    ┌──────────────────────┐    ┌──────────────────────┐
                    │ Annealing              │──▶│ Testing and            │
                    └──────────────────────┘    │ Examination of the     │
                                                 │ samples                │
                                                 └──────────────────────┘
```
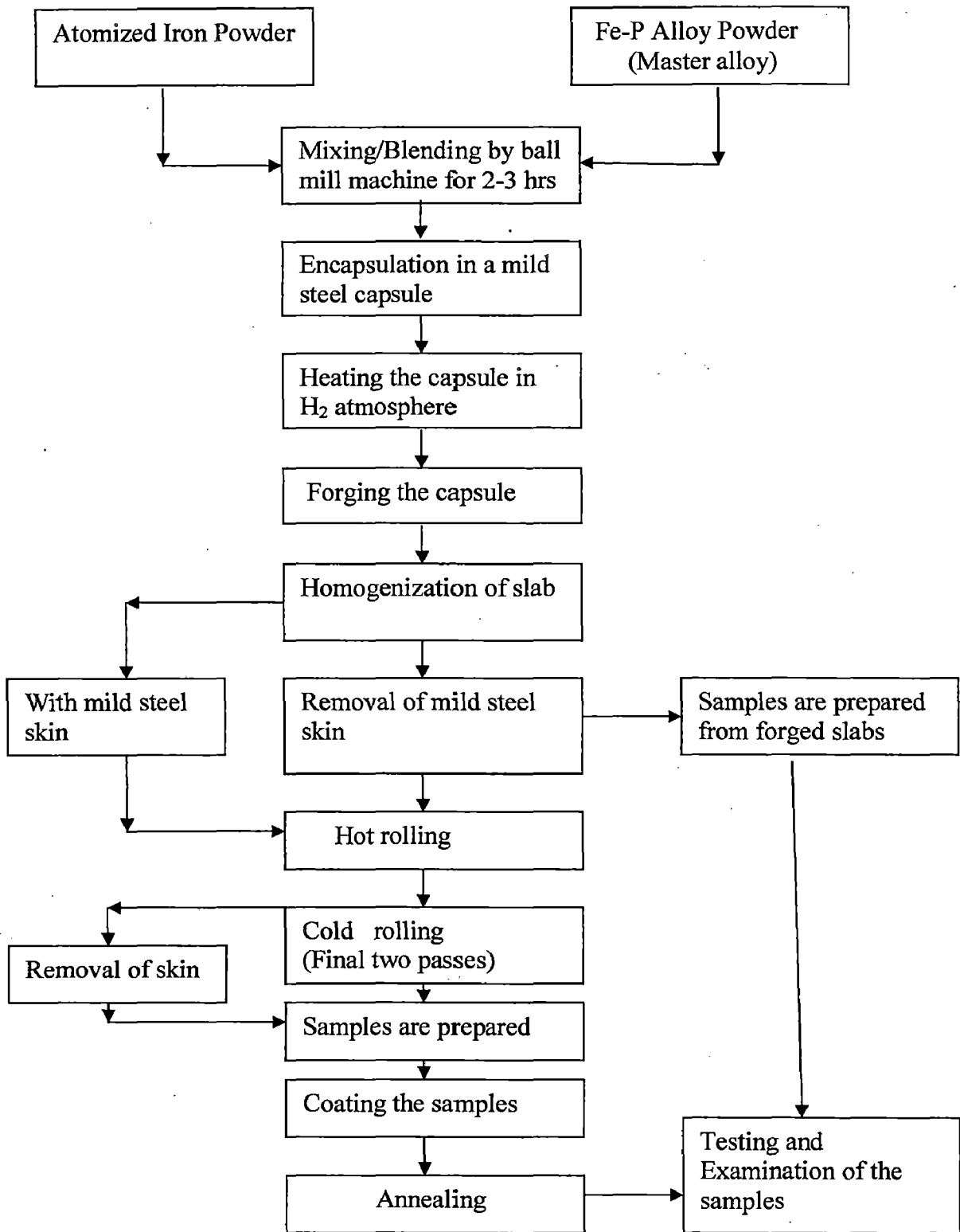
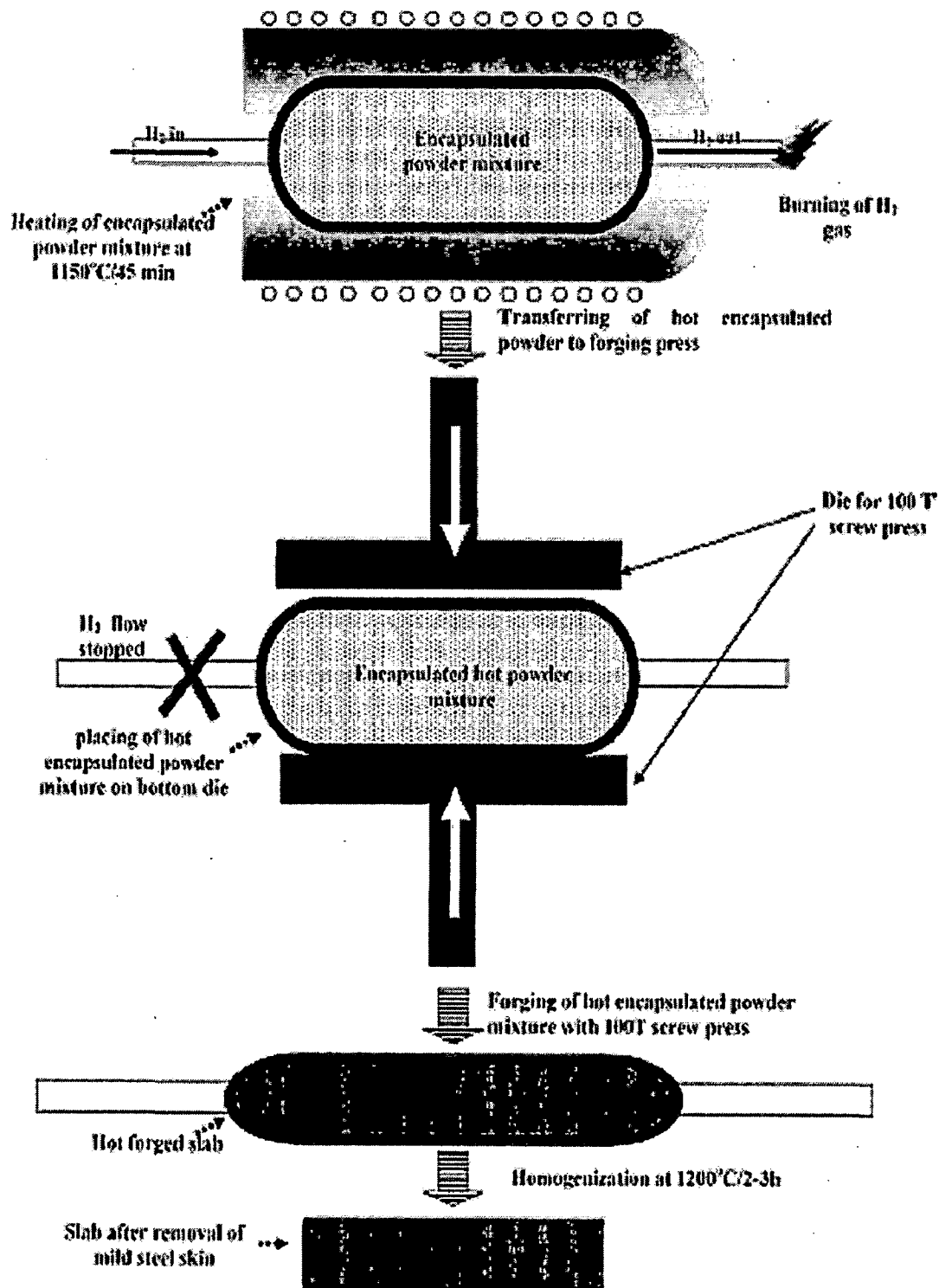Figure 4.3: Process flow chart

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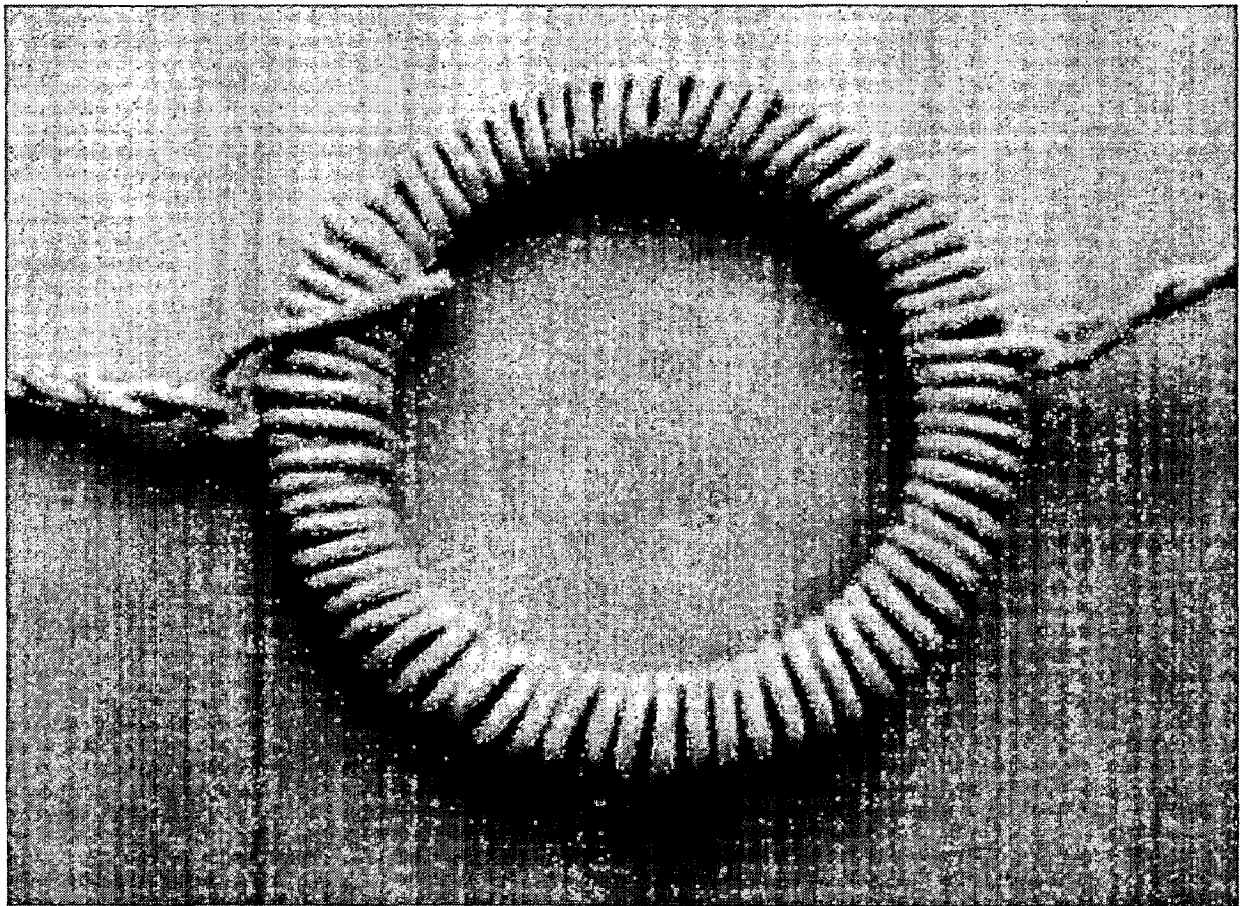Figure.4.4: Schematic diagram of Hot forging Process [26].

