Journal of Material Science and Mechanical Engineering (JMSME)
p-ISSN: 2393-9095; e-ISSN: 2393-9109; Volume 5, Issue 4; October-December, 2018, pp. 177-180

http://www.krishisanskriti.org/Publication.html

Effect of Heat Treatment on the Mechanical Properties of Fe - 0.13%P - 0.05%C Alloys

Yashwant Mehta *[a], Himanshu Gupta [a], G. Madhusudhan[a], Mayur Agarwal[a] and Rakesh Meena[a]

[a]*National Institute of Technology Srinagar*
E-mail: yashwant.mehta@gmail.com

Abstract—*The Delhi Iron Pillar withstood atmospheric corrosion for about 1600 years. Phosphorus leads to the strengthening of the iron pillar and imparts corrosion resistance to it. In modern steels Phosphorus content is limited to a maximum of 0.05%. The alloy composition chosen for the present study is similar to that of the Delhi Iron Pillar namely Fe - 0.13%P - 0.05%C. High Phosphorus content in steel contributes to increased yield strength and ultimate tensile strength. It causes an increment in hardness of steels too but elongation and reduction in the area at failure are decreased. However, such brittle behavior can be remedied by adding small amount of carbon. The mechanical properties of the aforesaid alloy are studied after heat treatment in the duplex phase region of Fe-P phase diagram. The rate of cooling of the alloy is varied (annealing, normalizing) to obtain different microstructures. These microstructures and fractured surfaces are studied and correlated with the mechanical properties.*

Keywords: *Corrosion resistance, annealing, normalizing, metallography, fractured surface*

1. INTRODUCTION

The Delhi Iron Pillar standing in Mehrauli village inQutabMinarcomplex Delhi has been a center of attraction for many engineers, specially corrosion technologists, as it has withstood corrosion for the last 1600 years even in the pollution of Delhi. The composition of the iron pillar is comparable to that of low carbon steel and shows a wide range as C (0.03-0.028), Si (0.004-0.056) and P (0.11-0.48). However, Phosphorus is traditionally known to be detrimental to the mechanical properties of steel and it is therefore avoided in modern steel making. The brittleness of phosphorus containing steel arises due to grain boundary segregation of phosphorus. This normally occurs due to prolonged exposure and tempering of phosphorus containing steels in the temperature range 250-600°C and the phenomenon is known as temper embrittlement. Phosphorus also increases ductile to brittle transition temperature of steel.

Phosphorus dissolves in both γ and α iron and forms the chemical compound Fe_3P, the solubility of phosphorus in α iron is 2.6 at.% at 1020°C and 1.2 at.% at 600°C, its solubility in γ iron is considerably less. Phosphorus dissolved in ferrite imparts cold shortness. An increase in phosphorus content increases the tensile strength but sharply reduces impact strength and ductility of steel. Phosphorus improves machinability in free-cutting steels. In terms of welding, phosphorus content of over 0.04% renders weldments brittle and increases the tendency to crack. The main objectives of this work are to determine mechanical properties viz. tensile strength. Metallographic study of samples and study of fractured surface.

2. MICROSTRUCTURAL INVESTIGATION ON ANCIENT INDIAN IRON

The idea of high phosphorus is already being implemented in some grades of weathering steels [1]. In order to understand the ways to overcome the problem of embrittlement in phosphoric iron by ancient blacksmiths many careful microstructural studies of ancient Indian irons (dating from the 5th Century AD up to 19th Century AD) was undertaken. An iron clamp was studied from Gupta period (6h Century AD) temple at Deogarh. The microstructure of the iron clamp was obtained and the composition was estimated by wet chemical analysis (using ICTP-AES) to be 0.30% P, 0.21% Si, 0.18% Al, 0.11% Ni, 0.024% C and 0.013% S. The origin for Ni and Al in this iron isn't known. However, compositional analysis of microstructure did not reveal any other element other than P. The Deogarhiron that was used for the analysis contained a high P, and most importantly, a very low carbon. Based on this survey, it was anticipated that the deleterious effect of P would have been avoided by the presence of small amount of C.

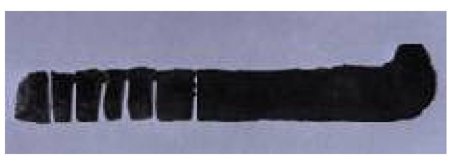

Figure 2.1 Iron Clamp from Gupta Temple at Deogarh [3]

Etched microstructure of ancient iron with nital revealed [2] iron was essentially ferritic. This is understandable because of its low carbon content. The only other element identified by local compositional analysis in the SEM and EPMA in the iron matrix was phosphorus [3]. This was again confirmed with the compositional analysis of several ancient Indian irons [4, 5]. However, the phosphorus distribution is found to be non-uniform due to micro- segregation that occurs during the extraction process [6].

