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Effect of Si addition on AC and DC magnetic properties of (Fe-P)-Si alloy

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We report a new (Fe-P)-Si based alloy with relatively high induction (1.8-1.9 T), low coercivity (< 80 A/m), high resistivity ($\sim 38 \mu\Omega$ cm) and low core loss (217 W/kg @ 1 T/1 kHz) comparable to the commercially available M530-50 A5 Si-steel. The attractive magnetic and electrical properties are attributed to i) the two phase microstructure of fine nano precipitates of Fe_3P dispersed in α -Fe matrix achieved by a two-step heat-treatment process and ii) Si addition enhancing the resistivity of the α -Fe matrix phase. As the alloy processing is by conventional wrought metallurgy method, it has the potential for large scale production. © 2016 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution 3.0 Unported License. [<http://dx.doi.org/10.1063/1.4944074>]

I. INTRODUCTION

Soft magnetic materials are extensively used in various electro-technical applications like motors, alternators, transformers, actuators, choke coils, and hence they are essential in many areas like automotive, defence, telecommunications etc. Among various soft magnetic materials like Fe-Ni or Fe-Co based alloys, soft magnetic ferrites, sintered materials and powder composites, electrical steels are the most widely used ones. In particular non-grain oriented electrical steel accounts for 80% of total production of all soft magnetic materials. Currently Si-steel and low carbon steel are widely used in the manufacture of alternators and motors; the former for better performance and the latter for cost effectiveness. The production of electrical steel globally was about 12.6 million tonnes and hence a slight enhancement in performance or cost effectiveness will have a huge impact in the overall market. Hence there is a constant drive to search for soft magnetic materials with enhanced performance at low cost.

Research on improving the efficiency of soft magnetic materials can be broadly classified into two categories viz. overcoming the issues associated with Si-steel and to develop alternate materials. Alloying,^{1,2} domain refinement,³ texturing,⁴ grain size refinement^{5,6} and coatings⁷ are some of the techniques employed to address the issues related to Si-steel. Alternately amorphous and nano-crystalline soft magnetic materials are being explored to replace Si-steel. Amorphous materials are devoid of magnetocrystalline anisotropy and hence exhibit good soft magnetic properties. One of the earliest amorphous soft magnetic material METGLAS (Fe-Ni-P-B) exhibited core loss performance six times better than conventional materials.⁸ The nanocrystalline soft magnetic materials with zero magnetostriction achieved by a combination of positive and negative magnetostrictive phases are also considered as alternatives for Si-steel. FINEMET (Fe-Si-Zr-B-Cu)⁹ reported by Yoshizawa was one of the first identified materials in this class. Thereafter various attempts have been made to explore this class of materials for applications¹⁰⁻¹² but issues like scalability,

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thermal stability, component fabrication have made these materials not suitable for applications in automotive industry.

Fe-P based alloys are considered as one of the promising materials which could replace Si-steel on account of two factors *viz.* i) the cost of Fe-P based alloys are expected to be about 20% lower than Fe-Si alloys¹³ and ii) phosphorous addition enhances the soft magnetic properties of pure Fe by lowering coercivity and, increasing permeability and resistivity.^{14,15} In wrought metallurgical process, due to its low solubility in Fe, phosphorous segregates at the grain boundaries resulting in brittleness. Extensive work on Fe-P has been carried out in powder metallurgical process but restricting the P content to a maximum of 0.8 wt.%. Hoeganaes Corporation commercially produces Fe-P powder (≤ 0.8 wt.%) for soft magnetic applications.¹³ Major challenge in PM processing of Fe-P powders is adoption of sophisticated compaction techniques to overcome the shrinkage related problems due to liquid phase sintering.