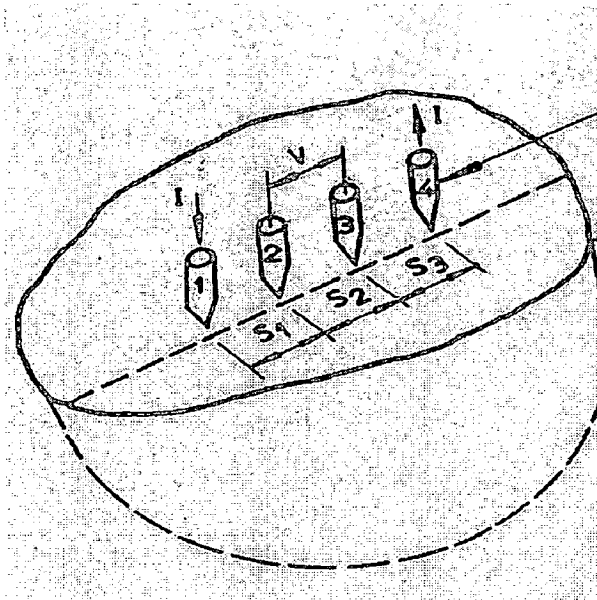Figure. 4.5: Primary and secondary winding on the ring/toroid sample

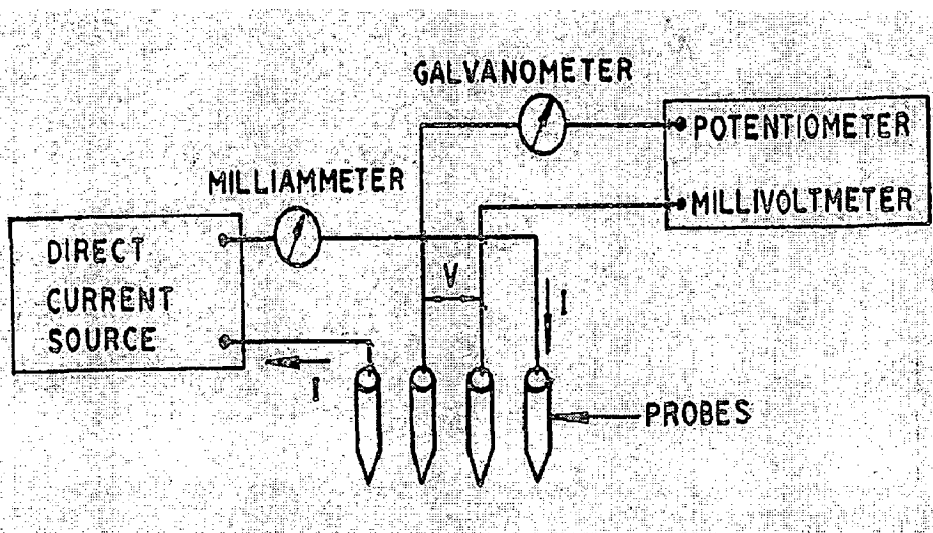Figure 4.6: Model for the four probe Resistivity Measurements.

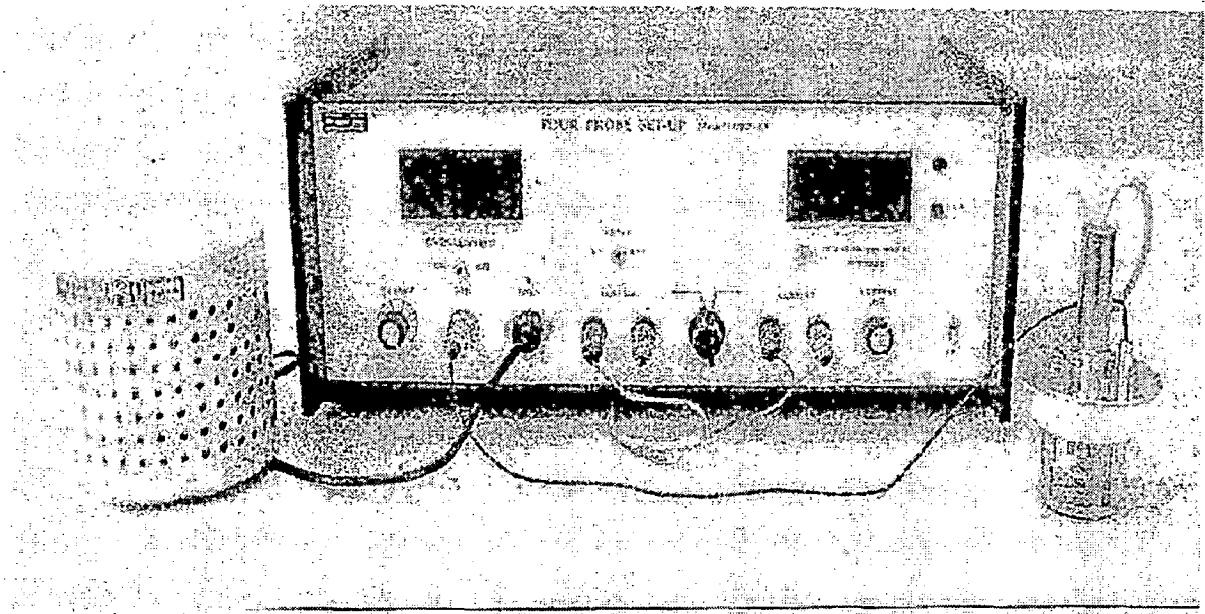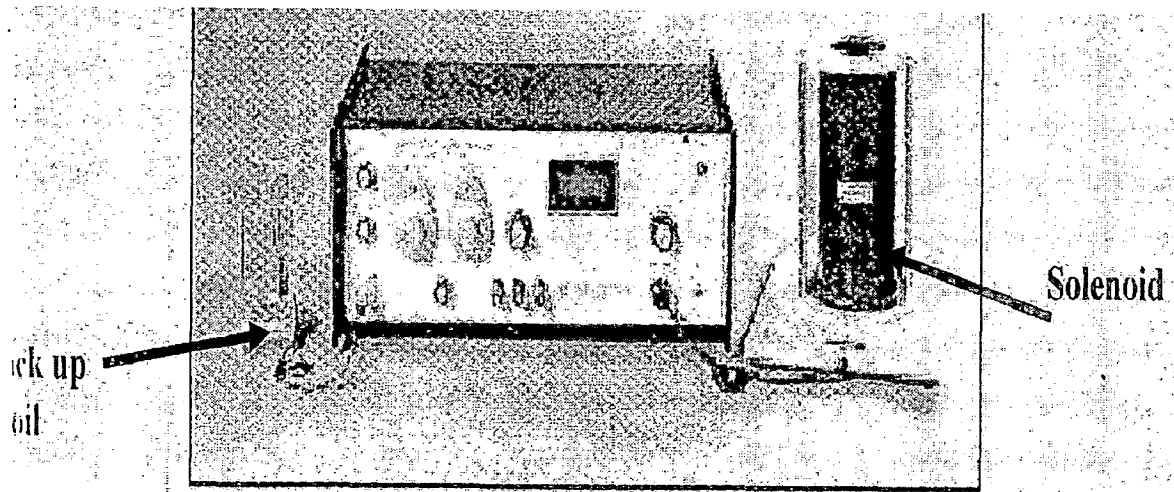Figure.4.7: Circuit used for Resistivity Measurements

Figure.4.8: Four Probe Set-Up

Figure.4.9: Hysteresis Loop Tracer [Model HLT-111]

CHAPTER 5
RESULT AND DISCUSSIONS

This chapter deals with the detailed characterization of magnetic materials developed in the present investigation as per the procedure laid down in chapter-4.The results are shown in the form of tables, figures, and microphotographs. In most of the cases the data on the various aspects of processing are clubbed together in a single table and/or placed at one place so that it is helpful in discussing the results then and there. The relevant points emerging out from over all results are further discussed at the end of this chapter.

5.1 DEVELOPMENT OF METHODOLOGY FOR THE PRODUCTION OF THIN SHEETS THROUGH HOT FORGING OFENCAPSULATED POWDER AND ROLLING

In order to develop the methodology for producing alloys in the form of thin sheets and wires through powder forging and rolling route, a large number of forgings were initially made using atomized iron powder and iron-phosphorous master alloy powder to have binary Fe-P alloys containing phosphorous in the range from 0.30-0.80wt%.The powders were encapsulated, heated in dry hydrogen atmosphere in a tubular furnace, forged, homogenized and rolled. All the process parameters of shaping namely holding temperature and time prior to forging, forging temperature, homogenization and rolling temperature etc. were first optimized to produce metallurgically sound forged and rolled sheets.

5.1.1 Observations and Results

A. Densification and Hardness during forming

Five powder metallurgical alloys were made in the present investigation.

These are as follows:

1. Fe-0.30P
2. Fe-0.35P
3. Fe-0.45P
4. Fe-0.65P
5. Fe-0.80P

All alloys were made by hot powder forging using channel die where side wall restriction to metal flow was imposed. Powder blend were taken for making these alloys.

Forged and homogenized slabs were characterized in terms density and microstructure. The micro structural characterization includes porosity measurement and grain size measurement. The forged and homogenized slabs were hot rolled and annealed to make thin sheets and wires for characterization purpose. As forged and homogenized as well as rolled and annealed microstructures with the experimentally measured volume percentage of porosities in Table 5.1. It may be inferred therefore that binary Fe-p alloys in the range of 0.30wt%P-0.80wt%P show increased density levels on forging in the range of 7.74-7.82 gm/cc.

Table 5.1: Experimental volume percentage of porosities of the alloys

Materials	Forged density(g/cc)	Rolled and annealed density(g/cc)	Volume % porosity in forged slabs	Volume % porosity in rolled and annealed
Fe-0.30P	7.60	7.74	5.66	3.16
Fe-0.35P	7.64	7.76	5.35	3.00
Fe-0.45P	7.67	7.79	4.83	2.50
Fe-0.65P	7.69	7.80	4.50	2.25
Fe-0.80P	7.71	7.82	3.50	1.65

Hardness at the surface of the forged and homogenized sheets as well as rolled and annealed wire samples was measured using Brinell hardness testing machine. The load of 31.25 kg for forged and homogenized samples and 15.625

Kg for rolled and annealed samples were applied for 20 second. Indentation marks were recorded and shown in Figure 5.3.The scattered values of hardness were recorded and shown in the form of plot in the Figure 5.1 and 5.2.It was observed that hardness increases with phosphorous alloying addition. Phosphorous causes more hardening of ferrite than the silicon in solid solution with iron.

B.Magnetic Properties of rolled toroids or rings made by powder forging and rolling of encapsulated binary Fe-P alloy powders

To have a detailed magnetic characterization for AC and DC application of rolled and annealed sheets evolved in the present study, five sheets made from different containing Phosphorous Fe-P alloys were used to form toroid or ring for determination of magnetic properties by Hysteresis loop tracer [Model: HLT-SOFT] equipment. However, the samples in the form of wire of same alloys were prepared for determination of magnetic properties by Hysteresis loop tracer [Model: HLT-111].

Firstly, the measurements were carried out on model: HLT-111 tracer equipment and the measured values were reconfirmed by the model: HLT-SOFT tracer equipment which is more sensitive and reliable. This equipment is based on advanced technology and fully computerized. It was observed that the properties measured by model: HLT-SOFT tracer are better than Model: HLT-111 with some differences.
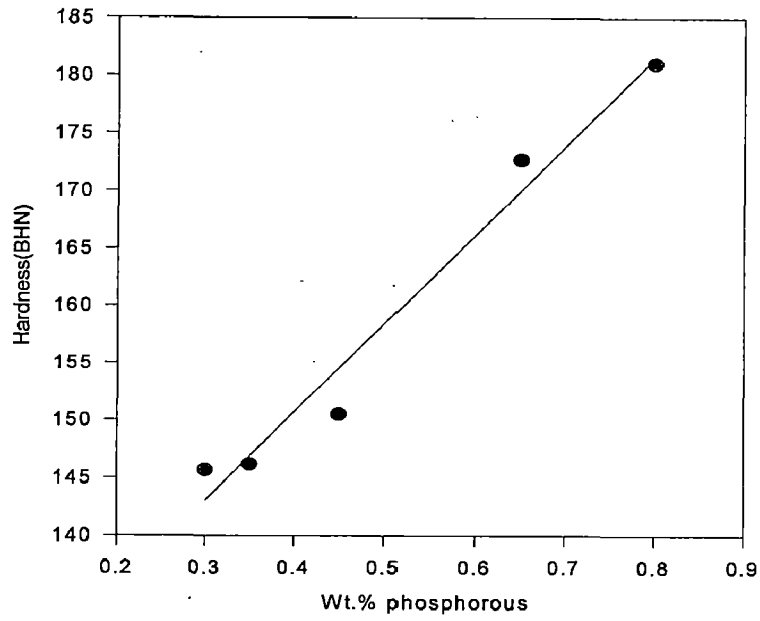
Figure.5.1.Variation of hardness with phosphorous for Fe-P alloys as forged and homogenized sheets
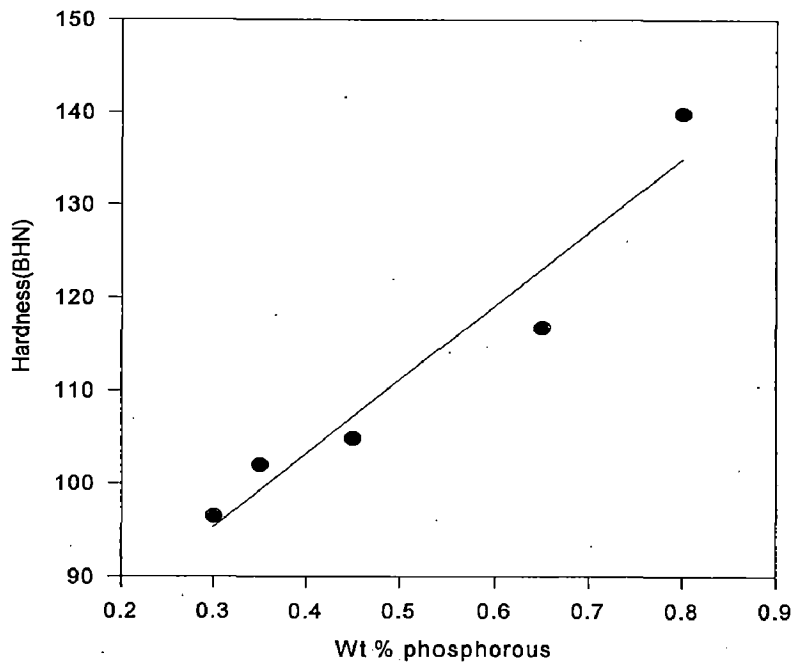
Figure.5.2.Variation of hardness with phosphorous for Fe-P alloys as rolled and annealed wires.

