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Effects of Process Parameters on Ferrite Grain Size of Commercially Pure Iron

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Abstract

Besides the tremendous development of various materials, e.g. polymeric, ceramic, composites materials, etc. ferrous material is still the most important engineering material because of its many favorable properties. For ferrous alloys, grain size is very important as it directly influences the physical, chemical and mechanical properties of the alloys. In this study the effects of various process parameters such as annealing temperature, holding time period during annealing and phosphorus (P) additions (0.11% and 0.21%) have been investigated. After annealing heat treatments at different temperatures for various time periods, the alloys were etched in nital to reveal the ferrite grain sizes and they were then observed in the optical and scanning electron microscope (SEM). Experimental results revealed that annealing temperatures were more dominating over annealing time periods to control ferrite grain sizes and that after 0.11% P, more addition of phosphorus (e.g. 0.21%) was not found to influence ferrite grain sizes significantly under any annealing condition practiced in this study.

Keywords: Pure iron; Fe-phosphorus alloys; Annealing; Ferrite grain; Grain refinement.

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1. Introduction

Reversible temper embrittlement has been frequently observed in many different low alloy steels serving at high temperatures, e.g. order of 500°C or during slow cooling from high temperature. This type of embrittlement might change the brittle transgranular fracture mode to intergranular one with subsequence deterioration in fracture stress and fracture toughness [1-6]. In many cases, phosphorus plays an important role for inducing this type of unwanted effect in low alloy steels. So, in general, phosphorus is considered as a detrimental element in low alloy steels. For interstitial free or low carbon structural steel, it also has a tendency to segregate at grain boundaries during cooling period after

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hot working and makes the steel brittle [7-9]. If the hot rolled steel is cold worked for special properties, e.g. good surface finish, better dimensional tolerances and/or high strength the mechanical properties, especially fatigue properties of the cold worked steels are deteriorated severely [10-11].

Phosphorus, in general, induces temper embrittlement and degrades the mechanical properties of steels in many cases. However, careful control of thermomechanical treatment (e.g. annealing temperature, annealing time period, rate of deformation etc.), phosphorus in steel might be beneficial in many cases. As per Hall-Petch relationship given below, refinement in grain sizes means heightening tensile strength of the steel.

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

where σ_o is yield strength, σ_i is friction stress, K is locking parameter and D is the grain diameter. Addition of small amount of phosphorus decreases the prior austenite as well as the ferrite-pearlite grain sizes in steels. It also increases the strength of steel by solid solution strengthening mechanism [12, 13]. In order to improve the corrosion resistance of thermomechanically treated (TMT) structural steels used for reinforcement of concrete, P added TMT (TMT-P) steel has been developed [14-17]. With the better understanding of the physical metallurgy of Fe-P interaction, it seems that P is gradually becoming to be a very important element for ferrous alloy, especially for steels. The aim of this present paper is to discuss the individual and combined effects of annealing temperature, annealing time period and phosphorus addition on the ferrite grain size of commercially pure iron.

2. Experimental Procedures

The materials used in this work were commercial grade of pure iron and phosphorus added pure irons (0.11% P: Fe-P-I and 0.21% P: Fe-P-II). The chemical compositions of these three materials are presented in Table 1. These steels were melted in vacuum by induction melting and forged at National Institute for Materials Science (NIMS), Tsukuba, Japan. In order to change the grain sizes of these test materials, they all were annealed at different temperatures such as 700, 800 and 900°C. All materials were annealed for three different time periods as 5 minutes, 1 hour and 10 hours at 700°C. However, for annealing at 800 and 900°C only one hour annealing period was used.

Table 1. Chemical compositions of the iron and iron-phosphorus alloys used.

