

## **Effect of Chromium on Magnetic Characteristics of Powder Processed Fe-0.35wt%P Alloy**

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### **ABSTRACT**

*The present investigation deals with hot powder preform forging technique for the development of high density iron-phosphorus based alloys. These alloys are known for hot-shortness and are therefore not considered suitable for high temperature working. To remove this problem proper soaking at high temperature to eliminate iron- phosphide eutectic and bring entire phosphorus into solution in iron was aimed. Attempting hot forging thereafter completely eliminates hot as well as cold shortness and thereby helps to form these alloys into very thin sheets. It has also been possible to eliminate the use of costly hydrogen atmosphere during sintering by use of addition of carbon as a reducing agent to form CO gas within the compact by reacting with oxygen of iron powder particles. The glassy ceramic coating applied over the compact serves as a protective coating to avoid atmospheric oxygen attack over the compact held at high temperature. Combined application of carbon and glassy ceramic coating has lead to economy in P/M processing for soft magnetic applications. Fe- 0.07C- 0.30- 0.35wt% P- 0.35wt% Cr alloy so formed yielded coercivity as low as 0.42 Oe, resistivity as high as 21.8  $\mu\Omega\text{cm}$  and total loss as low as 0.170 W/Kg. Such a combination of properties may suit their application in magnetic relays and transformer cores.*

**Key Words:** *Glassy Ceramic Coating; Preform Forging; Coercivity*

### **1. INTRODUCTION**

Iron-phosphorous alloy system for the production of magnetic materials is not known in wrought processing route in spite of the fact that phosphorous as an alloying element has all favorable characteristics to enhance magnetic properties of iron [1]. 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. Powder metallurgical processing, due to its predominantly solid state processing approach, has been very successful in exploiting potential of Fe-P alloy system for magnetic applications [2]. A number of products such as magnetic cores of contactors, relays, magnetic brakes and electricity meters have been produced out of this system [3].

The sintered parts mostly made of pure iron are now well established in the market for soft magnetic applications. These components at high sintered density possess high saturation magnetization but they exhibit low resistivity and high magnetic losses [4]. Increasing resistivity reduces the eddy current losses [5] and alloying (P and Cr) primarily increases the resistivity [6]. Phosphorus activates sintering process in Fe-P alloys by the formation of low-melting eutectic phase with iron [7]. It helps in carrying out alloy constituents into iron matrix which are otherwise difficult to diffuse. It also increases induction and permeability [8] and decreases coercivity of iron. Chromium increases the electrical resistivity of Fe-P based alloys and being a ferrite stabilizer, it also improves magnetic properties of Fe-based alloys [6]. Chromium also improves formability of the iron-based alloys significantly [9].

Conventional powder metallurgy processes enable compaction to be 93% of the theoretical density [10]. Double pressing achieves higher density but at increased cost. Existence of prior particle boundaries renders hot isostatic pressing unsuitable for magnetic applications. In view of this, in the present investigation, densification is carried out by cost effective hot powder preform forging technique. The process renders highest possible densification without resorting to hydrogen as sintering atmosphere. It is essentially the process where shaping and consolidation are deformation based. This causes redistribution of segregants if at all remained at the particle surfaces (deformation can displace these from grain boundary and disintegrate them to fine particles which easily dissolve inside the ferritic grains). Such a processing route provides superior magnetic properties in comparison to existing sintering approach.

## **2. EXPERIMENTAL PROCEDURE**

The present investigation utilizes atomized iron powder of M/S Hoganas India Ltd., Grade ASC 100.29. It has carbon ~ 0.01% and hydrogen loss value as 0.1%. Its particle size is -100 mesh. Low-carbon ferro-chrome powder (C < 0.01 wt %) (size -200 mesh) was prepared by grinding the ferro-chrome lumps. Iron-phosphide powder (C- 0.01 wt %) (size -100 mesh) was prepared by reacting iron powder with ortho-phosphoric acid and a subsequent thermal treatment (800<sup>0</sup>C/2h/H<sub>2</sub>) to yield Fe<sub>3</sub>P coating over iron powder.

