Study of mechanical properties of an iron phosphorus based alloy under various aging time

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Abstract. We reported to Fe-P based alloys observed as one of the promising materials which could replace Si-steel on the basis of two factors: i) the cost of Fe-P based alloys are well expected to be about 20\% lower than Fe-Si alloys. ii) Phosphorous addition enhances the soft magnetic properties of pure Fe. Elemental Fe with Fe-P and Fe-Si master alloys were taken in suitable weight ratios and induction melted to obtain Fe-0.4wt.% P-0.85wt.%Si alloy. Fe-P based alloys can be conventionally produced by powder Metallurgical route. The optical micrographs of Fe-P-Si alloys were aged at 500 °C with different aging time. Aged samples strength has increased due to the interaction of the moving dislocations with dispersed precipitates. The mechanical properties of the Fe-P-Si aged samples were increasing with aging time up to the peak aged then decrease for the over aged samples. TEM reveals the presence of two phases: Fe3P phase in \( \alpha \)-Fe matrix phase. The sizes of the precipitates were increasing from ~1.2 nm to ~2.2 nm with aging time.

Keywords. Mechanical properties, Fe-P alloys, aging time.

1. Introduction

Due to growing energy crises coupled with environmental issues, the rise of electric and hybrid electric vehicles is transforming the future. Some of the main components of an EV (Electric vehicle) are motors, battery and controller. As in the case of magnetic materials required for EV motors, soft as well as hard magnets of high performance materials are required. In the case of soft magnetic materials, it should possess high saturation magnetic flux density (Bs) with low core losses, high permeability, and better mechanical properties etc. [2] the iron to silicon ratio highly influences the properties and, with the ratio close to one and work hardening at elevated temperatures as compared to Fe-based TiAl alloys of last generation (TMN alloys)[1]. Sintered ferrous material is elaborately used in different engineering applications due to their high hardness and good wear resistance. The compression properties of the novel alloys are measured at room temperature and elevated temperatures. Where Ti-45Al-5Fe-5Nb alloy showed similar strengths at room temperature to sintered TiAl alloys of last generation application [1].

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with low concentrations of iron and silicon the best properties were obtained. As per the present experimental results, it is possible to multiply by two or three the present limit of 0·1 wt-% Fe in these alloys at natural aging (T4) and to obtain 7% of minimum elongation which are desired by the automotive industries. One of the benefits of working with saturated Cu microstructure is that, it could estimate the true temperature of the solution heat treatment by conducting a post-analysis of Cu content in the dendrites. Which can be helpful to improve the temperature distribution in heat treating processes and also helps in reducing the variability in properties.[3]

With the increase in the contents of iron and silicon in the alloys has led to decrease in the mechanical properties of the alloys. However, the reduction in tensile strengths and ductility was quite small. Therefore, the Al-5Mg-0.8Mn alloy (AA5083 alloy) could be used by higher contents of iron and silicon. When the materials are cast under near-rapid cooling, such that continuous strip casting process.[4]

Higher contents of iron alloys are necessary to change their microstructure by using heat treatments, and to improve their wear resistance. Suitable the individual application requirements. Changing in chemical composition and heat treatment carried out to this alloy related to microstructural characteristics and mechanical properties of high Cr white cast iron alloys are presented.[5]

The optical and SEM micrographs can be inferred the aluminum alloy leads to grain refinement and grain structure modification. The wear properties of AA7175 alloy improved by the addition of TiB2 and higher than that of the unreinforced aluminum alloy. The wear resistance kept increasing with decrease in particle size of TiB2 particulates. [6] It showed the raise in tensile strength due to the inclusion of phosphorus in pure iron; however, it decreased the ductility. The grain refining effects and rising in tensile strength on inclusion of Phosphorus (P) were found to be very significant. However, the change in mechanical properties occurs with the increase in annealing time at any temperature [7]. Under quasi-static loading, heat treated samples were increase in strength and ductility, for all the alloy compositions. The addition of TiB2 improves the tensile strength [8]

Microstructural studies revealed that the prepared samples were free from Fe3P phase precipitation and the average grain size increased with increasing the phosphorus content giving rise to the decrease of hysteresis losses.[9] Corrosion resistance has inversely proportional to porosity[10]. As the phosphorus content increases results into increase in the sintered density and Fe-3P alloys attaining near full density[11] (Fe-P)-Si based alloy with relatively high induction, low coercivity, high resistivity and low core loss comparable to the commercially available Si-steel[12].