Gopalan *et al.*¹⁶ have reported Fe-0.35 wt.%P alloy processed by melt spinning technique and reported a high induction of 1.9 T and a coercivity of 40 A/m. The high induction was due to the exchange coupling of the two ferromagnetic phases of α -Fe and tetragonal Fe₃P precipitates. The low coercivity was attributed to the domain walls of the α -Fe matrix phase sweeping over the fine nano precipitates as the domain wall width of the α -Fe matrix was 40-50 nm much higher than the precipitates. Chandrasekhar *et al.*¹⁷ have reported Fe-0.4 wt.%P alloy by wrought alloy process with attractive DC and limited AC magnetic properties. In this paper, we report the development of low Si containing Fe-P based alloy by an industrially viable wrought alloy process. The DC and AC magnetic properties (up to 1 kHz) of the alloy was found to be equivalent to commercially available M530-50 A5 Si-steel.¹⁸

II. EXPERIMENTAL PROCEDURE

Two Fe-P based alloys (~ 10 kg capacity) with Si content of 0.25 wt.% (L-Si) and 0.85 wt.% (H-Si) were prepared by melting pure Fe with Fe-P and Fe-Si master alloys in vacuum induction melting furnace. The cast ingot (65 mm diameter and 400 mm length) was hot forged and rolled to a final thickness of ~ 0.5 mm (Fig. 1(a)-1(c)). The sheet samples were subjected to the following two-step heat treatment procedure: a) Solution treatment at 900°C for 1 hr and b) annealing treatment at 600°C for 30 min. Phase analysis was carried out using x-ray diffractometer (Bruker AXS D8) and the microstructure of the samples was examined using Transmission Electron Microscope (TEM) (FEI Tecnai G2). The samples for TEM were prepared using a precision ion polishing system with Ar source. DC magnetic properties were measured using vibrating sample magnetometer (VSM) (MicroSense, USA Model No. EV 9) and coercimeter (Laboratorio Elettrofisico, Italy Model No. C03). The AC magnetic properties up to 1 kHz were evaluated using BH loop tracer (Laboratorio Elettrofisico, Italy Model AMH-20k-HS) using toroid samples made from the rolled sheets and commercial M530 – 50 A5 sheets. The resistivity measurements have been carried out

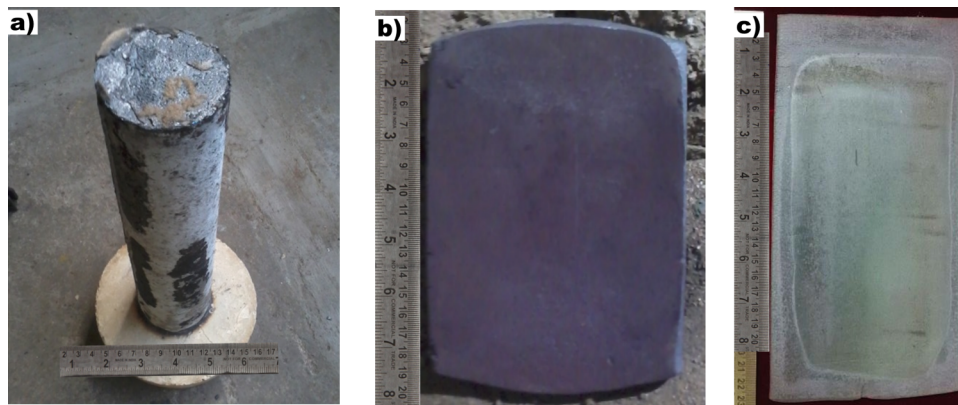


FIG. 1. Photographs at various stages of the alloy processing a) as cast ingot b) forged plate and c) hot rolled sheets.

using a four probe technique (Keithley 2182A nanovoltmeter and Keithley 6221 current source; USA). About 10 measurements were carried out at various currents and resistivity was obtained by fitting this data and rounded off to the nearest whole number.

III. RESULTS AND DISCUSSION

The initial magnetization curve and the demagnetization behaviour measured for the annealed samples measured using VSM and coercimeter are shown in Fig. 2(a) and 2(b). The L-Si and H-Si samples exhibited a saturation magnetization value of 1.9 T and 1.85 T respectively. The base alloy Fe-0.4 wt. % P magnetization was reported to be 1.9 T.¹⁷ The magnetization of L-Si was similar but for H-Si it was 1.85 (a reduction of 2.6%) which is more than expected, suggesting the decrease may not be a simple dilution effect but may be due to the reduction in the average moment of iron due to Si addition.