The powders are suitably mixed with 0.3wt% of carbon (in the form of graphite; size -100 mesh) to yield the following chemistry:

- (1) Fe-0.3 wt% C-0.35 wt% P
- (2) Fe-0.3 wt% C-0.35 wt% P-0.35 wt% Cr

The powder mix was filled in a rectangular die and the green compacts (preform) with 7mm thickness and 25mm x 50mm size were formed. The oxidation resistant glassy coating [11] was applied on the surface of preforms like a paint with a brush to serve as protective layer resisting oxidation at high temperatures and protecting reducing gaseous atmosphere of CO produced inside the compact at high temperature by reaction of C with O<sub>2</sub> (of iron powder). Coated preforms were baked at 120<sup>0</sup>C for two hours. These preforms were then transferred to a furnace held at a temperature 1050<sup>0</sup>C and soaked there for one hour. The hot preforms were immediately transferred to a forging die fitted in the press and were forged. Then the slabs were hot rolled at temperature 900<sup>0</sup>C to form a sheet of 0.1mm thickness. Toroids were stamped from the sheet using a die/punch arrangement. Glassy coating was applied on the toroids and these were again annealed at 850<sup>0</sup>C for three hours to relieve residual stresses. The dimensions of toroids were 1mm thickness, 50mm outer diameter, 40mm inner diameter with 74 primary and 6 secondary windings.

The samples prepared this way were characterized in terms of their density, microstructure, electrical resistivity and magnetic properties. The content of carbon and oxygen was analyzed by spectroscopic analyzer. Density was determined by Archimedes' principle. Microstructure (etched with 2% Nital) of the rolled and annealed sheets was analyzed by using image analyzer to estimate the grain size and volume percentage of porosity in the alloys. Scanning electron microscopy and X- Ray mapping confirmed the presence of ferrite phase and showed uniform distribution of phosphorus and chromium in iron matrix. Electrical resistivity was measured by four probe method. Magnetic properties were measured under d.c. mode.

### 3. RESULTS AND DISCUSSION

Incorporating carbon to the alloys has helped in number of ways, firstly as solid lubricant during cold compacting; secondly as solid state reducing agent to take care of oxygen situated at iron powder particles surfaces during high temperature processing. Thirdly pushing phosphorus into ferrite grain as solute and thereby discourages it to precipitate as phosphide along ferrite grain boundaries [12]. The complete chemistry of alloys developed (spectroscopic analysis) is given in Table 1 (as first column) along with the measured values of density (forged, rolled and annealed sheets), grain size and porosity percentage present in the sheets. It was observed that densification has improved with the present processing technique.

The microstructure shows residual alignment of porosity due to rolling (Fig. 1(a), (b)). Pores are elongated due to unidirectional compressive force [2]. Pore rounding and coagulation of smaller

pores into bigger pores are observed in the microstructures. It may be due to the presence of phosphorous content in the alloy [13].

Table: 1 Density, porosity and grain size of the alloys.

<b>Final Chemistry</b>	<b>Forged Density (g/cc)</b>	<b>Rolled and Annealed Density (g/cc)</b>	<b>Percentage Porosity</b>	<b>Grain Size (<math>\mu\text{m}</math>)</b>
<b>Fe-0.07C-0.30O-0.35P</b>	<b>7.68</b>	<b>7.76</b>	<b>1.42</b>	<b>88</b>
<b>Fe-0.07C-0.30O-0.35P-0.35Cr</b>	<b>7.62</b>	<b>7.73</b>	<b>1.53</b>	<b>94</b>

