

# OPTIMIZATION OF THE HOT ROLLING PROCESS PARAMETERS TO INDUCE BENEFICIAL EFFECTS IN IRON ALLOYS BY ADDITION OF DETRIMENTAL ELEMENT PHOSPHORUS

M.A. Islam<sup>1</sup> and Y. Tomota<sup>2</sup>

<sup>1</sup>Materials and Metallurgical Engineering Department, Bangladesh University of Engineering and Technology (BUET), Dhaka-1000, Bangladesh

<sup>2</sup>Institute of Applied Beam Science, Graduate School of Science and Engineering, Ibaraki University, Hitachi, Japan

## ABSTRACT

Steel is an alloy, mainly of iron and carbon with or without additions of other elements. Whatever may be the type of steel, it contains some other trace elements such as sulfur (S), phosphorus (P), arsenic (As), antimony (Sb), etc. In many cases, these elements degrade the mechanical properties of steels. So, for steel, P is considered as a detrimental element. However, through proper control of processing parameters during hot working, it is possible to make the detrimental P to be a beneficial alloying element. In this study, the heightening of tensile strength of commercial grade of pure iron by P addition has been studied. It has been found that addition of P significantly increases the tensile strength of the pure iron without severe deterioration in ductility level and fracture mode.

**Keywords:** Pure Iron, Iron-Phosphorus Alloy, Ductile Fracture, Tensile Strength.

## 1. INTRODUCTION

About 98% of the natural iron ores are used for the production of different types of ferrous alloys such as pig iron, cast iron, wrought iron and various types of steels [1]. In these iron ores (which are of mainly oxide types) P also remains mixed along with many other impurity elements. Interestingly, in many cases, the colours of iron ores and P are very similar (Fig 1).

During refining of these oxide type iron ores, it is very difficult to remove all impurity elements completely. Phosphorus is one of the impurity elements that remain present almost in all ferrous alloys. Steel is a ferrous alloy, which is usually used in the case of load bearing engineering structures, such as buildings, bridges, various transport vehicles, pressure vessels, power transmission components, etc. In the case of many different low alloy steels, serving at high temperatures, e.g. order of 500°C or during slow cooling from high temperature, P atoms try to segregate at grain boundaries and makes the steels brittle. This type of embrittlement might change the brittle transgranular fracture mode (either in fatigue or fast fracture) to intergranular one, with subsequent deterioration in the mechanical properties; especially fracture toughness values [2-4]. So, in general, phosphorus is considered as a detrimental

element in steels. In many cases, hot rolled steel is cold worked for some special properties, e.g. good surface finish, better dimensional tolerances and/or better strength. However, the fatigue properties of the cold worked steels are also deteriorated severely [5,6].

It is well established that P induces temper embrittlement and degrades the mechanical properties of many steels. However, careful control of the thermomechanical treatment, phosphorus in steel might be beneficial in many cases. As per Hall-Patch relationship given below, refining in grain sizes means heightening the tensile strength of the steel.

$$\sigma_o = \sigma_i + KD^{-1/2} \quad (1)$$

Where  $\sigma_o$  is yield strength,  $\sigma_i$  is friction stress,  $K$  is locking parameter and  $D$  is the grain diameter. Additions of small amount of phosphorus in iron/steel causes decrease the prior austenite as well as the ferrite-pearlite grain sizes because of pinning action of Fe-P clusters. It also increases the strength of steels by solid solution strengthening mechanism [7,8]. The purpose of this research work is to strengthen the ferrous alloy with P additions by avoiding its detrimental effects through controlling the hot rolling process parameters.