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Fe-0.30P Alloy

Fe-0.35P Alloy

Fe-0.45P Alloy

Fe-0.65P Alloy

Fe-0.80P Alloy

$100\mu m$
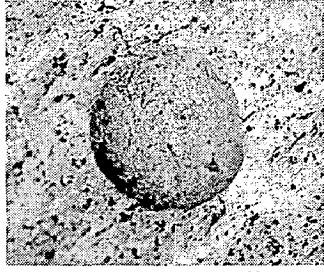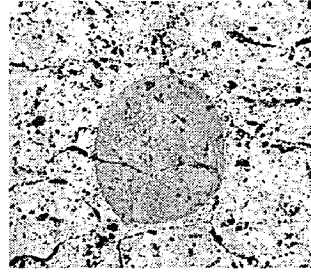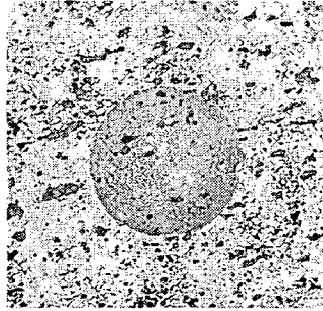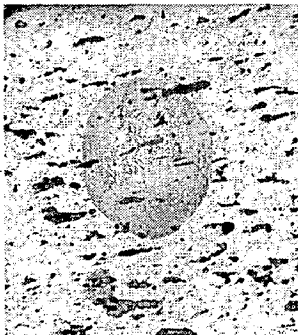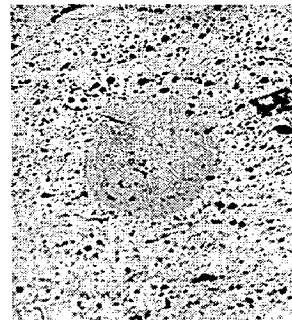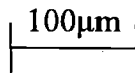
Figure 5.3: Brinell hardness indentations, at the section along rolling direction of the rolled and annealed wires, using 15.625 kg load for 20 second.

Since the DC or direct current applications are characterized by a constant applied field (from a battery type device).The most common DC applications are found in automobiles. Key magnetic characteristics for DC applications are permeability, coercive force, and saturation induction. In the present investigation toroid samples were tested and plotted the hysteresis curves under DC conditions by hysteresis loop tracer [model: HLT-SOFT], at 50 Hz. Some of them hysteresis curves have been reported in Figure 5.5-5.6 for the representation point of view. The maximum applied field was about 60 Oe.The results obtained are given in the Table 5.2. [7] has reported the saturation magnetization from 14,700 gauss to 15,200 gauss with maximum at 0.6% P in Fe-P alloys while in the present investigation the saturation magnetization has been obtained from 10,716 gauss to 17,500 gauss with a maximum at 0.45%P at same applied field. Moreover, the saturation magnetization 14,300 gauss has been also reported in [2] at 0.45%P of Fe-P alloy at 100 Oe applied field. It was observed that when the applied magnetic field begins to vary with time, the shape of the B-H curve becomes oval and the total loop area increases dramatically. The cause of this broading of the B-H curve is the detrimental effect of the eddy currents that results in a lowering of the magnetic performance at higher frequencies.

However, AC or alternating current applications are characterized by a constantly changing applied field. Key magnetic parameters in AC applications are permeability, saturation, and total core losses resulting from the alternating magnetic field [38].In the present investigation the hysteresis curves for same alloys were also plotted under AC condition at 150Hz by same equipment some of them curves have been shown in Figure 5.7-5.8.The results obtained are reported in Table 5.3.The methods used for measurement have been described with step by step in the article 4.2.2.

Table 5.2: Coercivity, saturation. magnetization, retentivity and permeability values of the alloys under DC mode.

Alloy	Coercivity(Oe)	Sat.magnetization(G)	Retentivity(G)	Permeability(µ)
Fe-0.30P	1.241	10716	8121	185
Fe-0.35P	1.206	15599	8354	270
Fe-0.45P	0.384	17499	10836	294
Fe-0.65P	0.353	16666	8229	288
Fe-0.80P	0.375	14145	6402	241
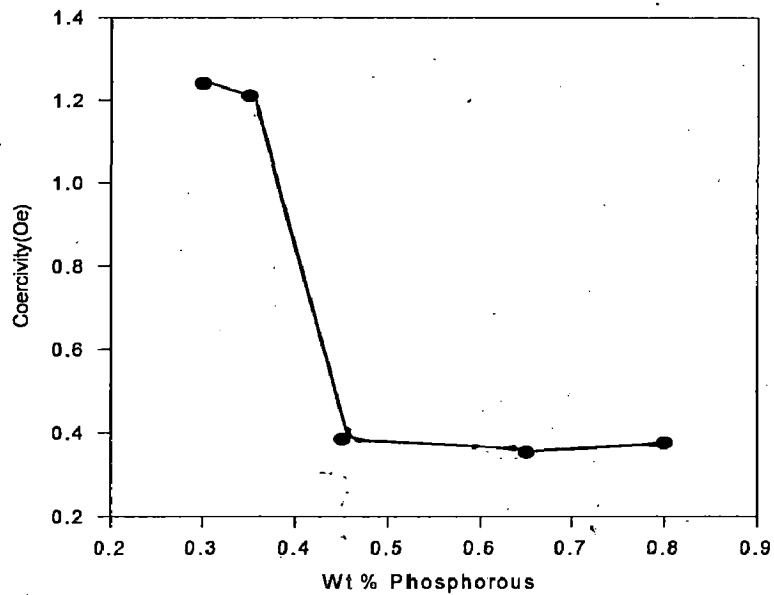
Figure5.4: Variation of Coercivity with phosphorous

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File View Help

Final Results

Specimen Code : .65P

Alloy System : Fe-P

Prior Treatment : xxx

Permeability at 57.79 Oe:185

Magnetization (Bs): 10716 Gs

Coercivity (Hc): 1.241 Oe

Remanance (Br): 8121 Gs

Hyst loss/Kg/Cycle(Wh): 0.401 Watt

01 June, 2009 :12:21

Peak Permeability: 1455 Gs/Oe

Initial Permeability: 502 Gs/Oe

Sample: Ring

MPL : 14.140 cms

Area :0.035 sq cm

Primary Turns : 72

Secondary Turns : 10

Density : 7.76 gms/cc

30000 Gs

-100.00 Oe

100.00 Oe

perm 50000

-30000 Gs

Ready

NUM

20K 2M 2M 20M .1A .2A .5A 1A 2A 5A 10A 20A O R D E CGS MKS Loss M Mag Auto AC DC

start HYSTERESIS LOOP T...

Figure.5.5: Hysteresis curves for Fe-0.30P alloy under DC mode.

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The screenshot shows a Hysteresis Loop Tracer window with the following content:

File View Help

Final Results

Specimen Code : .45P
Alloy System : Fe-P
Prior Treatment : xxx
Permeability at 59.59 Oe:294
Magnetization (Bs): 17490 Gs
Coercivity (Hc): 0.384 Oe
Remanance (Br): 10836 Gs
Hyst loss/Kg/Cycle(Wh): 0.538 Watt

01 June, 2009 :11:18

Peak Permeability: 607 Gs/Oe
Initial Permeability: 219 Gs/Oe

Sample: Ring
MPL : 12.570 cms
Area :0.100 sq cm
Primary Turns : 66
Secondary Turns : 10
Density : 7.76 gms/cc

30000 Gs
-100.00 Oe
100.00 Oe
perm 50000
-30000 Gs

Ready

20K 2M 2M 20M .1A 2A .5A 1A 2A 5A 10A 20A O R D E CGS MKS Loss Mag Auto AC DC

Figure.5.6. Hysteresis curves for Fe-0.45P alloy under DC mode.
It was observed that the noise in hysteresis curves (loops) may be minimized by increasing the number of toroids with proper insulation from one

80

another and be closely packed. Shorting of laminations due to burr on the edges also must be avoided. In the present investigation, the multi stand Teflon coated wire has been used for primary and secondary windings.

Table 5.3: Saturation magnetization, permeability and loss/kg values of the alloys under AC mode.

Alloy	Sat.magnetization(G)	Permeability(μ)	Loss/kg(Wc)in watt
Fe-0.30P	15677	271	16.797
Fe-0.35P	20182	349	32.173
Fe-0.45P	19462	326	39.506
Fe-0.65P	18072	313	17.15
Fe-0.80P	16867	288	12.239
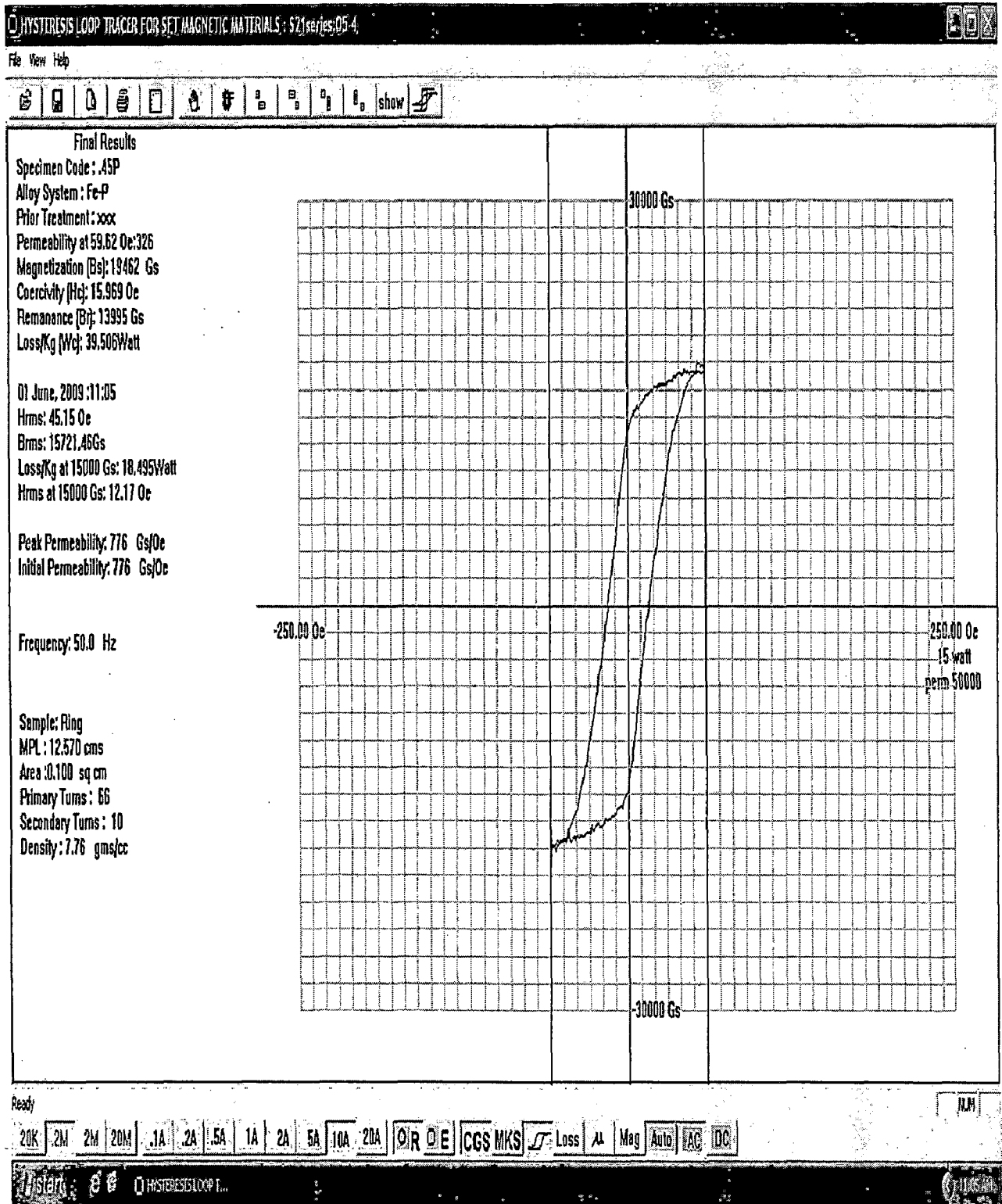
Figure 5.7: Hysteresis curves for Fe-0.45P alloy under AC mode.