Material identification	C (%)	Si (%)	Mn (%)	P (%)	S (%)
Pure iron	0.003	< 0.001	< 0.001	0.001	0.0011
Fe-P-I	0.003	< 0.001	< 0.001	0.110	0.017

Fe-P-II	0.003	< 0.001	< 0.001	0.210	0.005

After annealing, metallographic samples from all materials were prepared and etched with 5% nital (5% nitric acid and 95% ethanol) following standard procedure. They were then observed under a scanning electron microscope (SEM) to identify the microstructures and establish the grain sizes. The average ferrite grain sizes of all annealed steels were measured and presented in Table 2.

Table 2. Average ferrite grain sizes of the alloys after annealing.

Material identification	Annealing temperature and Time periods				
	700^{o}C/5min	700^{o}C/1h	700^{o}C/10h	800^{o}C/1h	900^{o}C/1h
Pure iron	35	40	45	58	83
Fe-P-I	17	18	22	27	42
Fe-P-II	15	16	19	24	40

3. Results and Discussion

3.1. *Effect of annealing time periods*

It has been mentioned that, for all three steels, annealing treatment was carried out for different time periods as 5min, 1h and 10h only at 700^{o}C. For all annealed steels of different conditions, it has been found that annealing time periods resulted in a very minor effect on the ferrite grain sizes, which is very clear from Table 2. For pure iron, annealing for 5min, 1h and 10h at 700^{o}C resulted in, respectively ferrite grain sizes of 35, 40 and 45 µm. For Fe-P-I alloy, they were respectively 17, 17 and 22 µm. Similarly for Fe-P-II, they were, respectively 15, 16 and 19 µm. Micrographs of ferritic structures of different alloys annealed for different conditions are shown in Figs. 1-6.

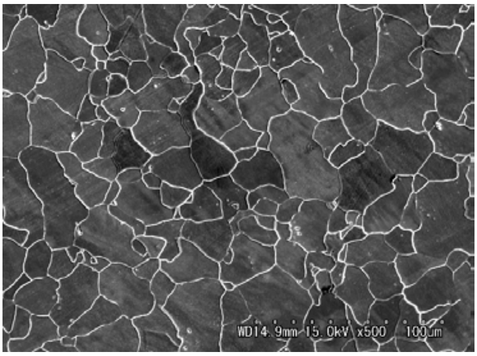

Fig. 1. Microstructure of pure iron annealed at 700^{o}C for 1 hour.

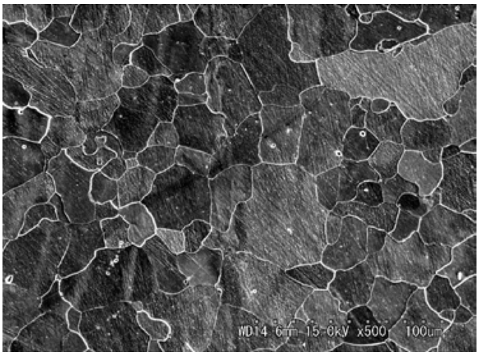

Fig. 2. Microstructure of pure iron annealed at 700^{o}C for 10 hour.

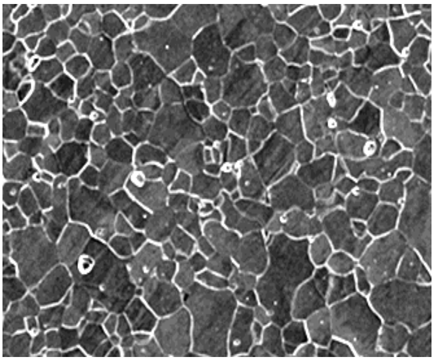

Fig. 3. Microstructure of Fe-P-I annealed at 700°C for 1hour.

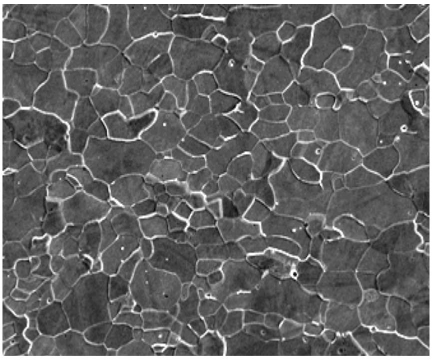

Fig. 4. Microstructure of Fe-P-I annealed at 700°C for 10 hour.

