

Microstructural evolution in Fe-0.13P-0.05C steel during compression at elevated temperatures

Mehta Y*, Rajput S K, Chaudhari G P and Dabhade V V

Department of Metallurgical & Materials Engineering, IIT Roorkee, India 247667

*Corresponding author E-mail: yashwant.mehta@gmail.com

Abstract. The microstructural evolution was studied in order to adjust the processing parameters for hot forming. Fe-0.13P-0.05C steel was subjected to hot compression tests using a thermo-mechanical simulator. The tests were performed at the temperatures ranging from 800°C-950°C. The strain rates chosen at all these temperatures were 0.01, 0.1 and 1 s⁻¹. The effects of the strain rates and hot compression temperatures on the microstructural aspects of the steel were examined using optical microscopy. The outcomes indicate that the mean grain dimension of the hot compressed Fe-0.13P-0.05C steel escalates with increases in the deformation temperature and also with decreases in strain rate. Dynamic recrystallization was observed to be the instrument of grain refinement. The minimum grain dimension of 5.6 µm was attained at 800°C and 0.1s⁻¹.

1. Introduction

The engineers who decide the processes of metal forming i.e., forging, hot rolling, extrusion, etc, are constantly trying to achieve progress in the quality of the metal based goods. Thus, there is need to develop an understanding about the relationships between the thermo-mechanical parameters and microstructural evolution of alloys. The study of microstructural evolution during the hot deformation allows the designers to understand the kinetics of metallurgical transformations [1].

Phosphorous was used extensively as an alloying element in the pre-industrial age. It helps the formation of a passive layer in the wet-dry environmental conditions. This passive layer protects the alloy product from further degradation. The Delhi Iron Pillar has withstood atmospheric corrosion for centuries [2], [3].

Although a lot of metallographic studies [4]–[9] have been conducted on hot compressed specimens of low carbon steels but studies on high phosphorous alloys are scarce. In this paper, an iron alloy having 0.13 wt.% phosphorous and 0.05 wt.% carbon was cast, and its microstructural evolution was studied. Specimens of the alloy were hot compressed at different processing conditions on Gleeble® 3800. The influence of strain rates and temperatures on grain size was also studied.

2. Experimental technique

High phosphorous steel was prepared by using the induction furnace and sand casting route. Iron scrap, ferro-manganese, ferro-silicon, ferro-phosphorous, and aluminium shots were melted in the furnace and cast at the appropriate time. The casting was sliced into small pieces, which were subsequently hot forged manually to make 12 mm rods. These rods were further machined on a lathe machine to make specimens of length 15 mm and 10 mm diameter. Specimens were hot compressed in Gleeble® 3800 thermo-mechanical simulator. The specimens were hot compressed at temperatures of 800°C, 850°C, 900°C and 950°C up to true strain of 0.7 with the strain rates of 0.01, 0.1 and 1 s⁻¹, after austenitization at 1050°C. A thermocouple (K type) was spot welded in the centre of the specimen to measure the temperature and control it. The specimens were heated to 1050°C with the heating speed of 5°C s⁻¹ under a vacuum of 1 Pa to transform it to austenite. The specimens were



permitted to hold for 10 s at 1050°C. Subsequently, the specimens were allowed to cool at 1°C s⁻¹ to the deformation temperatures, followed by water quenching to preserve the microstructure. A graphite foil and a nickel based lubricant were used to reduce friction and ensure good thermal conductivity between the samples and the ISO-T anvil.

The phase transformation temperatures A_{r3} and A_{r1} were determined using continuous cooling transformation (CCT) curves. The sample used for the CCT investigation is cylindrical having 85mm length and 10 mm diameter. It was heated to 1050°C at 5°C s⁻¹ and then allowed to soak for 10 s before being cooled to 25°C at 1°C s⁻¹. During cooling the initial and final deviancy indicated the A_{r3} and A_{r1} temperatures, respectively.

The hot compression samples were sliced in the centre at the axis of compression, polished with different grades of polishing papers and velvet cloth, and then subjected to etching with 2 % nital reagent. The microstructures were viewed with an optical microscope (Leica DMI 5000M). The grain dimension was found out using the ASTM line intercept technique.

3. Results and Discussion

The iron phosphorous phase diagram has a high temperature loop. Phosphorous is a ferrite stabilizer. Figure 1 shows a schematic of the γ loop of the Fe-P phase diagram.

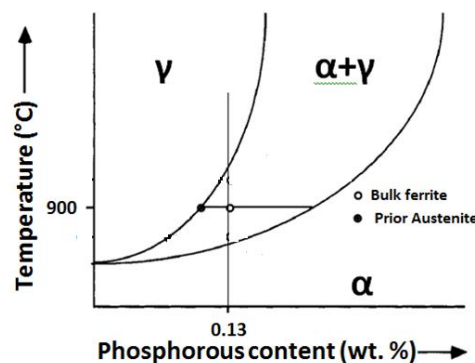


Figure 1. Representation showing the γ loop of Fe-P phase chart (adapted from [10])

Table 1 displays the composition of the steel which was determined using a MAGELLAN optical emission spectrometer at Vaishnav Steel Private Limited, Muzaffernagar, Uttar Pradesh India. An average of two readings is presented.

Table 1. Composition of the steel

Alloy	P	C	Si	Mn	Cr	Al	Cu	N	Fe
S1	0.13	0.05	0.26	0.2	0.13	0.003	0.023	0.004	99.2

Table 2 shows the transformation temperatures of the material which were ascertained by conducting dilatometry studies on a Gleeble 3800® thermo-mechanical simulator.