Fe-P based alloys are considered as one of the promising materials which could replace Si-steel on the basis of two factors: i) the cost of Fe-P based alloys are expected to be 20% lower than Fe-Si alloys. ii) Phosphorous addition enhances the soft magnetic properties of pure Fe; Fe-P based alloys can be produced conventionally by wrought alloy process or by powder metallurgical route. In powder metallurgical route, due to the lower solubility of P in Fe (5wt% in α-Fe) segregates at the grain boundaries resulting in brittleness. Alternatively powder metallurgical route, through liquid phase sintering (LPS) can also be incorporated but involves complex compaction techniques. The present work was carried out by wrought alloy process of low Si, Fe-P based alloy, which has attractive AC and DC magnetic properties.

2. Experimental Details

Elemental Fe with Fe-P and Fe-Si master alloys were taken in suitable weight ratios and induction melted to obtain Fe-0.4wt.% P-0.85wt.%Si alloy. The melt was under cast into a mould to obtain a diameter of 65 mm and 400 mm long ingot, and the alloys were undergone a radiography test to separate the pipe and sound portions.
The melted ingots were forged in open die forging to get a required shape and size for the rolling operation. Thin sheets typically ~0.5 mm thick was obtained followed by hot-rolling at 900 °C. The rolled sheet was solutioned at 1000 °C and subjected to an ageing at 500 °C from 10 min to 10 hours and then quenched by water.

The transmission electron microscope was used to study phase analysis of the precipitates and the other details of the microstructure. Micro-hardness of the samples was measured by using knoop – micro hardness tester with a load of 1 kg.

Average hardness of each specimen was determined by indenting the sample six times. The average values obtained were used to plot the age hardening curve. The tensile test of the samples was carried out using micro tensile testing machine at room temperature. The tensile specimens were prepared by ASTM E8 standard with the sample thickness 0.5 mm. The four-probe potentiometric technique was used to measure the resistivity of the samples.

2.1 Microstructure

A very small scale structure of materials is called microstructure. Structure should prepare above 25× magnification of microscope. The optical micrographs of Fe-P-Si alloys aged at 500 °C with different aging time. Fe-P-Si rolled sheets were solutioned at 1000 °C with 1 h soaking time followed by water quenching to form a supersaturated solid solution of P in α-Fe matrix. After Solutionizing the samples were aged at 500 °C with different aging time. The materials grain size was measured by the different aging time.

2.2 Microstructure Characterizations

Quantify microstructural characteristics, morphological and material property. Morphological property can be determined such as volume fraction, inclusion morphology, void and crystal orientations. Micrographs commonly used optical as well as electron microscopy. Material property can be determining of properties in micron and submicron level.

2.3 Transmission electron microscopy

TEM transmits electron through a specimen and form an image. The specimen is often less than 100nm thick in ultrathin section. The images have formed from the interaction electrons. The image is magnified and focused onto screen. Reveals the existence of secondary Fe3P Nano precipitates phase in α-Fe matrix phase. With increase in aging time the size of the precipitates were increasing as shown in table-1

| Aging Time (h) | Average Grain Size (µm) | Average Precipitate Size (nm) | No. of precipitates per unit area observed by TEM(10^18 m^-2) |
|---------------|------------------------|------------------------------|-----------------------------------------------------------|
| 0             | 156                    | -                            | 0.63                                                      |
| 0.5           | 125                    | 1.2                          | 0.27                                                      |
| 2             | 126                    | 2.0                          | 0.27                                                      |
| 10            | 132                    | 2.2                          | 0.24                                                      |
Table 1. Average Grain Size, Average Precipitate size and number of Fe3P precipitates per unit area in α-Fe matrix of Fe-P-Si aged samples for various aging time

2.4 **Knoop hardness test**

This test can also be called as micro hardness test. Here test were conducted by the standard of ASTM E-384. The testing purpose only have small indentation on particular brittle materials or thin sheets. A pyramidal diamond point had move on the polished surface. Test material is tested on known load (often 100g). It is happening with dwell time and resulting indentation which can be measured by the microscope.

2.5 **Micro Tensile Test**

Tensile test deduces the strength of the material subjected to a simple stretching operation. The Tensile specimens were prepared by ASTM E8 standard with the sample thickness 0.5 mm. The standard four-probe potentiometric technique was used to measure the resistivity of the samples.