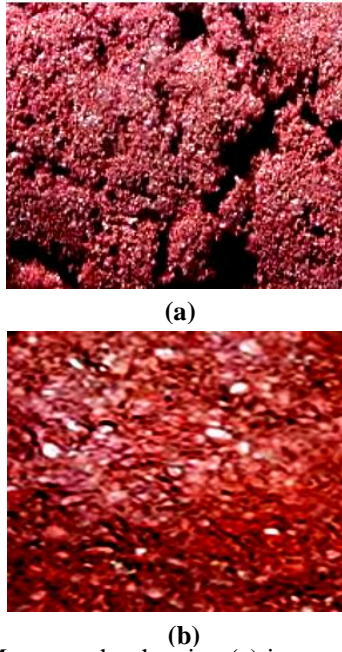


Fig 1. Macrographs showing (a) iron ore and (b) pure phosphorus.

## 2. EXPERIMENTAL

The materials used in this work were commercial grade of pure iron and phosphorus added iron (0.11% P: Fe-P-I and 0.21% P: Fe-P-II) alloys. The chemical compositions of these three alloys are presented in Table 1. These alloys were melted in vacuum by induction melting. The cast alloys were then rolled to thin sheet by multi-pass rolling system at 950°C at National Institute of Materials Science, Tsukuba, Japan. After hot working, all alloys were forced for faster cooling by compressed air flow in order to minimize grain growth as well as the surface oxidation of the rolled products.

Table 1: Chemical compositions (wt %) of the iron and Fe-P alloys used.

Material ID	C	Si	Mn	P	S
Pure Iron	0.003	0.001	0.001	0.001	0.001
Fe-P-I Alloy	0.003	0.001	0.001	0.110	0.007
Fe-P-II Alloy	0.003	0.001	0.001	0.210	0.005

After cooling of the rolled sheet to room temperature, small pieces were cut from all three rolled sheets. Then metallographic samples were prepared and they were etched with 5% nital following the standard procedure. They were then observed under optical microscope and SEM to identify the microstructures and establish the grain sizes. The ferritic microstructures of various alloys are shown in Fig 2. The average ferrite grain sizes of all these alloys were measured and presented in Table 2. Tensile samples were also prepared and tests were carried out by a Universal tensile testing machine at room temperature in the air. The dimensions of the tensile specimens are shown Fig 3.

The stress versus elongation curves thus obtained from tensile tests are shown in Fig 4. After tensile tests, fracture surfaces were cut carefully avoiding oxidation and any possible damage of the fracture surfaces and

they were observed under the SEM to investigate various fracture features. The fracture surfaces are presented in Fig 5.