Later Oberhoffer etchant was successfully employed to reveal pattern of P segregation in ancient Indian Irons [7]. The etch deposits Cu on regions low in P and therefore appears dark under optical microscope. Region high in P appears bright because of lower Cu deposition. The same etchant applied to iron clamp of Deogarh to understand the micro-segregation of P. It was found that areas depleted in P near the slag inclusions (figure 2.2a). The variation in P concentration was observed by the difference in gray coloration. With the regions in low P appearing more grayish than region high in P, which appeared comparatively light colored (figure 2.2b).

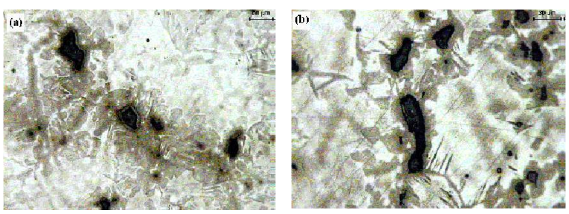

Figure 2.2 Optical microstructure of the iron after etching in Oberhoffer etchant. The P depleted regions appear dark. Notice the P depletion next to the entrapped slag inclusions [3].

A suitable heat treatment of the phosphoric iron in two phase (α+γ) region of the Fe-P phase diagram (figure 2.3) will lead to the precipitation of γ phase along the α phase grain boundaries. As the γ phase possesses a lower P solubility, the segregation of P to the grain boundary regions can be avoided. On later cooling down the sample to room temperature at an appropriate cooling rateall the allotriomorphic and Widmanstatten austenite along the grain boundaries will transform by massive transformation to ferrite [8]. However, the differences in phosphorus composition between the grain boundary and interior grains will remain even after the grain boundary austenite has transformed to ferrite, if appropriate cooling rates are selected after the high temperature anneal. It is this difference in P composition that results in ‘ghost’ structures on etching with Oberhoffer or nital etchants [8].Therefore, the first important idea to avoid P segregation along the grain boundaries is to create the right soaking conditions in order to precipitate austenite allotriomorphs along ferrite grain boundaries [8].

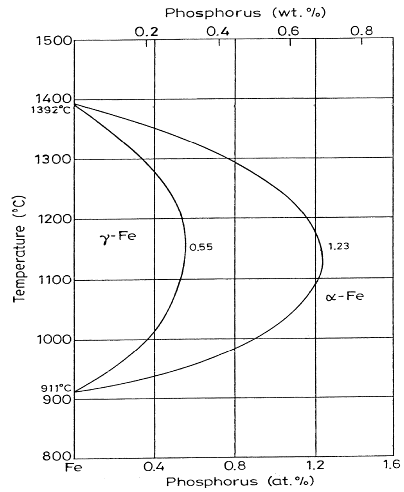

Figure 2.3 High temperature gamma loop region of the Fe-P phase diagram [9]

3. PROCEDURE

Soaking of Fe-P-C alloy of given aforesaid composition at 900°C for 4 h (Figure 3.1)and later cooled at two different rates, one is in air i.e., normalization and another furnace cooled i.e., annealing.

Figure 3.1 Soaking of raw samples at 900°C for 4hrs

The composition of all the elements are as follows; (in wt%) 0.05% C, 0.13% P, 0.26% Si, 0.2% Mn, 0.13% Cr, 0.003% Al, 0.023% Cu, 0.004% Ni, 99.2% Fe.

Machining of the samples was done by lathe machine to give it the standard shape for tensile test specimen. Tensile test standard specimen was then tested using UTM for the determination of tensile strength and ductility by measured by % elongation in both the samples. Change in gauge length of tensile specimen gives the % elongation. Fractured shape of the specimen is cup and cone type, ductility is further confirmed by the study of fractured surface through SEM image analysis.

Specimen preparation for the metallographic study includes grinding on Si-C emery papers followed by polishing. The etching reagent used for this alloy is Oberhoffer’s reagent, the composition of the reagent is as follows; 500ml distilled water, 500ml ethanol, 0.5gm $SnCl_2$, 1gm $CuCl_2$, 30gm $FeCl_3$.

Oberhoffer etchant is used to reveal the phosphorus depleted regions by the dark contrast.

4. RESULTS& DISCUSSIONS

Tensile test results-

Table 4.1 - Sample 1 (normalized)

Proof Stress (MPa)	UTS (MPa)	% Elongation
162.21	212.12	20%

Table 4.2 - Sample 2 (annealed)

Proof Stress (MPa)	UTS (MPa)	% Elongation
137.25	182.17	16%

Metallographic inspection - Sample 1 (normalized)

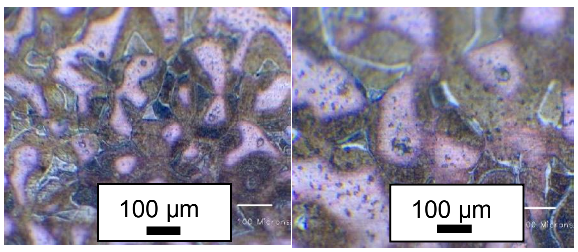

Figure 4.1 Microstructure of air-cooled sample.