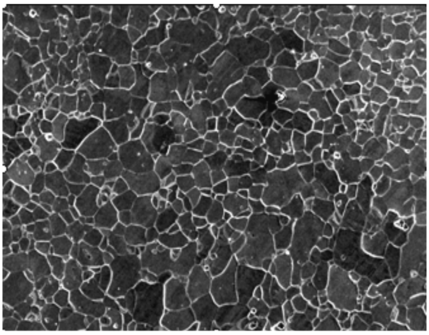

Fig. 5. Microstructure of Fe-P-II annealed at 700°C for 1 hour.

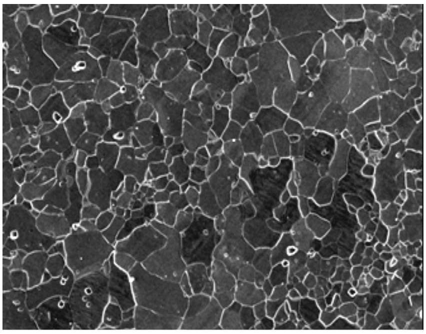

Fig. 6. Microstructure of Fe-P-II annealed at 700°C for 10 hour.

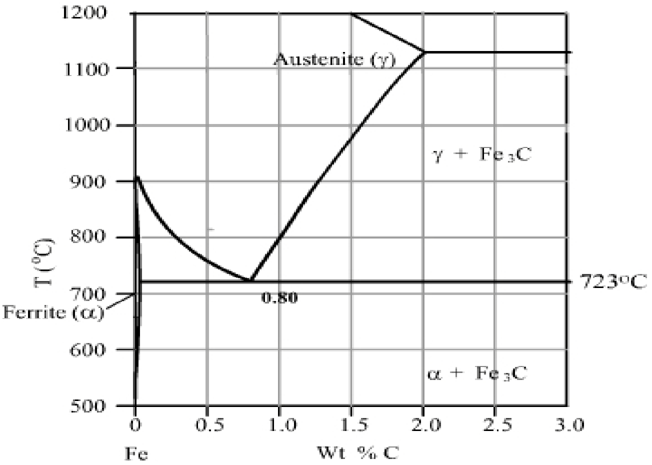

Fig. 7. Part of iron-carbon system.

From the partial form of iron-carbon diagram presented in Fig. 7, we can see that before 910°C ferrite phase of pure iron does not change to gamma (γ) or austenite. In the case of commercial grade of pure iron used in this present study, there is 0.003% carbon. So, the ferrite (α) transformation temperature to austenite might be reduced to some extent. However, 700°C temperature is far way from its transformation temperature. As a result, during holding at 700°C the only possibility of grain coarsening is through merging the ferrite grains to each other. Merging of ferrite grain size is controlled by solid state diffusion rate of iron, which is a very slow process. Because of slow diffusion rate, only some fine grained ferrites get chance to be merged fully. On the other hand, incomplete merging takes place for coarse grained ferrite, by which some coarse grained ferrites are formed with subsequent formation of fine ferrite grains also. This means that there is possibility to form some new fine grained ferrite. So, at 700°C no significant change in the average ferrite grain sizes took place.

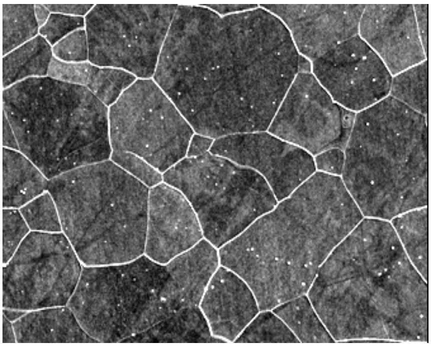

Fig. 8. Microstructure of pure iron annealed at 800°C for 1 hour.