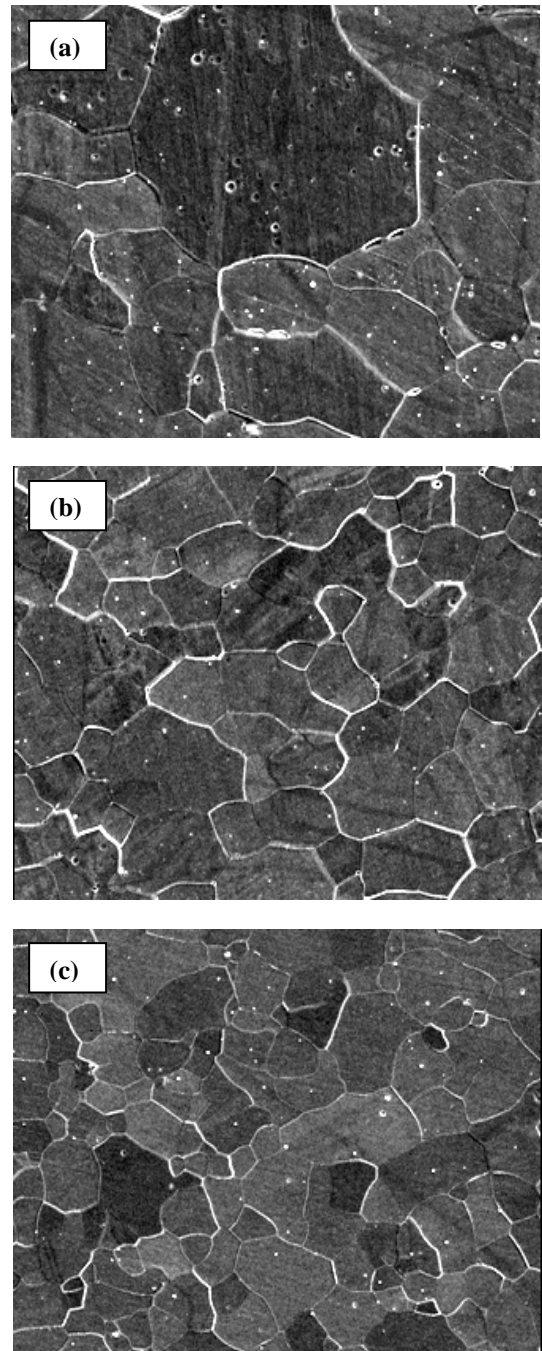


Fig 2. Microstructures of different iron alloys (a) pure iron, (b) Fe-P-I and (c) Fe-P-II alloys showing their relative grain sizes.

Table 2: Average ferrite grain sizes ( $\mu\text{m}$ ) of the alloys after hot rolling.

Material ID	Ferrite Grain Size, $\mu\text{m}$
Pure Iron	83
Fe-P-I	42
Fe-P-II	40

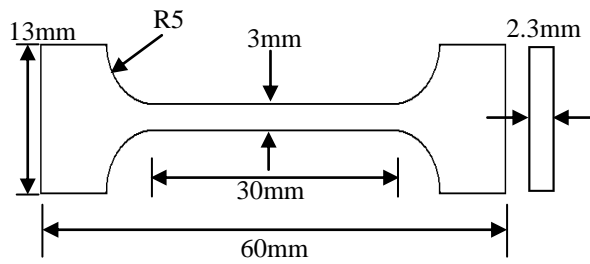


Fig 3. Geometry of tensile test specimen.

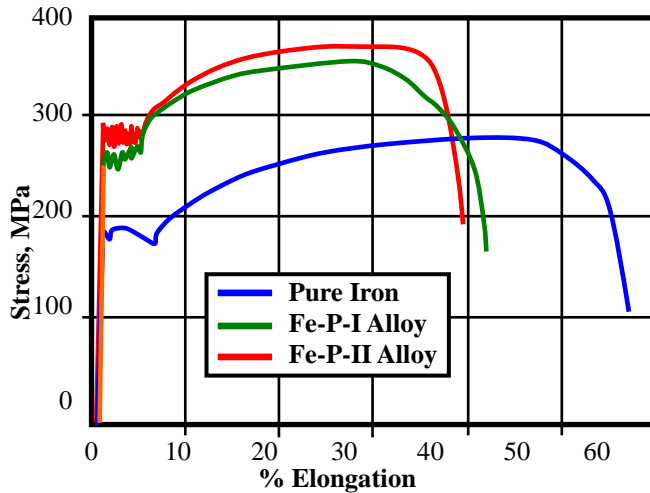


Fig 4. Nominal stress versus elongation curves of various iron alloys tested at room temperature in the air.