Metallographic inspection - Sample 2 (annealed)

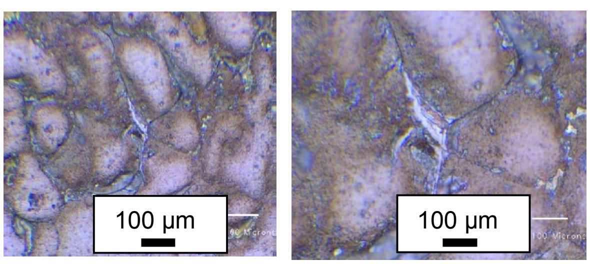

Figure 4.2 Microstructure of furnace cooled sample

SEM results for sample 1 fractured surface -

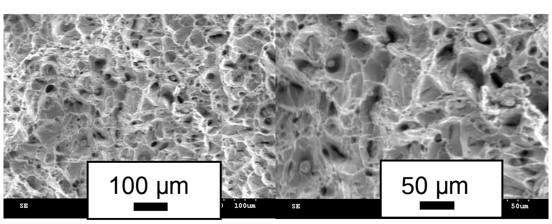

Figure 4.3 Fractured surface of air-cooled sample

SEM results for sample 2 fractured surface -

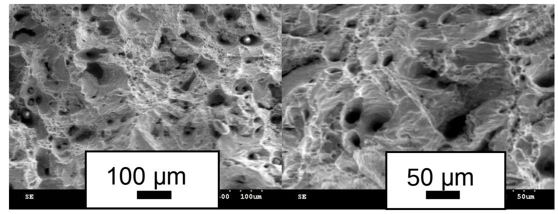

Figure 4.4 Fractured surface of furnace cooled sample

The basic aim was to heat treat the alloy in the duplex phase ($\alpha+\gamma$) region by maintaining proper temperature, so that phosphorus can be kept away from grain boundaries. Since the solubility of carbon is higher in austenite than phosphorus, therefore at higher temperature the carbon drives phosphorus away from the grain boundary by site competition effect. This improves mechanical properties by reducing the chances of grain boundary embrittlement due to phosphorus.

Microstructure of phosphoric iron show interesting features by making use of Oberhoffer etchant. Because of low carbon content, the microstructure is essentially ferritic. Oberhoffer etchant deposits Cu on the regions low in phosphorus and therefore these regions appear dark. Regions high in phosphorus appear bright because of low Cu deposition. As shown in figures4.1 and 4.2 the grain boundary is depleted with phosphorus. Hence it is dark colored. And the area in between grain boundaries appears bright.

In SEM results the black spots (dimples) indicates the ductile fracture as can be seen in figures 4.3 and 4.4.

5. CONCLUSION

The general trends previously observed by many authors were confirmed by those observed in the current study of phosphoric iron. From results and microscopic investigation, we see that ductile Fe-P can be achieved by avoiding phosphorus segregation to the grain boundaries. This can be done by precipitating austenite allotriomorphs, by utilizing a critical amount of carbon to segregate carbon/precipitate carbide (in case of high carbon) at grain boundaries.

For removing phosphorus from the grain boundary soaking temperature should be high. When the temperature is high, carbon diffuses towards the grain boundaries thus driving off the phosphorus from the grain boundaries by site competition effect. High carbon content favors carbide formation at grain boundaries, on increasing the soaking time for diffusion of phosphorus.

After a suitable heat treatment, the ultimate tensile strength of the material depends upon the rate of cooling. In normalized sample ultimate tensile stress is 212.12MPa which is greater than ultimate tensile stress of annealed sample, which is 182.17MPa. But normalized sample has more ductility than the annealed one.

The elongation of the annealed sample is lower than the normalized sample. The reason is that phosphorus returns to the grain boundaries when the steel is held at temperature just below the A_1 temperature for a few hours or so.

6. ACKNOWLEDGEMENTS

We wish to thank Shri Ashok Aggarwal, Vaishnav Steel, Muzaffarnagar for providing the alloy casting.

REFERENCES

[1] Misawa T, Kyuno T, Suetaka W and ShimodairaS *Corros. Sci.* 11**,**1971, 35

[2] Balasubramaniam R 2002 *Delhi Iron Pillar* - New insights (Shimla: Indian Institute of Advanced Study)

[3] Piccardo P, Ienco M G, Balasubramaniam R and Dillmann P 2003 *Curr. Sci*. 87, 2004, 650-653

[4] R. A. Hadfield, *J. Iron Steel Inst*. 85, (1912)153, 171.

[5] Neogi P 1914 *Iron in ancient India* (Calcutta: Indian Association for the Cultivation of Science)
Percy J 1864 *Metallurgy-Iron and steel* (London: John Murray)

[6] Vizcaino A, Budd P and McDonnel J G 1988*Bull. Mater.Mus.* 29 13

[7]Stewart J W, Charles J A and Wallach E R *Mater. Sci. Technol***16**(2000)283, 291

[8] Gouthama and R Balasubramaniam,*Bull. Mater. Sci.,* Vol. 26, No. 5, August 2003, pp. 483-491

[9] Kubaschewski O 1982 Iron-Binary phase diagrams (*Berlin: Springer Verlag*) pp. 84-86