Coercivity was measured using coercimeter equipment. The sample is magnetized by an axial magnetic field using a solenoid. The transverse component of the flux lines coming out of the magnetized sample is measured using a hall probe placed close to the sample. The coercivity is measured as that axial field required to demagnetize the sample so that no flux lines emanate out of the sample. The measurement is repeated by reversing the current of the magnetizing solenoid and the average value is taken to eliminate any offset in the applied field. The entire setup is placed in a mu-metal shield to eliminate the earth magnetic field. From Fig. 2(b) it can be seen that both alloys exhibited a coercivity of about 70 A/m. The DC magnetic properties of these alloys are similar to those reported by Gopalan *et al.* for the Fe-0.35wt.%P¹⁴ and Chandrasekhar *et al.* for the Fe-0.4wt%P wrought alloy.¹⁵

AC magnetic properties were measured up to 1 kHz restricting the induction up to 1 T. Figure 3 shows the hysteresis loop of the two alloys measured at 50 Hz and 1 kHz. The figure also includes the hysteresis loop of the commercial M530-50 A5 Si-steel for comparison. It can be seen from Fig 3 that L-Si exhibits a wider loop compared to H-Si and M530-50 A5 Si-steel both at 50 and 1 kHz. Figure 4 shows the core loss of the three samples measured up to a frequency of 1 kHz. The core loss at 50 Hz for L-Si and H-Si alloy was 2.7 W/kg and 2.2 W/kg respectively and both values are comparable to 2 W/kg of M530-50 A5 Si-steel. However at 1 kHz the core loss of L-Si alloy was 355 W/kg while H-Si was 217 W/kg which is comparable to that of M530-50 A5 Si-steel measuring 205 W/kg. At low frequencies (i.e. <50 Hz) the core loss was similar for all the three alloys (inset

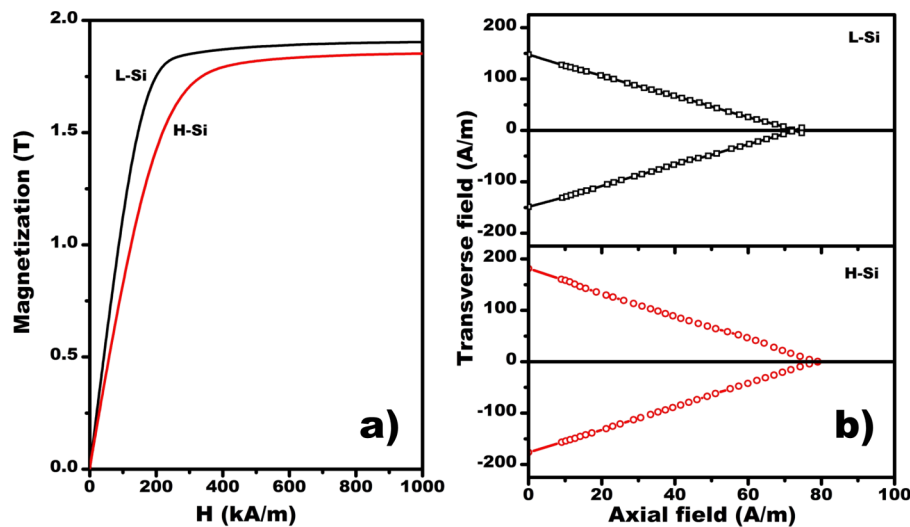


FIG. 2. The DC magnetic properties of the two alloys a) Initial magnetization curve measured using VSM showing a relatively high magnetic induction and b) coercivity measured using a coercimeter.