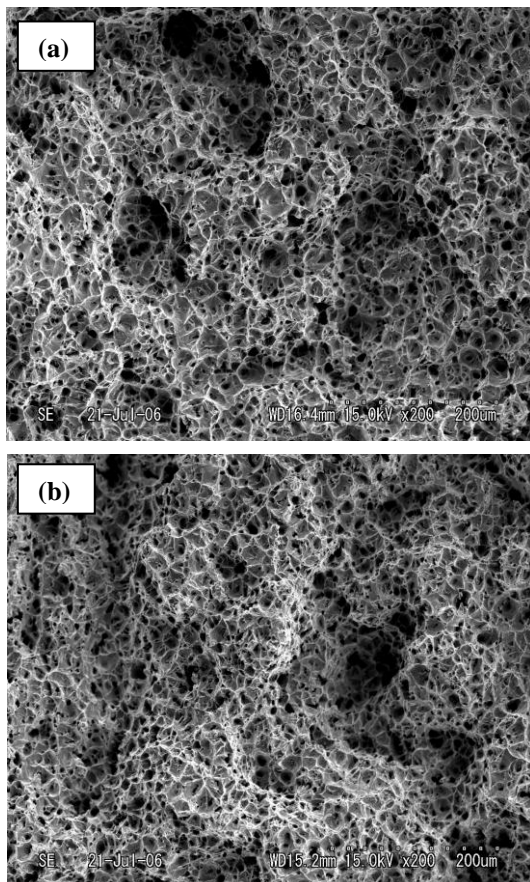


Fig 5. Microvoid coalescence type ductile fracture on tensile specimens (a) pure iron and (b) Fe-P-II alloy tested at room temperature in the air.

### 3. RESULTS AND DISCUSSION

In the earlier section, it has been mentioned that P is a detrimental element for iron alloy because it reduces the toughness of the alloys and also changes the normal fracture behaviour. At the same time, it has also been shown by the mathematical relationship that finer grain size induces more strength in the alloy. From Fig 2, it is clear that addition of P in the commercial grade of pure iron refines its ferrite grains gradually. Addition of 0.11%P resulted about 50% decrease in the ferrite size. At high temperature, Fe-P clusters acts as a barrier to coarsen the ferrite grain. So, merging of adjacent ferrite grains becomes difficult. As a result, at high temperature, ferrite grain of Fe-P alloys remains relatively finer. Grain refinement of ferrous alloys by P addition has also been mentioned by other investigators [9,10]. It has also been mentioned that the alloy ingots were hot rolled severely to form thin sheet. During rolling, austenite grains of the steel elongate in the direction of rolling. However, at high hot rolling temperature very quick dynamic recovery takes place and gradually equiaxed type finer austenite grains are formed.

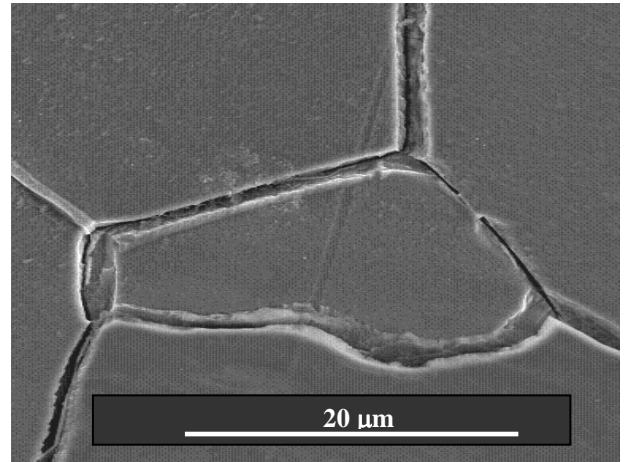


Fig 6. Optical micrograph showing the mismatches (blank spaces) between two adjacent grain boundaries of pure iron.

Here it is important to mention that P has a great tendency to segregate at ferrite grain boundaries and makes them brittle. Because of this, the toughness of steels with P segregation at grain boundaries decreases drastically. The low magnification micrographs presented in Fig 2, apparently, reveals no mismatch or blank space at grain boundaries. However, it is possible to reveal mismatches of various degrees at grain boundaries under SEM at high magnification (Fig 6). At high temperature, the movement of solute atoms in iron is very high. As a result, for solute atoms, there exists no significantly better microstructural site to be segregated over there [3,4]. However, with decrease in temperature during cooling of the hot worked iron alloy, movement of solute atoms becomes restricted. On the other hand, the difference in energy levels of grain and grain boundaries become higher because of free space (mismatch) at grain boundaries. So, solute atoms (here P) start to segregate at grain boundaries preferentially. This type of segregation



induces brittleness in the iron alloy and causes degradation in mechanical properties. The degradation in mechanical properties becomes in severe stage, if the cooling rate is slower, e.g. slow cooling after rolling or forging operation. In these cases, the brittle failure mode of the steel might change from its normal transgranular fracture mode to intergranular one, Fig 7.