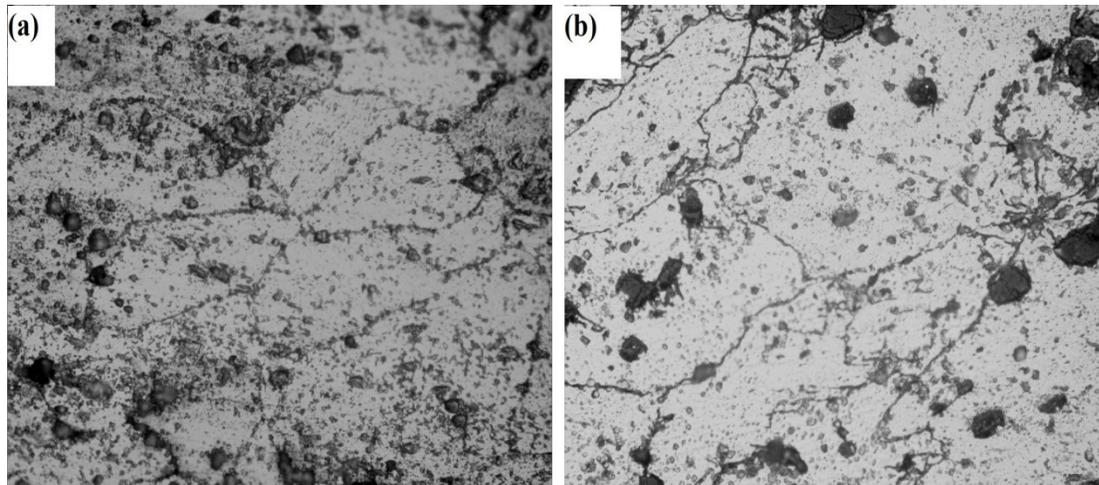


Fig. 1: Microstructures of rolled and annealed alloys etched with 2% Nital at 200X. (a) Fe-0.07C-0.30O-0.35P alloy and (b) Fe-0.07C-0.30O-0.35P-0.35Cr alloy

Phosphorus and chromium are both ferrite stabilizers and are added to iron below its solubility limit; therefore only alpha phase is present (Fig. 2 (a), Fig. 3 (a)). The alloys developed in the present investigation are free of any segregation of the alloying elements along the grain boundaries. They get distributed uniformly in the entire structure (Fig. 2 (b) - (c), Fig. 3 (b) - (d)).

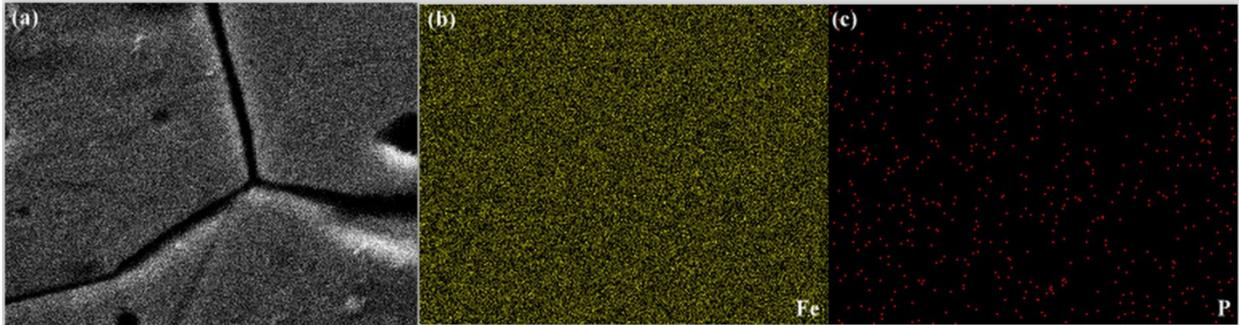


Fig. 2: (a) Compositional Image (Secondary Image), X- Ray Mapping showing uniform distribution of alloying elements in Fe-0.07C-0.3O-0.35P alloy at 3000X. (b) Fe and (c) P