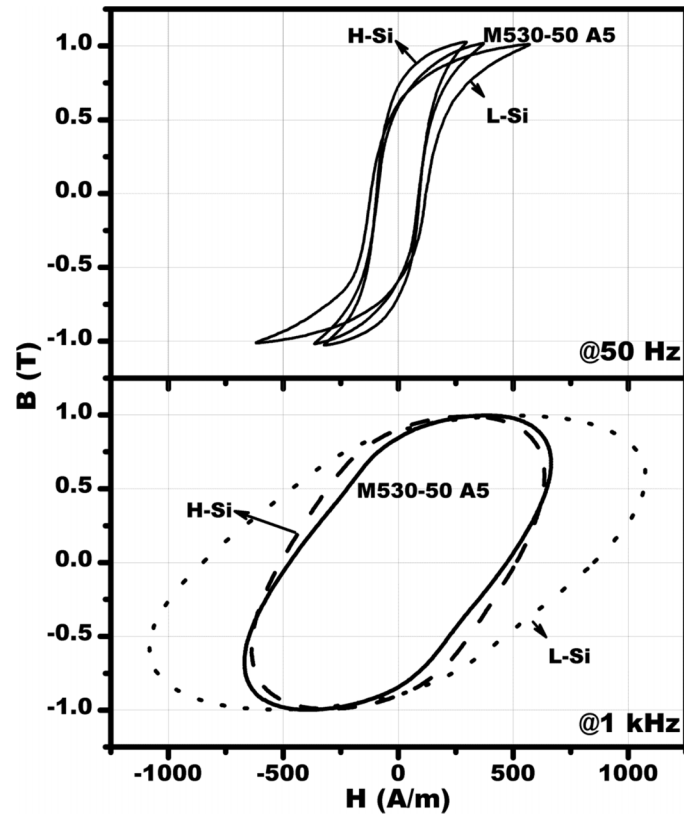


FIG. 3. Hysteresis loop measured at $B_{max} = 1$ T for the three alloys at 50 Hz and 1 kHz showing similar behaviour at low frequency and the hysteresis of the L-Si alloy deviating from the other two alloys at 1 kHz.

Fig. 4) but with increasing frequency the core loss of L-Si alloy was found to increase more rapidly than H-Si and M530-50 A5 Si-Steel (Fig. 4).

In soft magnetic materials, the total loss is a combination of the hysteresis and eddy current loss. It is well known that, the hysteresis losses dominate at low frequencies (<50 Hz), and at higher

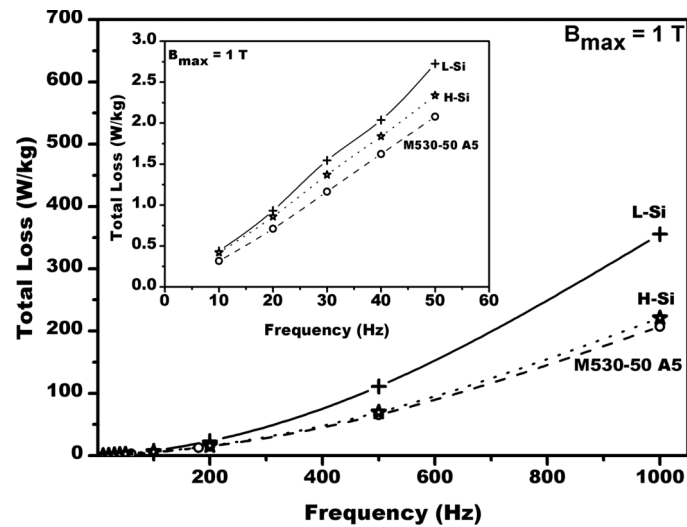


FIG. 4. Core loss versus frequency behaviour for the three alloys showing the H-Si alloy matching the performance of M530-50 A5 Si-steel. The inset shows the nearly similar core loss of the three alloys at lower frequencies.

frequencies the eddy current losses plays a key role. Hence in order to minimize total loss it is important that soft magnetic materials exhibit low coercivity and high resistivity.

As discussed earlier the DC magnetic properties of all the three alloys are similar which explains the comparable core loss values at low frequencies. It has been found that H-Si and M530-50 A5 Si-steel have almost equal resistivity (38 and 37 $\mu\Omega$ cm) whereas L-Si exhibits a relatively low value of about 17 $\mu\Omega$ cm. The observed difference in the resistivity values is well reflected in the variation of core loss at high frequencies.