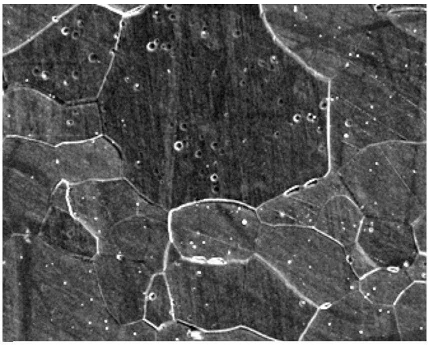

Fig. 9. Microstructure of pure iron annealed at 900°C for 1 hour.

3.2. *Effect of annealing temperatures*

From Table 2, it is very clear that compared to annealing time periods, annealing temperature resulted more influence on the ferrite grain sizes, which is true for all three iron alloys. For pure iron, one hour annealing at 700°C resulted in ferrite grain size of 40µm, which were respectively 58 and 83µm for annealing at 800°C and 900°C for the same annealing time period. For other two steels, similar results were also found, Table 2. Significant increase in ferrite grain sizes with annealing temperature is very clear from Figs. 8 to 13. It has been mentioned that solid state diffusion rate of iron is a slow process. However, with increase in temperature the diffusion rate increases significantly [18]. In the case of annealing at 800°C, two mechanisms are operative. First, fine grained ferrites merged with each other more quickly and for coarse grained ferrites, degree of partial merging will be better. Some ferrite grains with higher carbon content (if they exist), due to non-homogeneities in carbon distribution ferrite grains with higher carbon content might also transform to austenite and resulted in new ferrites during cooling cycle. As the specimens were cooled slowly in the furnace, they got sufficient time to be merged at relatively higher temperature. So, altogether, significant increase in the ferrite grain sizes took place. For annealing treatment at 900°C, arguably more initial ferrites were transformed to austenite. During holding at this high temperature austenite grains were coarsened. In the case of coarse grained austenite limited numbers of ferrite nuclei are formed during cooling through recrystallization process. During slow cooling period, they all grew and resulted in coarse grained ferrites.

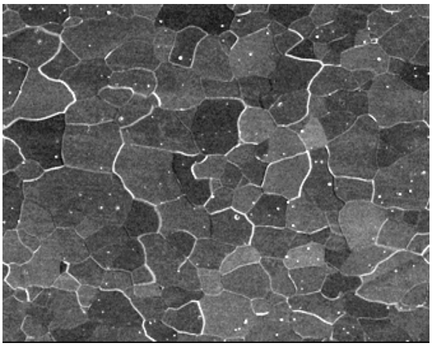

Fig. 10. Microstructure of Fe-P-I annealed at 800°C for 1 hour.

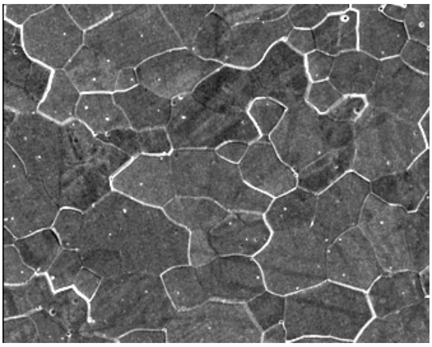

Fig. 11. Microstructure of Fe-P-I annealed at 900°C for 1 hour.

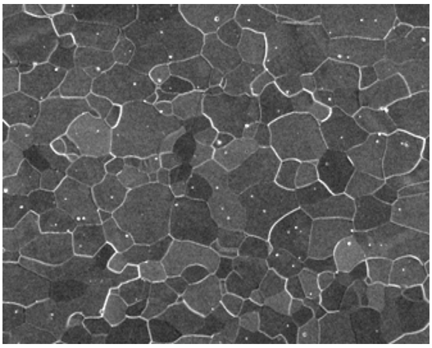

Fig. 12. Microstructure of Fe-P-II annealed at 800°C for 1 hour.