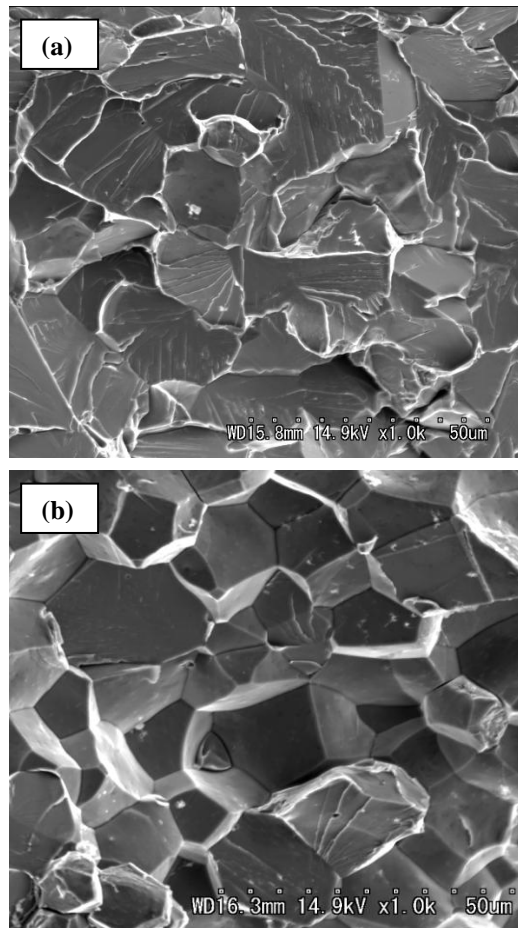


Fig 7. SEM micrographs showing (a) normal transgranular brittle failure and (b) P segregation induced intergranular type brittle failure.

From Fe-P binary phase diagram presented in Fig 8 it is clear that P stabilizes the ferrite and up to 0.65% P is soluble in ferrite at around 1100°C. With decrease in temperature or increase in P, very fine Fe-P precipitates (10-400 nanometer size) are formed [11]. Slower cooling rates accelerates this precipitate formation. With increase in Fe-P precipitates, brittleness of the hot worked steel also increases. As a result, the steel gradually loses its load bearing capability and acceptability as structural materials. If this high P containing steel is cold worked, then it breaks easily because of cold shortness of the steel. All these unwanted outcomes are possible to overcome by selecting proper hot working process parameters. For first step, the high P containing steel is rolled from relatively higher temperature to avoid any possibly segregation of P during hot working period. As a part of second initiative to avoid the detrimental effect of P, if it is possible to deform the steel during hot working vigorously in a very powerful rolling machine, the grain

size of the steel will be finer, which means structures with more grain boundary areas. These higher proportions of grain boundaries eventually decrease the concentration of P precipitates. After finishing hot working, the product is then cooled at a relatively faster rate to allow P precipitation as minimum as possible, which is as another technique to avoid or minimize P segregation. These overall initiatives allow the steel to retain the beneficial effects of P, i.e. fine grained cold shortness free high strength structural steel.

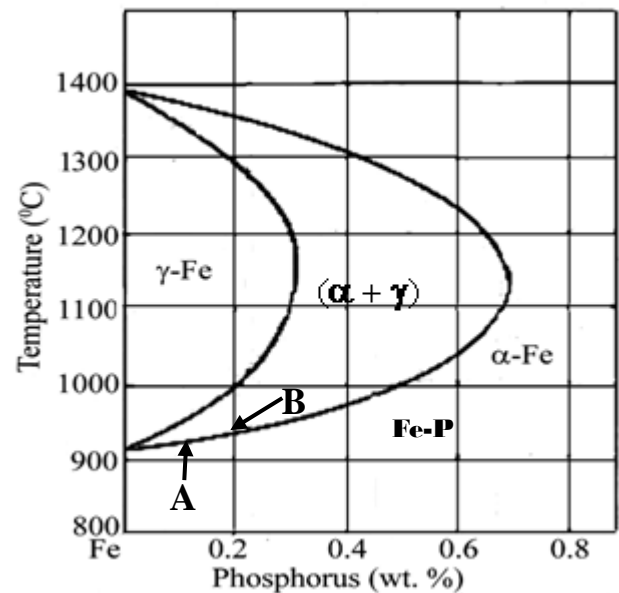


Fig 8. Fe-P binary phase diagram.