File View Help

Final Results
Specimen Code : .80P
Alloy System : Fe-P
Prior Treatment : xxx
Permeability at 58.62 Oe:288
Magnetization (Bs): 16867 Gs
Coercivity [Hc]: 6.012 Oe
Remanance [Br]: 7595 Gs
Loss/Kg [Wc]: 12.239Watt

01 June, 2009 :10:48
Hrms: 46.37 Oe
Brms: 12783.44Gs
Loss/Kg at 15000 Gs: 9.846 Watt
Hrms at 15000 Gs: 30.63 Oe

Peak Permeability: 870 Gs/Oe
Initial Permeability: 184 Gs/Oe

Frequency: 50.0 Hz

Sample: Ring
MPL : 14.140 cms
Area :0.050 sq cm
Primary Turns : 73
Secondary Turns : 10
Density : 7.76 gms/cc

30000 Gs

-250.00 Oe

250.00 Oe
15 watt
perm 50000

-30000 Gs

Ready

20K 2M 2M 20M .1A .2A .5A 1A 2A 5A 10A 20A O R O E CGS MKS Loss Mag Auto AC DC

Start   HYSTERESIS LOOP T...

Figure 5.8: Hysteresis curves for Fe-0.80P alloy under AC mode.

In the present investigation two samples were made under similar processing parameter of Fe-0.35P and Fe-0.65P to test the DC and AC magnetic properties. These samples were prepared by same route. It did not require shaping operation for skin removal because the forged and homogenized product were hot rolled with mild steel skin up to 3mm to obtained less than 1mm thickness sheet by removing the skin by grinder. The main purpose of this to

avoid the oxidation by which material can be saved to great extend during hot rolling, to save the machining time and to obtain the better surface finished product. The hysteresis loop of these alloy under DC and AC conditions have been shown in the Figure 5.9-5.12.
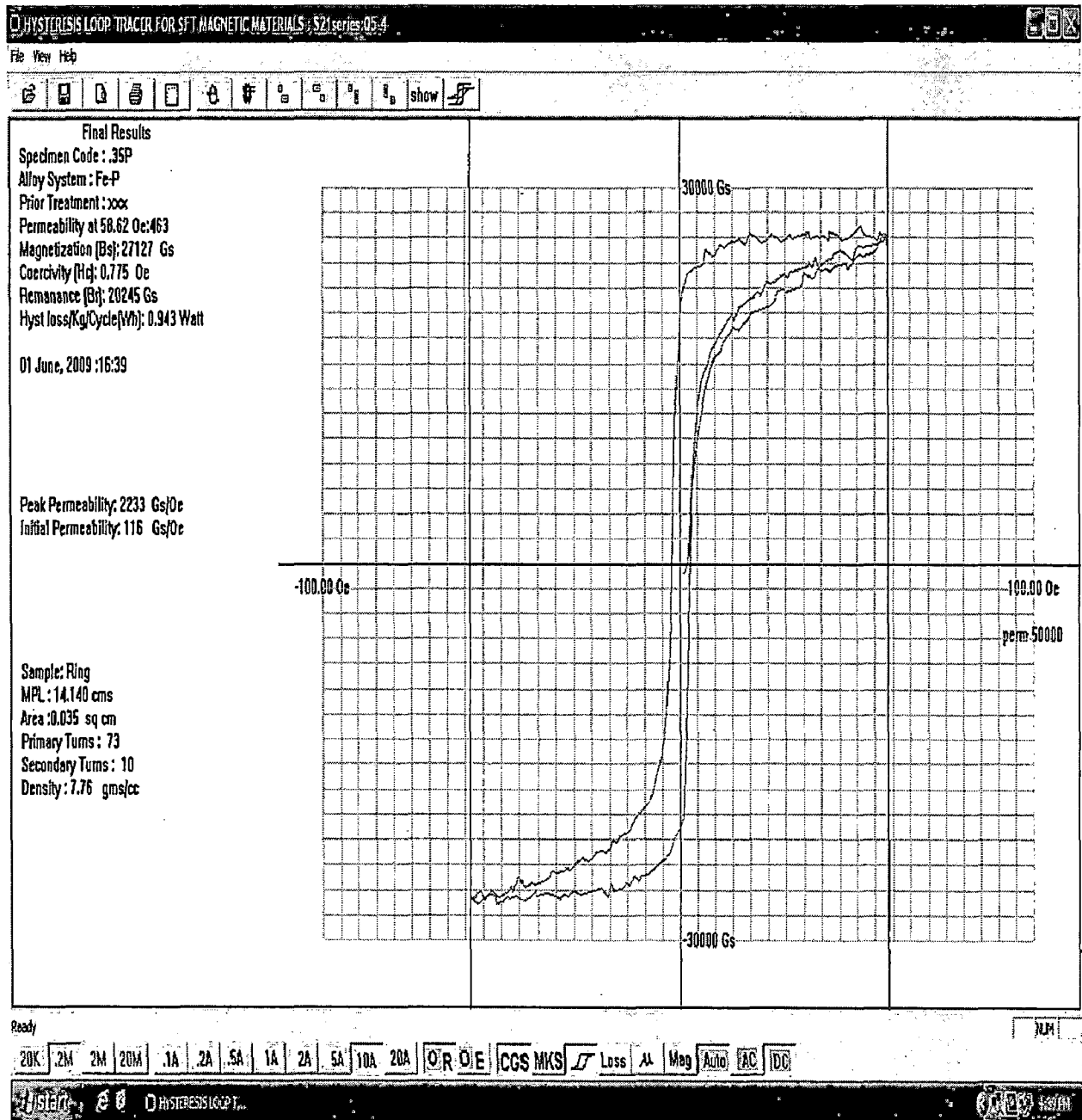
Figure.5.9: Hysteresis curves for Fe-0.35P alloy (rolling with skin) under DC mode.
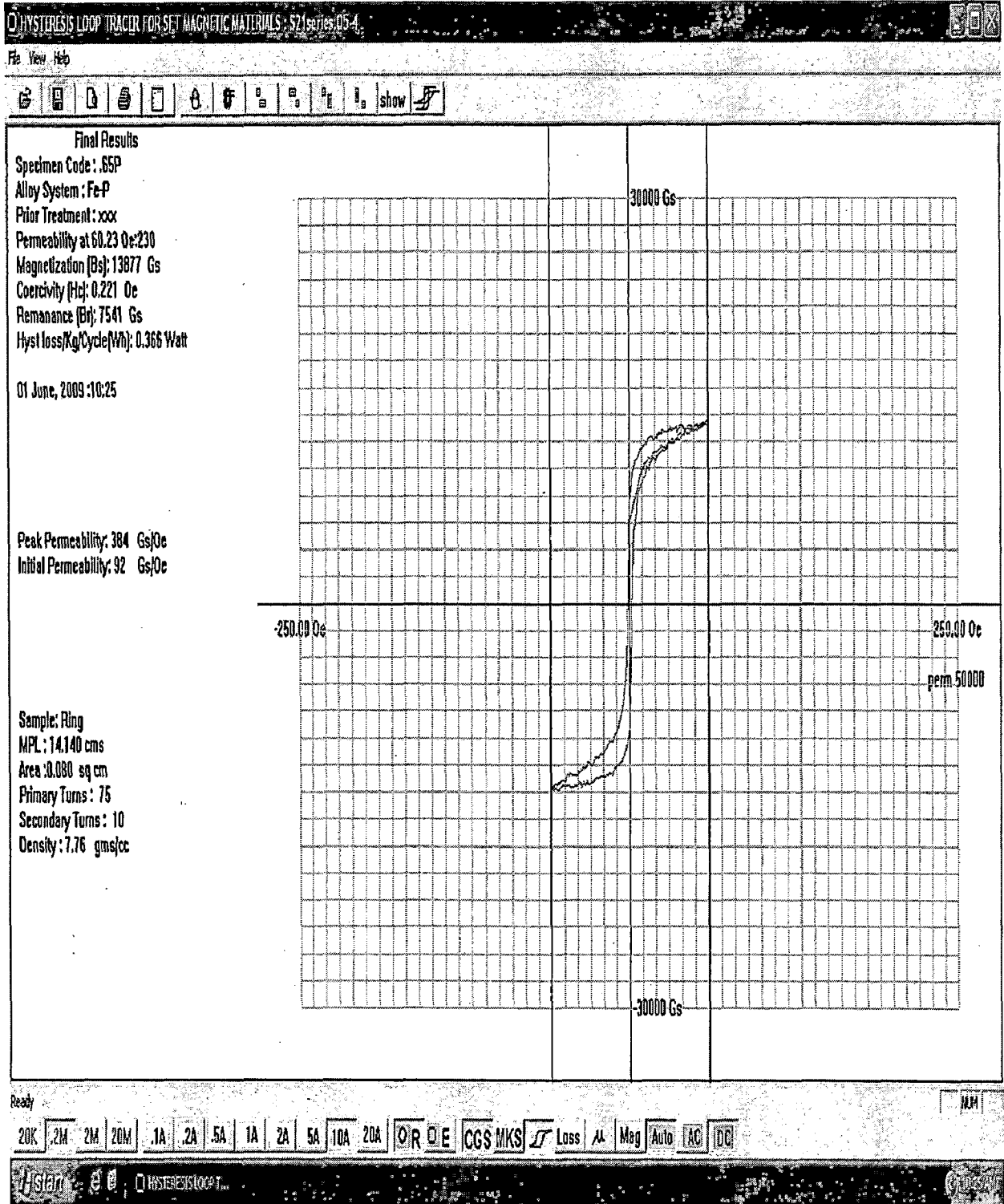
Figure.5.10: Hysteresis curves for Fe-0.65P alloy (rolling with skin) under DC mode.

File View Help

Final Results
Specimen Code : .35P
Alloy System : Fe-P
Prior Treatment : xxx
Permeability at 58.62 Oe:530
Magnetization (Bs): 31046 Gs
Coercivity (Hc): 10.865 Oe
Remanence (Br): 22023 Gs
Loss/Kg (Wc): 47.236Watt

01 June, 2009 :16:44
Hrms: 45.11 Oe
Brms: 25147.46Gs
Loss/Kg at 15000 Gs: 13.638Watt
Hrms at 15000 Gs: 7.82 Oe

Peak Permeability: 1873 Gs/Oe
Initial Permeability: 1873 Gs/Oe

Frequency: 50.0 Hz

Sample: Ring
MPL : 14.140 cms
Area :0.035 sq cm
Primary Turns : 73
Secondary Turns : 10
Density : 7.76 gms/cc

30000 Gs

-100.00 Oe   100.00 Oe
                    15 watt
                    perm 50000

-30000 Gs

Figure.5.11. Hysteresis curves for Fe-0.35P alloy (rolling with skin) under AC mode.

Figure.5.12: Hysteresis curves for Fe-0.65P alloy (rolling with skin) under AC mode

As shown in Figures 5.9-5.12 the retention of mild steel skin does not affect magnetic properties. The samples rolled with skin also exhibited better surface finish as the skin protects the sample from extensive oxidation. It also results in significantly reduced material loss. During hot working the losses due to oxidation at 1273K account for up to 40% material loss if the skin is removed before rolling. Lastly retaining the skin also saves expensive and time consuming

machining on the shaper. Further, skin also significantly reduces formation of edge cracks during rolling.

C.Electrical resistivity and Hysteresis losses

It was observed that electrical resistivity increases linearly with increase in phosphorous content of the alloy up to the highest phosphorous content in Fe-P alloys, which is 0.80wt% for the present investigation. The alloying element formed solid solution with ferrite and distorted the ferrite lattice and thereby hindered the passage of electrons. The resistivity values of these alloys were recorded and shown in the Table 5.4.

Therefore, it may be inferred that from the point of view of having high electrical resistivity, the phosphorous content in the binary Fe-P alloys should be high, unless it adversely affects forming and/or magnetic properties.

Table 5.4: Electrical resistivity

Material	Resistivity($\mu\Omega$-cm)
Fe-0.30P	17.21
Fe-0.35P	18.04
Fe-0.45P	20.21
Fe-0.65P	23.69
Fe-0.80P	25.02

5.1.2. Processing and characterization of Fe-P alloys

The density of as forged 000Si000 alloy, which is produced by forging of encapsulated electrolytic grade iron powder, is 7.2 gm/cc and the density of sheet formed from this alloy is 7.3 gm/cc[1].Since the density of wrought iron at 20^0C is 7.87 gm/cc.The density of as forged Fe-0.45P(7.79 gm/cc) and Fe-0.80P(7.82 gm/cc) alloys produced in the present investigation by hot forging of

encapsulated powder and rolling are higher than the density obtained for conventionally processed Ancorsteel 45P(which is 7.45 gm/cc) and 80P(which is 7.41 gm/cc) when the alloy powders are compacted at 689.5 MPa(50tsi) and sintered at $1260^{0}C$[33].