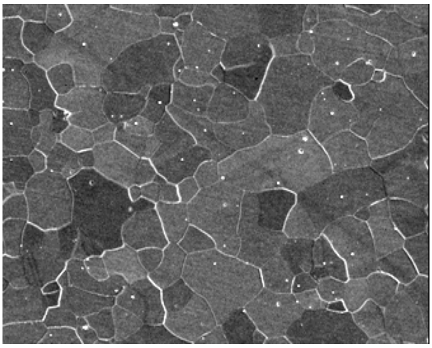

Fig. 13. Microstructure of Fe-P-II annealed at 900°C for 1 hour.

3.3. *Effect of phosphorus content*

Addition of 0.11% phosphorus in pure iron significantly reduced the ferrite grain sizes. However, more phosphorus (e.g. 0.21%) addition after 0.11% did not show significant change in grain sizes (Table 2).

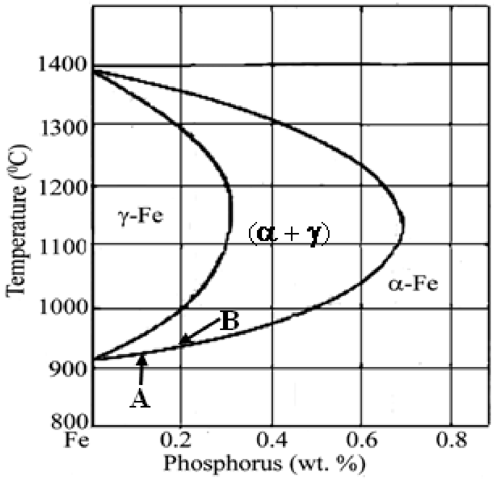

Fig. 14. Fe-P binary phase diagram.

From Fe-P binary phase diagram presented in Fig. 14, it is evident that phosphorus is a ferrite stabilizer (note: in the diagram α denotes ferrite and γ denotes austenite). It retards the transformation of ferrite to austenite during heating period. On the other hand, during cooling period after solidification, austenite grains could not grow sufficiently because of their early transformation to ferrite. As a result, large numbers of ferrite grains were formed in P-added iron. So, both Fe-P alloys showed relatively finer ferrite grains. Compared to Fe-P-I alloy, the P content in Fe-P-II alloy is nearly double. However, no significant refinement in ferrite grain sizes was observed. Similar observation has also been mentioned by other [12, 13, 19]. The possible reason is that the line connecting line of the lower critical temperature points of Fe-P alloys containing P between 0.1 (point A) and 0.2% (point B) is almost flat. This means lower critical temperatures of Fe-P-I and Fe-P-II alloys were almost very similar. Fe-P binary phase diagram indicates that between 0.3 and 0.6% P, the lower critical temperature curve is relatively steeper rather than flat like that for Fe-P alloys containing 0.1 and 0.2% P, where more refinement of ferrite grains is expected. In this present work, 900°C was considered as the maximum annealing temperature. As per the Fe-P binary phase diagram in Fig.14, during annealing no or a very limited number of ferrite grains were transformed to austenite. So, the finer ferrite grains in Fe-P alloys are due to their initial finer ferrites. As higher temperature makes the finer ferrites to merge with each other much more quickly, so higher temperature annealing resulted in coarser ferrite grains.

4. Conclusions

In this research work the effects of annealing temperature, time period and phosphorus additions on ferrite grain refinement have been investigated. From the experimental results of this research work it is revealed that, for all ferrous alloys considered for this research, increase in the annealing time periods resulted in a very minor change in the ferrite grain size. However, increase in the annealing temperature caused a significant ferrite grain coarsening effect. Addition of 0.11% phosphorus decreased the average ferrite grain sizes of the commercially pure iron by more than 50%. However, further increase in the phosphorus content (to double; 0.21% in the place of 0.11%) resulted in a very minor effect on the refining the ferrite grain sizes.

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