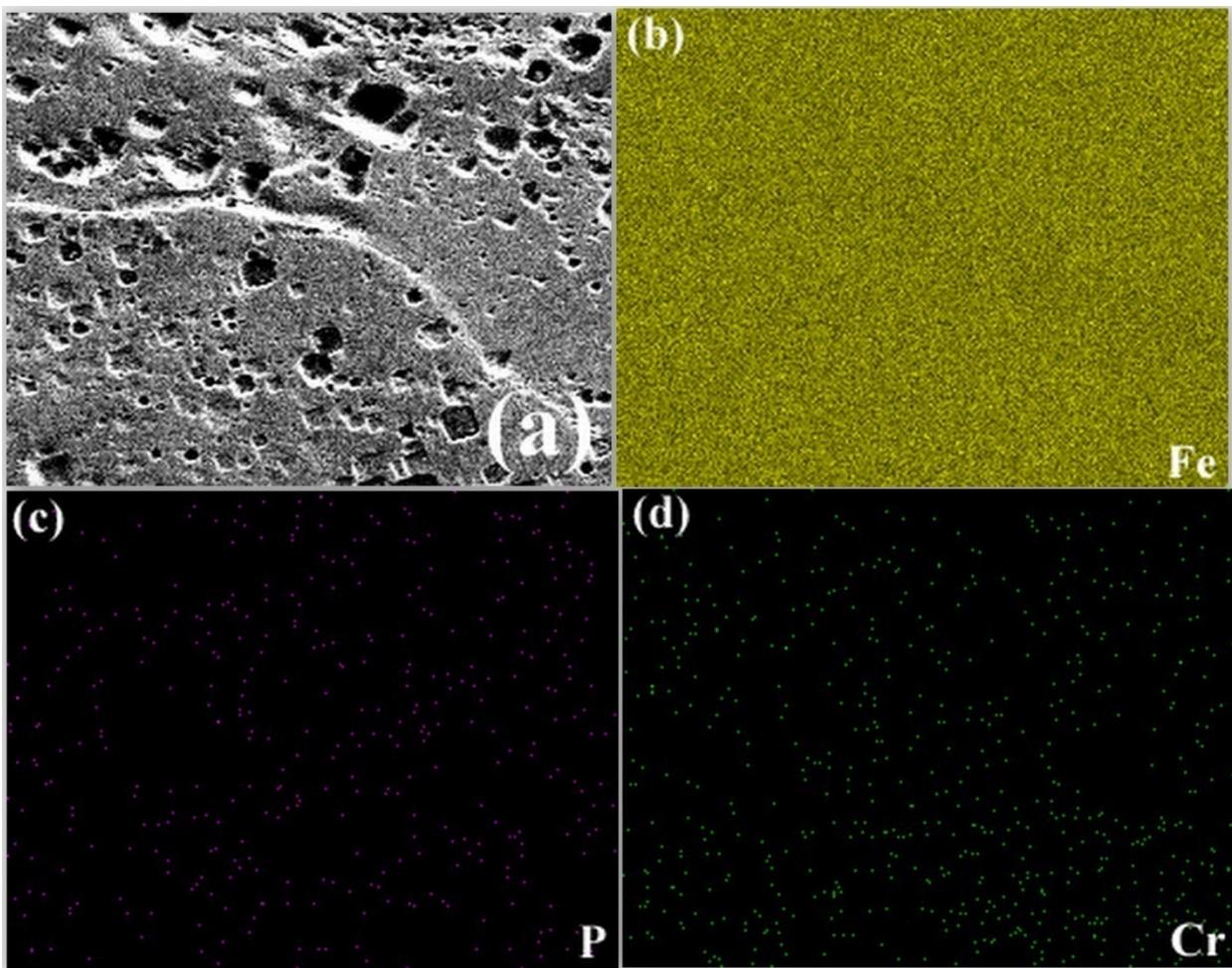


Fig. 3: (a) Compositional Image (Secondary Image), X- Ray Mapping showing uniform distribution of alloying elements in Fe-0.07C-0.3O-0.35P-0.35Cr alloy at 3000X. (b) Fe, (c) P and (d) Cr.

The resistance of iron increases greatly due to alloying additions like P and Cr [6]. Table 2 represents the electrical resistivity and d.c. magnetic properties of the alloys and compares with the commercially available soft magnetic material.

Table: 2 Comparison of electrical and magnetic properties of the alloys developed in present investigation with commercially available soft magnetic material.

<b>Composition</b>	<b>Electrical Resistivity (<math>\mu\Omega\text{cm}</math>)</b>	<b>Coercivity (Oe)</b>	<b>Permeability</b>	<b>Total Loss (W/Kg)</b>	<b>Saturation Magnetization (G) at 100 Oe</b>
<b>Fe-0.07C-0.30O-0.35P</b>	18.2	0.44	10385	0.175	12873
<b>Fe-0.07C-0.30O-0.35P-0.35Cr</b>	21.8	0.42	10941	0.170	12247
<b>CRGO</b>	48.0	0.50	30559	0.95	19469

Coercivity of Fe-based alloys falls as we add alloying elements such as P and Cr to Fe. The higher the alloying content, lower is the coercivity value [8]. The superimposed hysteresis loops of Fe-0.07C-0.30O-0.35P alloy and Fe-0.07C-0.30O-0.35P-0.35Cr alloy are shown in Fig. 4.