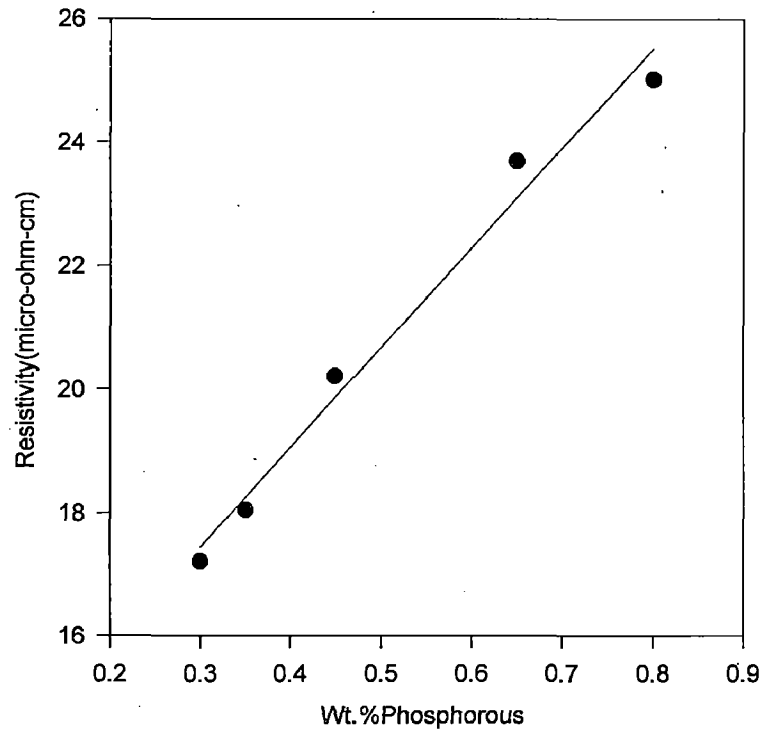
Figure.5.13: Variation of resistivity with phosphorous for Fe-P as rolled and annealed sheets

Thus the phosphorous addition in iron powder helps in increasing the final density when hot forging and rolling technique is employed for consolidating iron phosphorous based powder alloys. Increase in density by phosphorous addition has been reported earlier as well [9] when compacting and sintering route of shaping was adopted. However, in that case the reason was high compacting pressure followed by high temperature liquid phase sintering. In the present case nothing like phase sintering is involved and yet there is significant improvement in density (more than that of compacting and sintering route as stated above) that too at relatively low temperature processing.

It is therefore very clear that high rate forging followed by solid state homogenization (at temperature lower than conventional sintering temperatures) has brought about densification of greater magnitude. **Thus the solid state diffusion of phosphorous alone is capable of bringing about significant densification as reported in the present investigation. Moreover, the forming technique evolved in present investigation is more economical and simpler to adopt in comparison to conventional compacting and sintering technique and recently developed Ancordense technique where sophisticated equipments are involved hitherto employed in developing quality magnetic materials based on Fe-P system.**

Ancordense technology is a material and process combination that utilizes elevated temperature compaction technique to increase green density, green strength and decrease ejection forces [Properties of several [46].

Magnetic induction is closely related to applied field strength. However, in literature [9, 23, 38] it is given corresponding to very low field strength namely in the range of 15-40 Oersted.Therefore, it is difficult to compare the results obtained with others available in literature. However the values obtained in the present investigation indicate that the saturation magnetization at a given magnetizing force increases with phosphorous contents up to 0.65 wt. %.The higher induction of the phosphorous containing materials can be partially explained by their somewhat higher density [9].Moreover, saturation induction is less sensitive to processing variables and is primarily influenced by the amount and type of alloy system [10].

The test result for the measurement of magnetic properties of different alloys are given in Table 5.2.These results show that on increasing phosphorous from 0.30% to 0.65 %,Coercive force is lowered from 1.241 Oe to 0.353 Oe.The improvement in soft magnetic characteristics is obvious on increasing phosphorous content.

Resistivity is increased with increase in phosphorous content for Fe-P alloys, which is in agreement to the finding of earlier work [9, 29].It has been reported [29] that phosphorous additions increase the electrical resistivity of iron, to 19 µΩ-cm at 0.50% P and 22 µΩ-cm at 1% P.It has been also reported [34] that the resistivity increases linearly from 13.0 µΩ-cm for pure iron to about 29 µΩ-cm with 1% phosphorous.

The test results for the measurement of resistivity of different alloys are given in Table 5.4.These results show that on increasing phosphorous from0.30% to 0.80%, resistivity is increased from 17.21 µΩ-cm to 25.02 µΩ-cm.

Hysteresis losses are proportional to the area of the hysteresis loop. It has been reported in Table 5.2 that phosphorous reduces the coercive force, which means that the hysteresis loop becomes narrower and consequently the hysteresis losses are reduced. If the eddy current losses are measured at the same frequency, induction, thickness and density the losses only depend on the resistivity of the material [26].The eddy current is given by the following equation:

$$\text{Eddy current loss (W/kg)} = K \times \frac{t^2 \times f^2 \times B^2}{\rho \times d}$$

Where:

   K = Constant

   t = Thickness

   f = Frequency

   B = Induction B_{max} appropriate for the application

   ρ = Electrical resistivity of the material

   d = Density of the material

Thus it can be inferred stated that the binary Fe-P alloys prepared by using the technique evolved in the present investigation behave more or less similarly to the alloys which are prepared from powders using the conventional compacting and sintering technique as for as their resistivity is concerned. However, the

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present technique permits higher addition of phosphorous without any deleterious effect of shrinkage/distortion etc.It was also observed that the strips/sheets of length 60-70cm and thickness 3mm with mild steel skin are formed without any failure of Fe-P binary alloys with phosphorous contents from 0.30% to 0.80%.It has been shown in Figure 5.14 after 50% reduction in thickness of length 30cm.

5.2. METALLOGRAPHIC AND SEM EXAMINATION

The literature survey on powder processed Fe-P alloys gives very scanty and scattered information pertaining to microstructures of these alloys. In the present investigation attempt has been made to examine the pore and grain structure during processing of these alloys. All the structures are single-phase structures with porosities within or at the grain boundary regions. Quantity of porosity and as well as pore size appears to be very sensitive to the constitution of the alloy.

SEM examinations have been shown in Figure 5.21-5.24 in the present investigation of the alloys to ensure that whether the black spot shown in the metallographic examination were inclusions or porosities. This examination were carried out by secondary electron detector and back scattered electron on unetched and etched samples

5.2.1. As forged and homogenized microstructures: As forged and homogenized microstructure were etched (4% nital) to reveal the grain boundary and shown in Figure 5.16. Pores are randomly distributed inside the grains. There is no evidence of presence of iron phosphide phase along the grain boundaries even at such a high percentage of phosphorous, which is normally reported as double boundaries in compacted and sintered systems. The volume

92
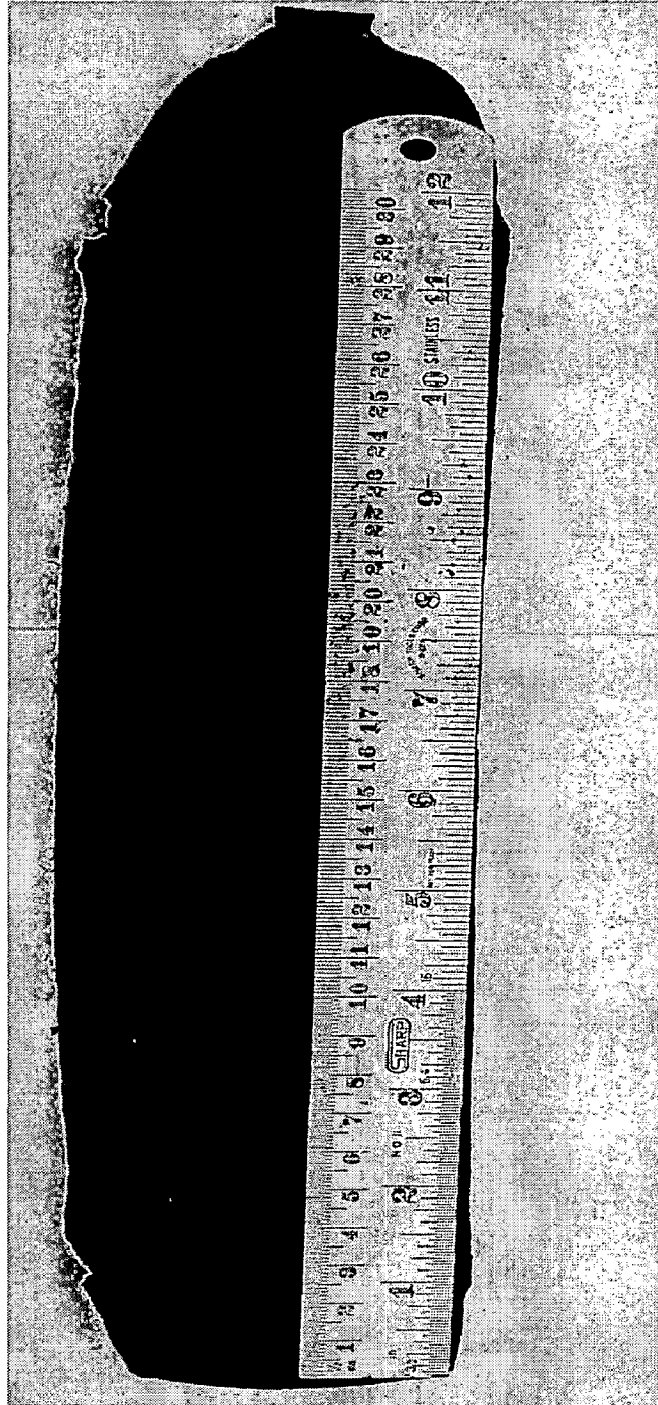
Figure.5.14: Rolled strip/sheet powder forged alloy of length 30cm and thickness 6mm with skin.
% porosities have been reported in Table 5.1 of the alloys in the present investigation.

5.2.2. Rolled and annealed microstructures: There are two sections of rolled and annealed wires have been made for microstructure analysis. The cross section of rolled wire along rolling direction showed porosities which are

elongated towards the rolling direction. The cross section of the wires showed the almost rounded porosity. Large and small round pores are observed in the microstructures. This was due to the fact that in the section rolling the compressive forces acted from the two directions uniformaly.The rolled and annealed sections of wire were etched to reveal grain boundaries and are shown in Figure 5.18 and 5.20. The microstructures of the system whose phosphorous contents is with in 0.30-0.80% do not show significant variation in pore and grain structure except by way of their shape and size in accordance with there respective density values. The volume % porosities have been reported in Table 5.1 of the alloys in the present investigation.

5.3. GENERAL DISCUSSIONS

Hot powder forging route used in the present investigation for making laminations out of Fe-P based powder alloys appears to be very effective for high density, good combination of mechanical properties, high resistivity and finally good set of magnetic properties. This is obvious from the Table 5.2-5.3 where all the alloys have been documented for their magnetic characteristics. On the basis of information given in the table, it can be said that the alloys are suitable for different kinds of soft magnetic application.

Mechanical and magnetic properties often have not been co-related in the literature, except for the fact that mechanically soft materials may also be magnetically soft. However, Fe-p based powder alloys are exception to this fact. It has been observed while following compacting and sintering route and as well as duly supported by the present investigation following forging route, that strengthening obtained by sintering/homogenization/annealing/stress relieving improves magnetic characteristics and also improves elongation values. It requires revision of our concept that stronger the Fe-P based alloys better are the magnetic properties. More appropriately it is not the strength but the toughness that should be linked with soft magnetic characteristics. Toughness takes into account of three important features simultaneously viz. strength,

elongation and density. The present investigation therefore, clearly sums up the fact that in order to get best magnetic characteristics out of powder Fe-P based alloys; they must be toughened to maximum extent [22].