#### 4. CONCLUSIONS

In this work, effect of phosphorus additions on microstructures and tensile properties of commercial grade of pure iron by optimizing hot working process parameters such as hot working temperature, deformation rate and cooling rate after hot working have been studied. After detail experimental investigation, the following conclusions are made.

1. The detrimental effects of P in the iron alloys are possible to avoid by selecting relatively higher hot rolling temperature, increasing the rate of deformation during hot rolling and cooling rate after final pass of rolling.
2. Addition of P decreases the ferrite grain size and increases the tensile strength of the commercial grade of pure iron.
3. Gradual addition of P in pure iron has been found to increase the tensile strength with decrease in ductility. However, 0.21% P addition did not reduce the elongation level below the minimum level (e.g. 16%) required for structural applications.

## 6. REFERENCES

1. Mineral Information Institute, Website: [www.mii.org/Minerals/photoiron.html](http://www.mii.org/Minerals/photoiron.html).
2. N. Naudin, J.M. Frund and A. Pineau, "Intergranular fracture Stress and Phosphorus Grain Boundary Segregation of a Mn-Ni-Mo Steel", 1999, Scripta Materiala, 40:1013-1019.
3. M.A. Islam, J.F. Knott and P. Bowen, "Kinetics of phosphorus segregation and its effect on low temperature fracture behavior in 2.25Cr-1Mo pressure vessel steel" 2005, Journal of Materials Science and Technology, UK, 21(1):76-84.
4. M.A. Islam, P. Bowen and J.F. Knott, "Intergranular Fracture on Fatigue Fracture Surface of 2.25Cr-1Mo Steel at Room Temperature in Air", 2005, Journal of Materials Engineering and Performance, ASM International, 14(1):28-36.
5. M.A. Islam and Y. Tomota, "Investigation of Effects of Phosphorus on the Fatigue Life of Carbon Steels", 2010, International Conference of SPPM2010, held in Dhaka on 24-26 February, No.E19
6. M.A. Islam, T. Nemoto and Y. Tomota, "In-situ measurement of Fatigue Crack During Plane Bending Fatigue Test Without any Additional Set-up", 2010, International Conference of SPPM2010, held in Dhaka on 24-26 February, No.E19.
7. N.L. Zhenyu, Y. Qiu, L.X. Xiu and G. Wang, "Solidification Structure of Low Carbon Steel Strips with Different Phosphorus Contents Produced by Strip Casting", 2006, Journal of Materials Science and Technology, 22(6):755-58.
8. Y. Furuya, S. Matsuoka, S. Shimakura, T. Hanamura and S. Torizuka, "Effects of Carbon and Phosphorus Addition on the Fatigue Properties of Ultra fine-grained Steels", 2005, Scripta Materiala, 52(11): 1163-1167.
9. Key to Metals, "Effect of Phosphorus on the Properties of Carbon Steels: Part One". Web Address: [www.keytometal.com/](http://www.keytometal.com/)
10. N. Yashida, O. Umezawa and K. Nagai, "Analysis on Refinement of Columnar GAMMA Grain by Phosphorus in Continuously Cast 0.1 mass% Carbon Steel", 2004, Journal of ISIJ, 44(3):547-555.
11. M.P. Renavikar, "Small Strain Deformation Behaviours of Interstitial Free (IF) Steel", 1997, Ph.D. Thesis, University of Pittsburgh.