The permeability increases greatly due to alloying additions like P and Cr [9]. Higher value of permeability observed in Fe-0.07C-0.30O-0.35P-0.35Cr was due to large grain size (94  $\mu\text{m}$ ) and low porosity percentage (1.53). Total magnetic loss is the sum of eddy current loss and hysteresis loss [5]. Combined addition of P and Cr has decreased the total magnetic loss of the alloys developed. This may be due to the fact that the alloy has simultaneously high resistivity (21.8  $\mu\Omega\text{cm}$ ) and low coercivity values (0.42 Oe). Saturation magnetization of iron increases when phosphorous is added up to 0.8wt % and decreases thereafter [13]. In the present investigation addition of 0.35wt% of Cr, lowers saturation magnetization at a rate corresponding to simple dilution [9].

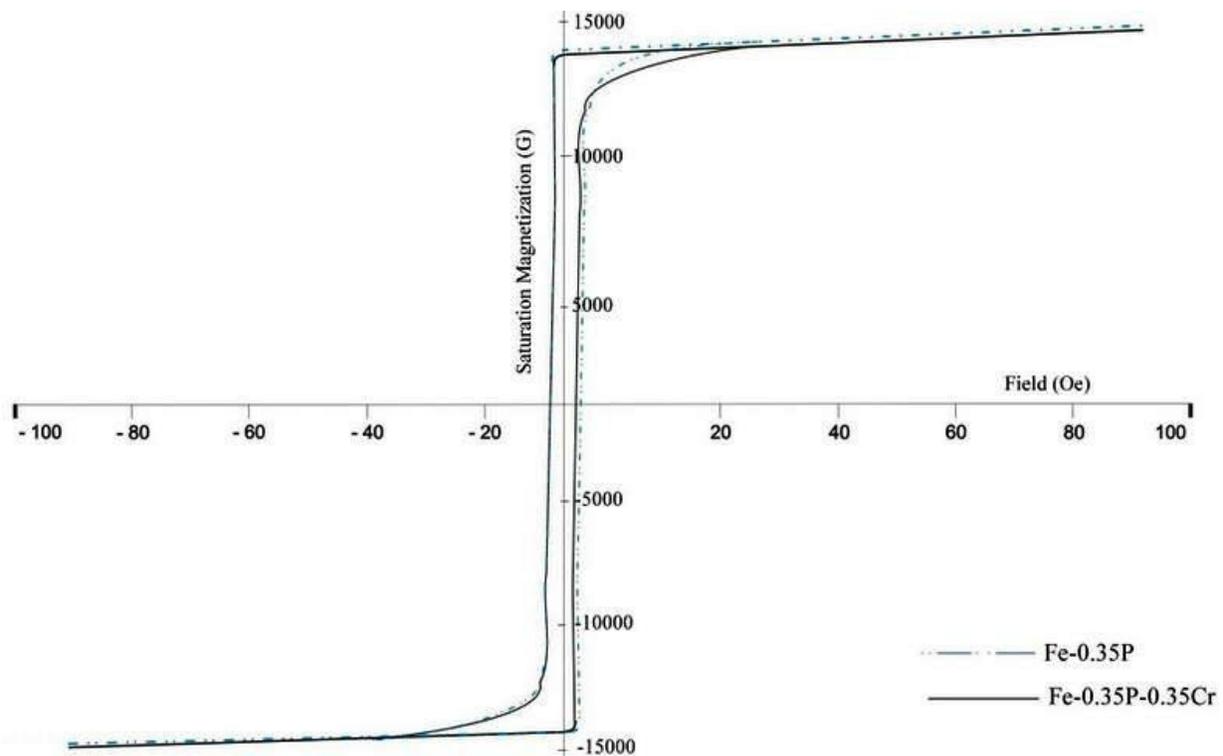


Fig. 4: Superimposed hysteresis loops of Fe-0.07C-0.3O-0.35P alloy and Fe-0.07C-0.3O-0.35P-0.35Cr alloy

Such a combination of properties is achieved by the use of hot powder preform forging technique employed in the present investigation.

#### 4. CONCLUSIONS

- 1) The forming technique does not require any binder. Thus the system remains uncontaminated.
- 2) The use of ceramic protective coating eliminates the need of hydrogen protective atmosphere during heating.
- 3) Combined application of glassy ceramic coating and use of graphite as a reducing agent has lead to economy in P/M processing.
- 4) The technology developed in the present investigation showed very low coercivity and total loss values.

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