3. **Result and Discussion**

3.1 **Microstructure Study**

Figure-1 shows the optical micrographs of Fe-P-Si alloys aged at 500 °C with different aging time. Fe-P-Si rolled sheets were solutionized at 1000 °C with 1 h soaking time followed by water quenching to forms the supersaturated solid solution of P in α-Fe matrix. After Solutionizing the samples were aged at 500 °C with different aging time. The averaged grain sizes of the samples were measured using intercept method and inserted in figure 1. It was observed that the solutionized samples become finer in grain size after aging whereas there was not significant change in grain size with aging time. Because the aging temperature of 500 °C was lower than the recrystallization temperature. Hence, the grain growth was not significant. Optical microstructural images (with inserted average grain size) of Fe-P-Si alloy in a) the solutionized condition, b) under aged (0.5 h), c) peak aged (2 h) and d) over aged (10 h) at 500 °C.
3.2 Transmission Electron Microscopy Study

TEM study of solutionized samples show the single phase of α-Fe (P) and no significant Fe₃P precipitates were observed because of the presence of supersaturated solid solution of P in α-Fe matrix as shown in figure 2(a). While the SAED pattern from [100] zone axis of α-Fe(P) matrix phase reflects the diffraction spots from Fe₃P precipitates. Hence, there were some small volume fraction of fine Fe₃P precipitates are present which is highly difficult due to their fine size and the coherency with the BCC-Fe matrix. TEM images of Fe-P-Si unaged sample shows the absence of precipitates; inserted high magnification figure 2(b) SAED pattern from [100] zone axis of α-Fe(P) matrix phase shows the diffraction spots of Fe₃P precipitates.

Figure 1. Microstructure of aging study

Figure 2. TEM Images
Further, the TEM investigation of Fe-P-Si aged samples as observed from figure 2 reveals the presence of secondary Fe₃P Nano precipitates phase in α-Fe matrix phase. With increase in aging time, the sizes of the precipitates were increasing as shown in table 1. In figure 2 (b), the inserted SAED patterns of the Fe-P-Si aged samples along the zone axis of [001] shows the diffraction spots corresponding to Fe₃P phase. These precipitates also inhibit the grain growth with aging time. This is also a reason of no significant grain growth with aging time.

3.3 TEM Images of aging study

TEM images of Fe-P-Si aged samples showing Fe₃P nano precipitates dispersed in Fe matrix phase. The inset shows the SAED pattern from [100] zone axis of α-Fe matrix with diffraction spots corresponding to Fe₃P phase

![TEM Images of aging study](image)

Figure 3: TEM Images of aging study

3.4 Mechanical Properties

The micro hardness of Fe-P-Si alloy with respect to ageing time at 500°C is shown in Figure 4. The hardness increases upon ageing, reaches a peak value at 2 h and decreasing further, where 10 h is considered as over ageing. The change in the hardness with aging time can be explained by precipitation hardening mechanism.

The increased in the strength of an aged samples is due to the interaction of the moving dislocations with dispersed precipitates. Initially when the precipitates size was very small, the dislocation cuts through the precipitates zone and strengthen the material. As the aging time increase, the size of the precipitates increase and the inter particle distance decreases.

When the precipitates size is high, the dislocation unable to cut through the precipitate, then bows to bypass. As the distance between the precipitates during over aging increases, the strength of the alloy decreases.
Figure 4 shows the stress-strain curve of the Fe-P-Si aged samples with varying aging time. Strength of the parent iron increases due to the presence of P by strengthening the ferrite matrix. Hence also involves precipitation (Fe₃P) hardening effect. From figure 5 it is observed that the tensile strength of the aged samples increased with aging time due to precipitates hardening mechanism as explained above. While there is not significant effect on the Variation of Knoop micro hardness of Fe-P-Si aged samples at 500 °C with different aging time and the optical image of the indentation on the specimen were inserted ductility of the Fe-P-Si aged samples with aging time.
4. Conclusion

In this work, effect of aging studies on the mechanical properties of Fe-P-Si alloys have been studied by doing the aging treatment on the solutionized rolled sheets at a temperature of 500 °C (lower than the recrystallization temperature) with varying aging time. From the results of the proposed work, we observed the following concluded remarks:

1. There is no significant grain growth of the aged sample with varying aging time. This can be due to low kinetic energy for grain growth because of the lower aging temperature (below recrystallization temperature) and due to inhibits the grain growth by secondary Fe₃P nano-precipitates phase dispersed in α-Fe matrix phase.

2. TEM reveals the presence of two phases: Fe₃P phase in α-Fe matrix phase. The sizes of the precipitates were increasing from ~1.2 nm to ~2.2 nm with aging time.

3. The mechanical properties of the Fe-P-Si aged samples were increasing with aging time up to the peak aged then decrease for the over aged samples. The enhancement in the strength of the Fe-P-Si alloys by aging can be explained by precipitates hardening mechanism.

4. The enhancement in the resistivity of the Fe-P-Si aged samples is due to the presence of fine Fe₃P nanophase precipitates acting as a scattering center for the flow of the electron.

5. Hence Fe-P-Si aged samples can be used as potential materials as a stator in a motor for the automotive applications due to their high mechanical and electrical properties.

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