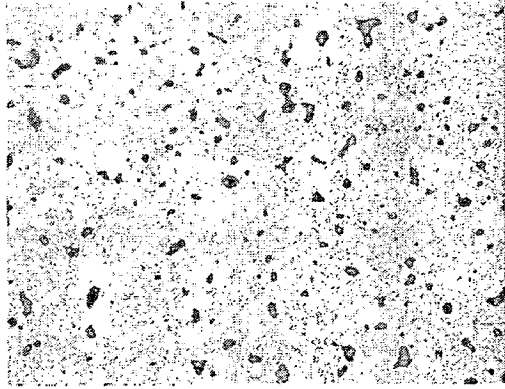
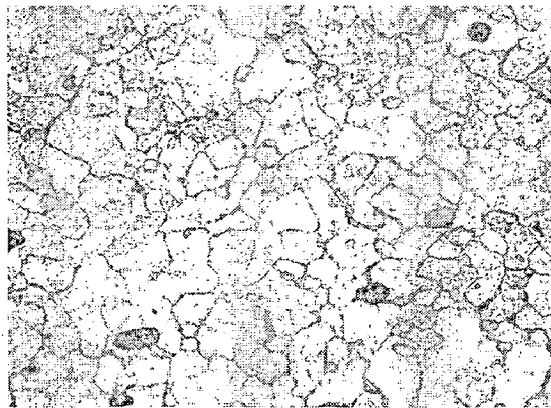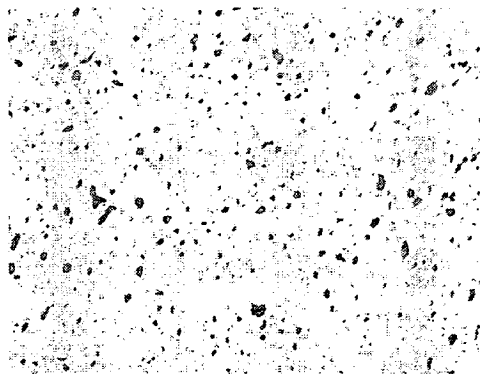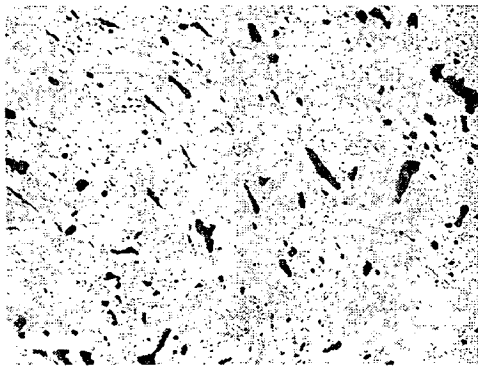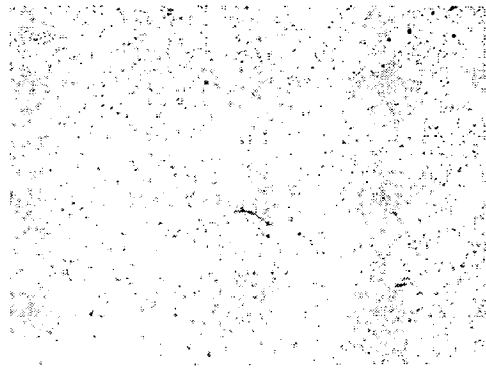Fe-0.30P Alloy
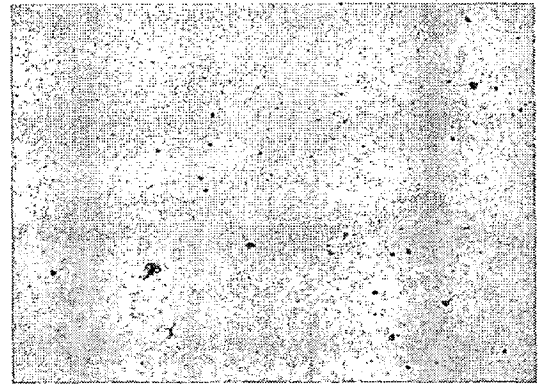
Fe-0.35P Alloy
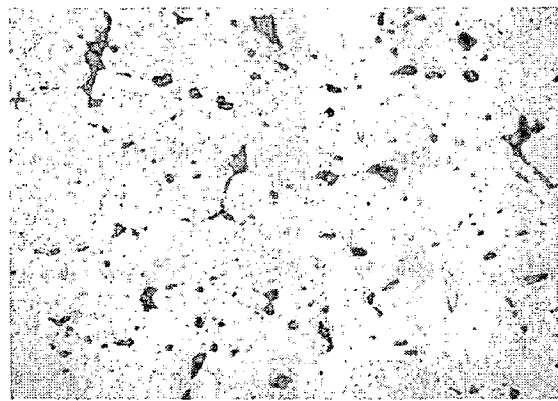
Fe-0.45P Alloy
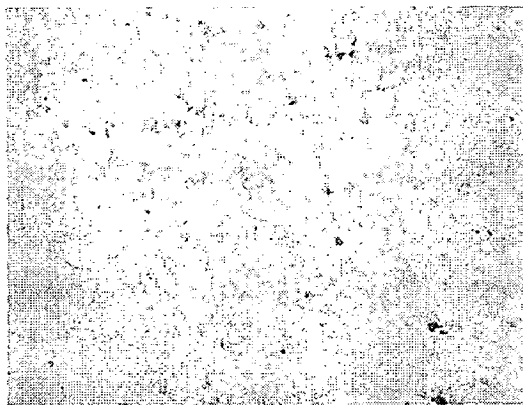
Fe-0.65P Alloy

100μm

Fe-080P Alloy

Figure.5.15. Microstructure (unetched) of forged and homogenized samples. Figures show the porosity distributed along grain boundaries as well as inside the grains for entire range of phosphorous in these alloys.

Fe-0.30P Alloy
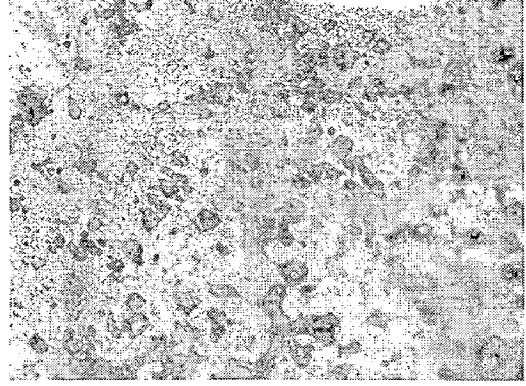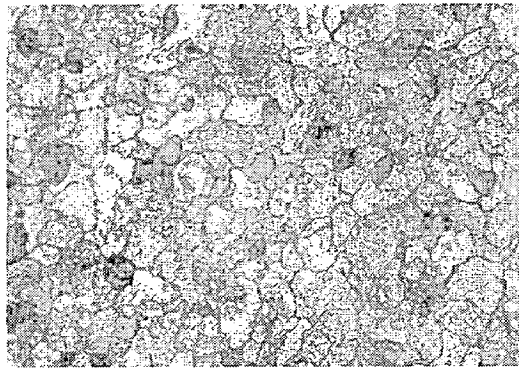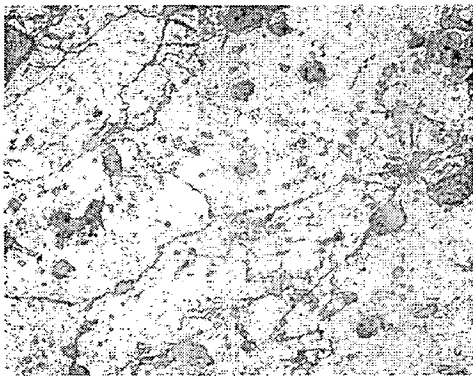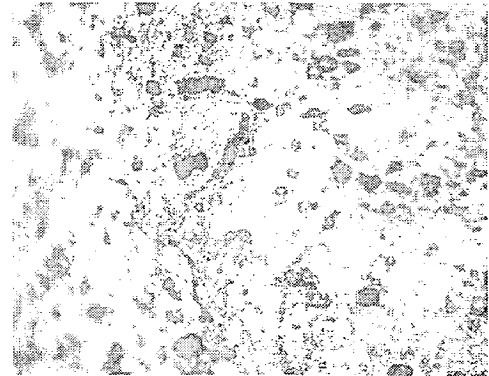
Fe-0.35P Alloy

Fe-0.45P Alloy

Fe-0.65P Alloy

Fe-0.80P Alloy

|—50µm—|
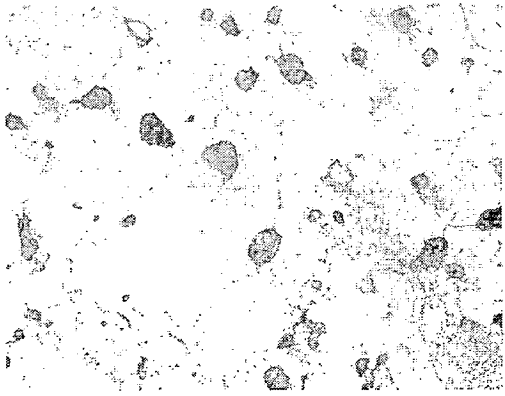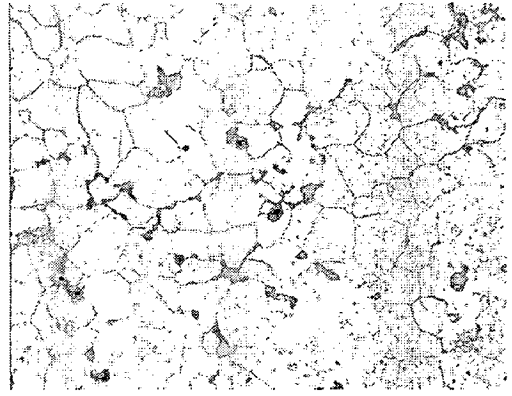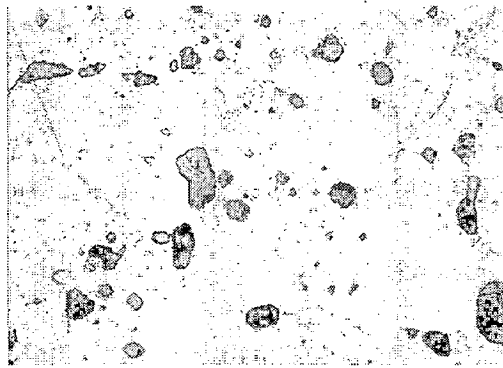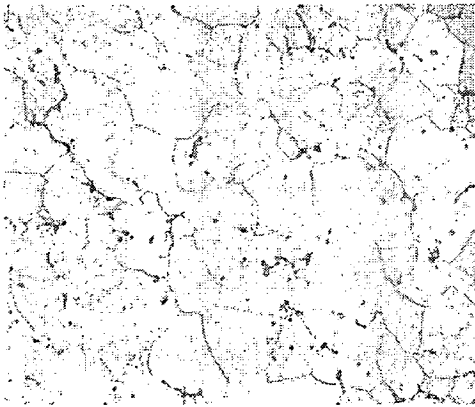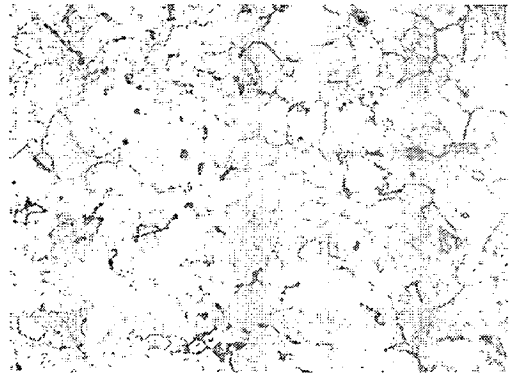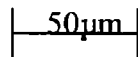
Figure.5.16.Microstructure (etched) of forged and homogenized samples. Figures show the porosity distributed along grain boundaries as well as inside the grains for entire range of phosphorous in these alloys. Some of them are elongated.
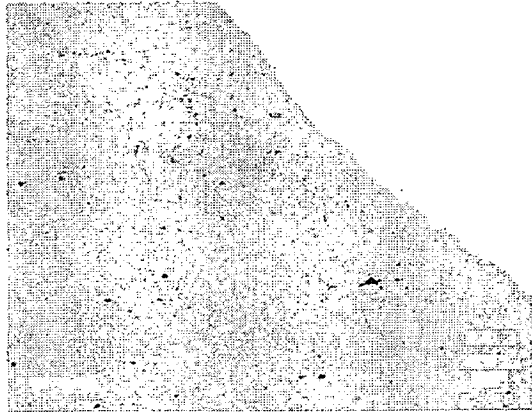
Fe-0.30P Alloy
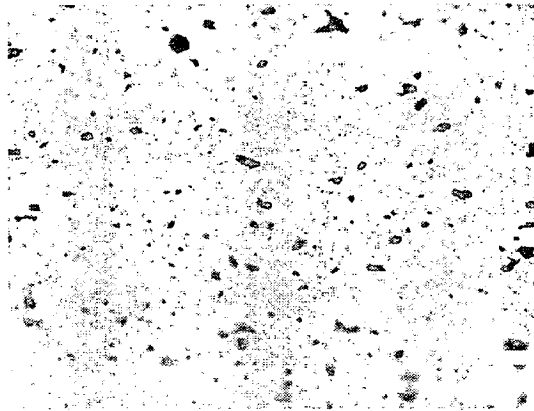
Fe-0.45P Alloy

Fe-0.65P Alloy |100μm| Fe-0.80P Alloy
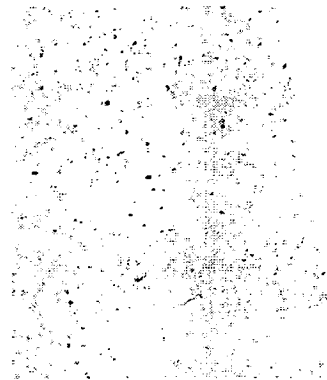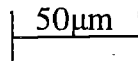
Figure.5.17. Rolled and annealed alloy (unetched) microstructures
section along rolling direction. Figures show the porosity distributed alo
boundaries as well as inside the grains for entire range of phosphorous
alloys. Some of them are elongated.