The higher resistivity value in H-Si compared to L-Si is due to the higher Si content.¹⁷ However it is interesting to note that a resistivity of ~ 38 $\mu\Omega$ cm has been achieved with a Si content of 0.85 wt. % in Fe-P alloy whereas a similar resistivity is possible only with 1.6 wt. % Si in pure Iron. The lower Si content in our alloy is expected to have consequent metallurgical advantages during large scale production.

To understand further the magnetic and electrical properties, TEM studies have been carried out on the L-Si and H-Si samples and the bright field images are shown in Fig. 5(a) and 5(b). The bright field images show the presence of nano-precipitates (<10 nm) for both the alloys. The nano precipitates have been identified to be Fe_3P from the extra diffraction spots in the selected area diffraction pattern (inset in Fig. 5(a) and 5(b)). This observation is consistent with the earlier results reported in Fe-P alloys by Gopalan *et al.* and Chandrasekar *et al.* In the bright field images the striking feature is the difference in the number density of the nano precipitates between the two alloys. The H-Si alloy has more volume fraction of nano precipitates of Fe_3P than that of L-Si. The only difference in composition between the two alloys is the presence of additional Si. From the increased number density of the Fe_3P precipitates in H-Si alloy it appears that the Si contributes indirectly by increasing P:Fe ratio favouring the enhanced precipitation of Fe_3P . The enhanced nano-precipitation probably explains the observed resistivity values as they tend to act as scattering centres for the flow of electrons.¹⁹

Si addition also would increase the resistivity of the matrix phase.²⁰ Thus the combined effect of both P and Si in pure Fe, while retaining soft magnetic properties, increases the resistivity significantly leading to AC magnetic performance matching to that of relatively high Si containing commercial alloy. A more detailed micro analysis using advanced technique like 3 dimensional atom probe (3DAP) may provide better understanding on the exact partitioning behaviour of Si and P between the matrix and the precipitates and the mechanism of enhanced nano-precipitation. In conclusion we report a new (Fe-P)-Si based alloy with relatively high induction (1.8-1.9 T), low coercivity (<80 A/m), high resistivity (~ 38 $\mu\Omega$ cm) and low core loss (217 W/kg at 1 T/1 kHz) comparable to the commercially available M530-50 A5 Si-steel.

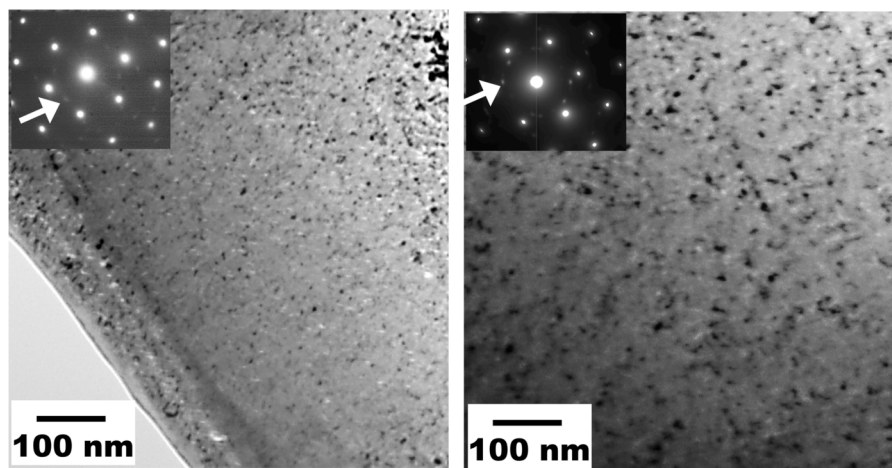


FIG. 5. TEM micrographs of a) L-Si alloy and b) H-Si alloy with latter clearly showing enhanced number density of the Fe_3P nano precipitates. Inset shows the diffraction patterns obtained from the (110) zone axis of the α -Fe matrix phase with white arrows indicating the diffraction spots corresponding to the Fe_3P nano-precipitates.

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