Fe-0.30P Alloy

Fe-0.35P Alloy

Fe-0.45P Alloy

Fe-0.65P Alloy

50μm
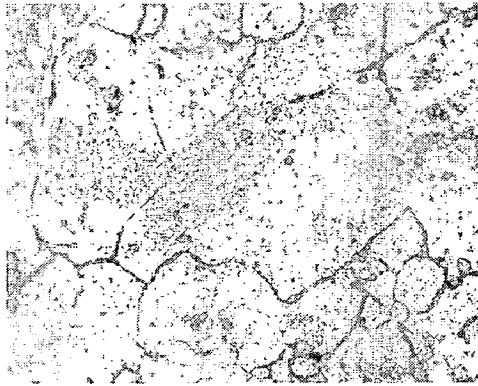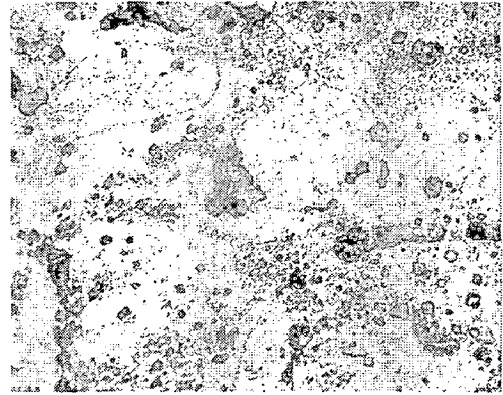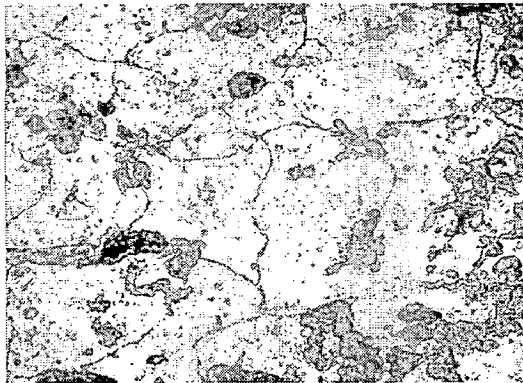
Fe-0.80P Alloy

Figure.5.18: Rolled and annealed alloys (etched) microstructure of wire section along rolling direction. Figures show the single phase with porosity distributed along grain boundaries as well as inside the grains for entire range of phosphorous in these alloys. Some of them are irregular size and shape and elongated.

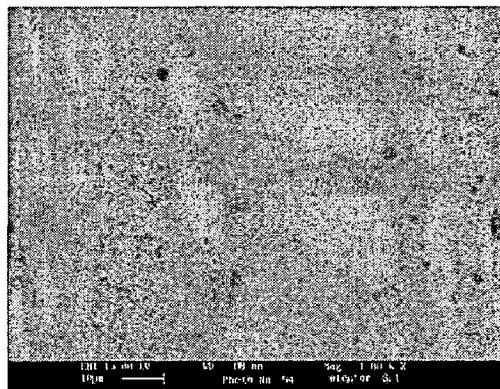Fe-0.30P Alloy

Fe-0.35P Alloy

Fe-0.45P Alloy

Fe-0.65P Alloy

$100\mu m$
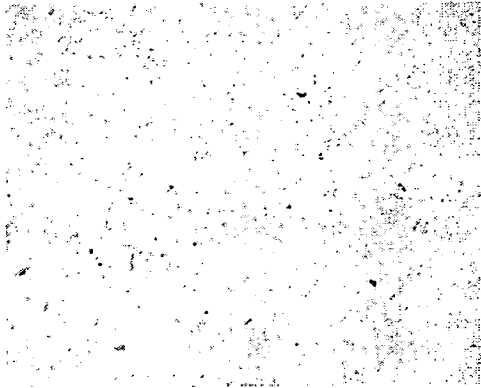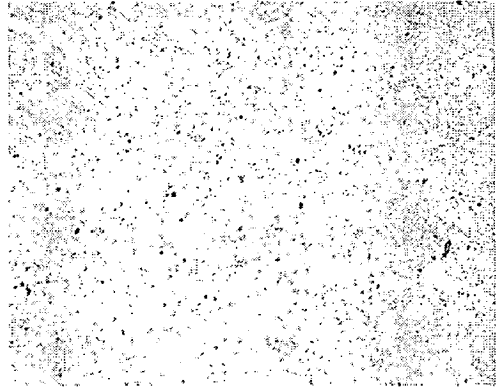
Fe-0.80P Alloy
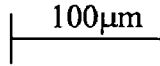
Figure.5.19. Microstructure (unetched) of cross-section of rolled and annealed wires. Figures show the porosity distributed in sample entire range of phosphorous in these alloys. Some of them are elongated.
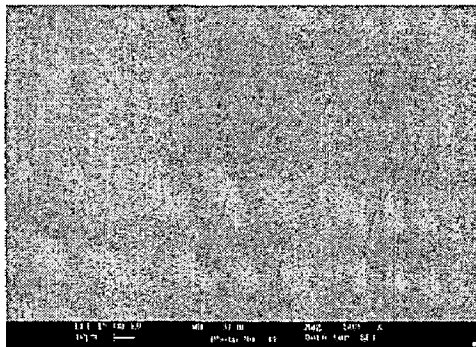
Fe-0.30P Alloy

Fe-0.35P Alloy

Fe-0.45P Alloy

Fe-0.65P   Alloy

| 50μm |
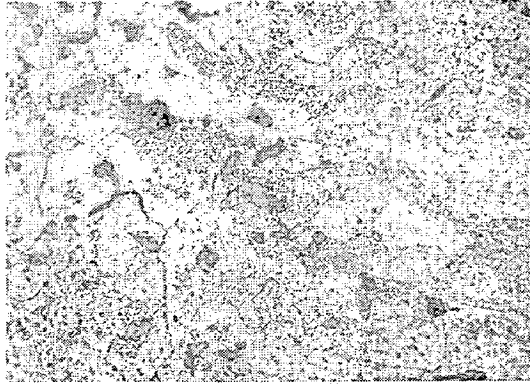
Fe-0.80P Alloy

Figure.5.20: Microstructure (etched) of cross-section of rolled and annealed wires. Figures show the single phase with porosity distributed along grain boundaries as well as inside the grains for entire range of phosphorous in these alloys. Some of them have bigger rounded shape.
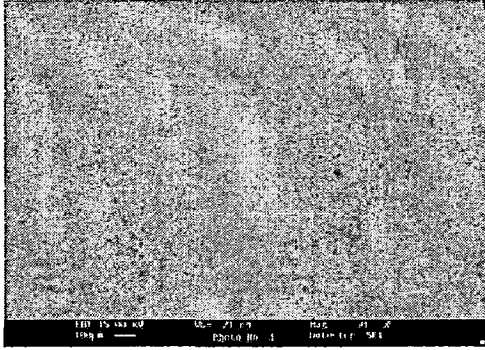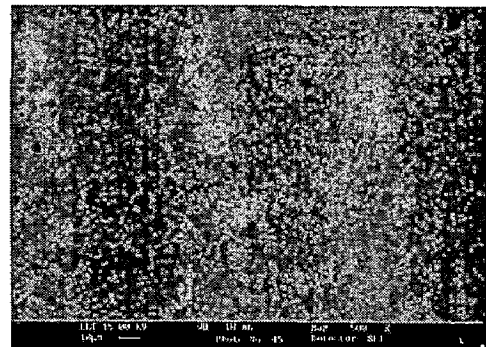
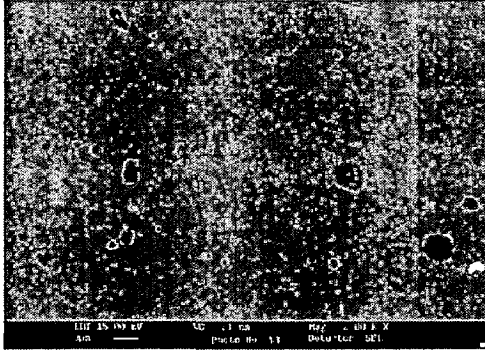Fe-0.30P Alloy
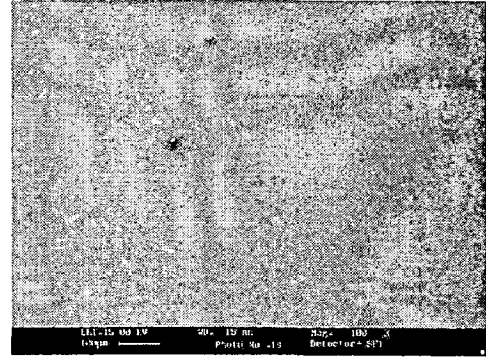
Fe-0.35P Alloy

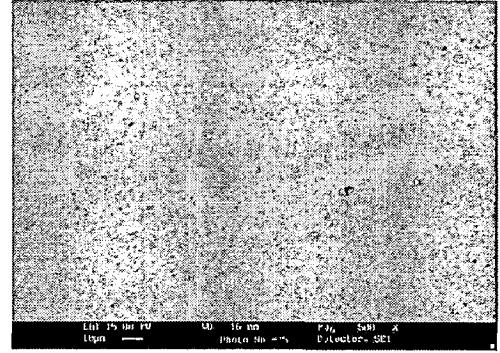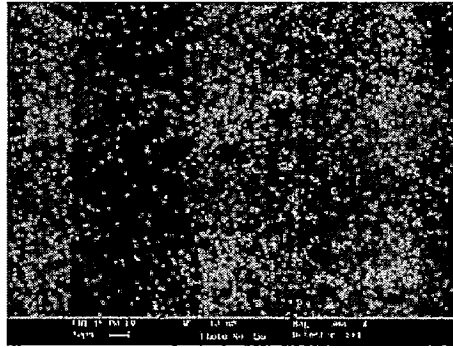Fe-0.45P Alloy

Fe-0.65P Alloy

Fe-0.80P Alloy

Figure.5.21: SEM examination by Secondary electron detector of rolled and annealed wire section along rolling direction. Magnification has been indicated on each figure Figures reveal the porosity and inclusions for entire range of phosphorous in these alloys.

Fe-0.30P Alloy

Fe-0.35P Alloy

Fe-0.45P Alloy

Fe-0.65P Alloy

Fe-0.80P Alloy

Figure.5.22: SEM examination by secondary electron detector of cross section of rolled and annealed wires. Magnification has been indicated on each figure. Figures reveal the porosity and inclusions for entire range of phosphorous in these alloys.
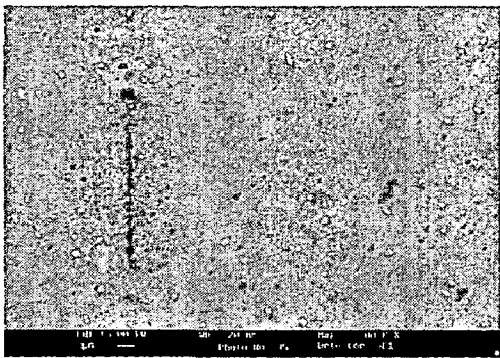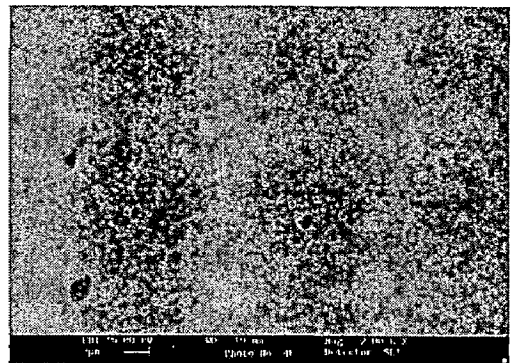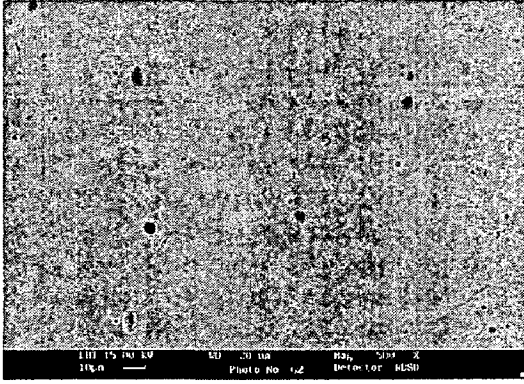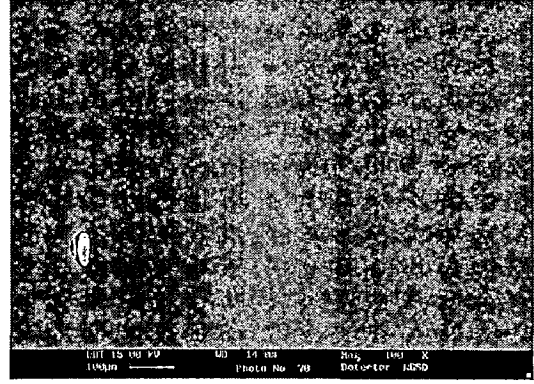
Fe-0.30P Alloy

Fe-0.35P Alloy

Fe-0.45P Alloy

Fe-0.65P Alloy

Fe-0.80P Alloy

Figure.5.23: SEM examination by back scattered electron of rolled and annealed wire section along rolling direction. Magnification has been indicated on each figure. Figures reveal the porosity and inclusions for entire range of phosphorous in these alloys.

104
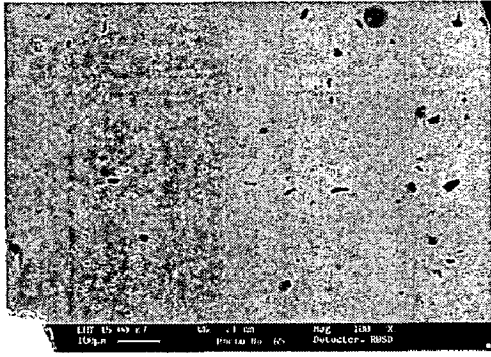
Fe-0.30P Alloy

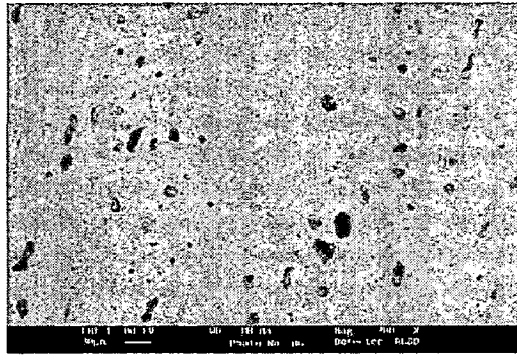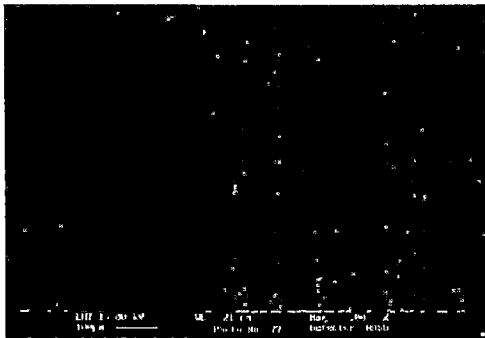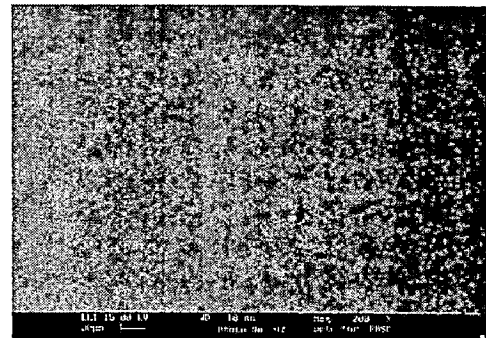Fe-0.35P Alloy

Fe-0.45P Alloy

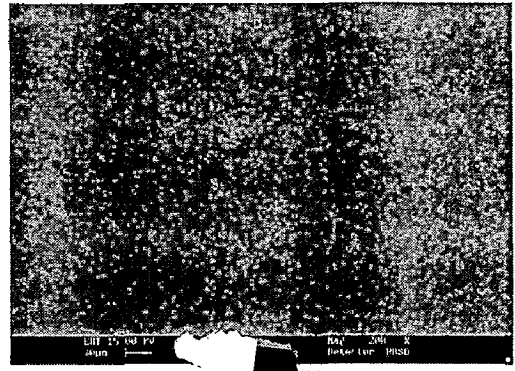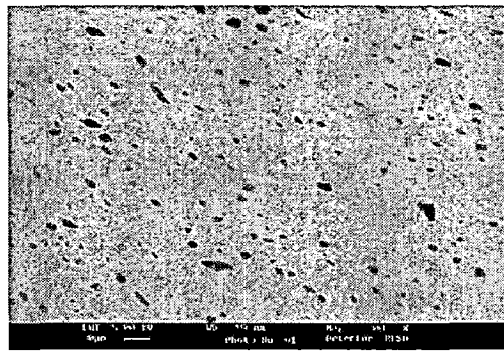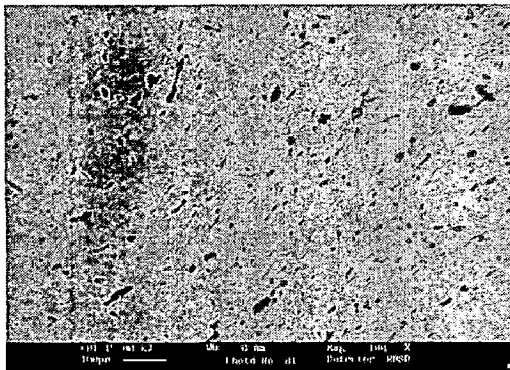
Fe-0.65P Alloy
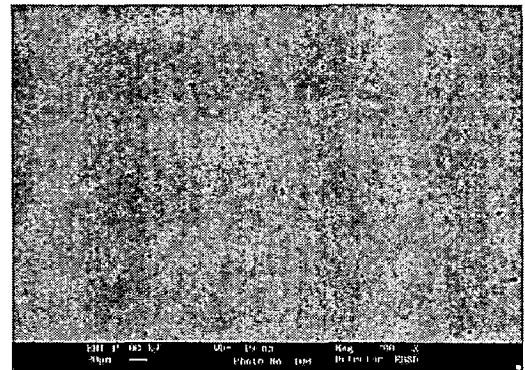
Fe-0.80P Alloy

Figure.5.24: SEM examination by back scattered electron of cross section of rolled and annealed wires. Magnification has been indicated on each figure. Figures reveal the porosity and inclusions for entire range of phosphorous in these alloys.

CHAPTER 6

CONCLUSIONS

- The present investigation based on hot powder forging route offers a good scope for enhancing density of parts made from Fe-P alloys. The density levels obtained are higher than compacting and sintering route processed products.

- It was observed that density in general increases with phosphorous alloying addition.

- It was also observed that hardness of forged and homogenized as well as rolled and annealed parts increases with phosphorous alloying addition.

- The saturation magnetization has been obtained from 10,716 gauss to 17,500 gauss with a maximum at 0.45%P at 60Oe applied field.

- Lowering of coercivity level due to phosphorous addition upto 0.65% is found to be comparatively better than that due to silicon addition upto 6.5%.It is a great achievement in the present investigation.

- Coercivity levels obtained in the present investigation range from 1.241 to 0.353 Oe whereas for equivalent sintered products it is from 2.0 to1.2 Oe.

- The saturation magnetizations of Fe-P alloys are found to be linearly dependent with the density of alloys. As density level increases, the saturation magnetization raises.

- The strips/sheets of length 60-70cm have been obtained without any appreciable failure for the alloys developed in the present investigation.

- Microstructures of the alloys consist of single phase with porosities distributed along grain boundaries as well as inside the grains for entire range of phosphorous in these alloys.

CHAPTER 7

SUGGESTION FOR FUTURE WORK

- Processing parameters need to be further refined to avoid the oxidation of sample during rolling at 1000^0C. Retaining the mild steel skin while rolling of forged P/M compact may significantly improve the product quality as well as lower the material and processing costs.

- Fe-P alloys containing 0.45wt%P and 0.65wt%P showed higher saturation magnetization. In order to further enhance saturation magnetization, alloy system such as Fe-0.45%P-4%Co and Fe-0.65%P-4%Co also need to be developed employing processing technique of the present investigation.

- Magnetic properties of the Fe-P alloys have been taken under DC and AC condition at constant frequency.However, it also needs to be performed under same condition of same alloy at varying frequency.

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APPENDIX

Publications & Conferences

1. S.K.Chaurasia, Ujjwal Prakash and P.S.Mishra, "Development of P/M Fe-P soft magnetic materials," National symposium, MR-09, IIT Bombay, (7-9 May, 2009), (accepted).

2. S.K.Chaurasia, Ujjwal Prakash, P.S.Mishra and K.Chandra, "Comparisons of Sintering Technology with powder forging for Fe-P based alloys," (Under progress).

3. S.K.Chaurasia,Ujjwal Prakash,P.S.Mishra and K.Chandra," Role of Phosphorous in enhancing soft magnetic properties of iron,"(Under Progress)

Units and Conversions

- ## Field

    1 Oersted   = 0.795 Amp/cm = 79.6 Amp/meter
    1 Amp/meter = 0.01 Amp/cm   = 0.0125 Oersted
    1 Amp/cm   = 100 Amp/meter =1.256 Oersted
    1 kAmp/meter = 10 Amp/cm   =12.56 Oersted

- ## Flux

    1Maxwell   = $(10)^{-8}$ Webers = 10^{-8} Volt.sec
    1 Volt.sec = 1 Weber   = 10^{8} Maxwell
    1 Weber   =1 Volt.sec   = 10^{8} Maxwell

- ## Induction or Flux density

    1Gauss   = 10^{-4} Tesla   = 10^{-6} Volt.sec/cm^2
    1 Volt.sec/cm^2 = 10^{4} Tesla   = 10^{8} Gauss
    1mili Tesla   =10^{-7} Volt.sec/cm^2 = 10 Gauss
    1 Tesla   =10^{-4} Volt.sec/cm^2 =10^{4} Gauss

Magnetism

Cgs units	mks units
$B = H + 4\pi M$	$B = \mu_0 H + M$
B in guass	B in weber/meter^2 (tesla)
H in Oe	H in Amperes/meter
M in emu/cm^3	M in weber/meter^2
μ (vacuum) = 1	μ_0(vacuum) =$4\pi \times 10^{-7}$weber/ampere meter

Table A

Demagnetizing Factors for Ellipsoids of revolution for prolate spheroids, c is the polar axis.

C/a	$N_c/4$	C/a	$N_c/4$	C/a	$N_c/4$
1.0	0.333 333	4.0	0.075 407	20	0.006 749
1.1	308 285	4.1	72 990	21	6 230
1.2	286 128	4.2	70 693	22	5 771
1.3	266 420	4.3	68 509	23	5 363
1.4	248 803	4.4	66 431	24	4 998
1.5	0.232 981	4.5	0.064 450	25	0.004 671
1.6	218 713	4.6	62 562	30	3 444
1.7	205 794	4.7	60 760	35	2 655
1.8	194 056	4.8	59 039	40	2 116
1.9	183 353	4.9	57 394	45	1 730
2.0	0.173 564	5.0	0.050 821	50	0.001 443
2.1	164 585	5.5	48 890	60	1 053
2.2	156 326	6.0	43 230	70	0 805
2.3	148 710	6.5	38 541	80	0 637
2.4	141 669	7.0	34 609	90	0 518
2.5	0.135 146	7.5	0.031 275	100	0.000 430
2.6	129 090	8.0	28 421	110	363
2.7	123 455	8.5	25 958	120	311
2.8	118 203	9.0	23 816	130	270
2.9	113 298	9.5	21 939	140	236
3.0	0.108 709	10	0.020 286	150	0.000 209
3.1	104 410	11	17 515	200	125
3.2	100 376	12	15 297	250	083
3.3	096 584	13	13 490	300	060
3.4	093 015	14	11 997	350	045
3.5	0.089 651	15	0.010 749	400	0.000 036
3.6	86 477	16	09 692	500	24
3.7	83 478	17	08 790	600	17
3.8	80 641	18	08 013	700	13
3.9	77 954	19	07 339	800	10

From 'Introduction to Magnetic Materials' by B.D. Cullity (Addison - Wesley Pub. Co.)1972