Table 2. Transformation temperatures of S1 steel determined by dilatometry studies

Transformation Temperature	S1
A_{r1} (°C)	790
A_{r3} (°C)	965

Figure 2 shows the microstructures recorded using optical microscopy. At greater temperatures and/or at lesser strain rates the grains become equiaxed. Higher degree of dynamic recrystallization

causes such behaviour. However, at lesser temperatures and greater strain rates many grains appear flattened since they could not recrystallize, and were compressed. The phases present are ferrite (white colour) and a small amount of pearlite (dark colour).

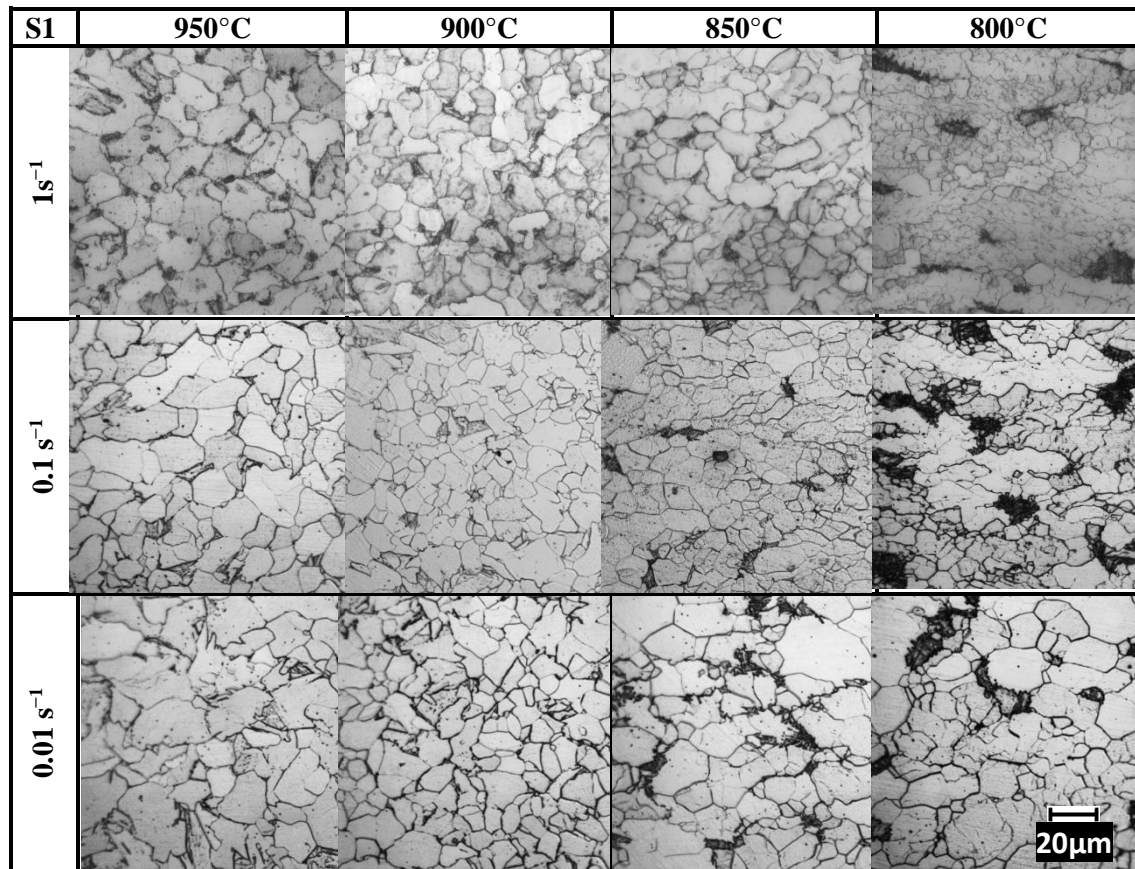


Figure 2. Microstructures of deformed specimens of S1 steel at different strain rates and temperatures.

During hot deformation, the temperatures can rise adiabatically. The maximum increment in the temperatures with strain rates has been presented at table 3. The microstructures should be considered to be produced at the temperature of testing plus the increment in temperature during the test. For example the first microstructure in figure 2 has been produced at 955°C. Thus the grain size of the microstructure corresponds to the deformation temperature of 955°C.

Figure 3 shows the flow curves of high phosphorous steel hot compressed at the test temperatures of 800°C, 850°C, 900°C and 950°C, and at the strain rates of 0.01, 0.1, and 1 s⁻¹. The flow curve varies as a function of both temperature and strain rate. At low strain rates, flow curves displayed a sequence of initial hardening with a subsequent peak stress. For fixed strain rate, the flow stress values first diminished then augmented, and finally diminished with an increase in temperatures. This occurs due to the variation in the hardness of α which is lesser at higher temperatures than that of γ [11]. The flow curve at strain rate of 1 s⁻¹ and 900°C shows dynamic recovery. Multiple dynamic recrystallization peaks can be seen at strain rate of 0.01 s⁻¹ and 950°C as novel grains propagate at the cost of strain hardened past grains. At constant temperature, stress peaks shift towards higher strains with the increase in strain rate. Comparing flow stress at 850°C & 0.1s⁻¹ with that obtained by Tsuji and co-workers [12], it can be said that the addition of phosphorous to low C steel retards dynamic recrystallization.

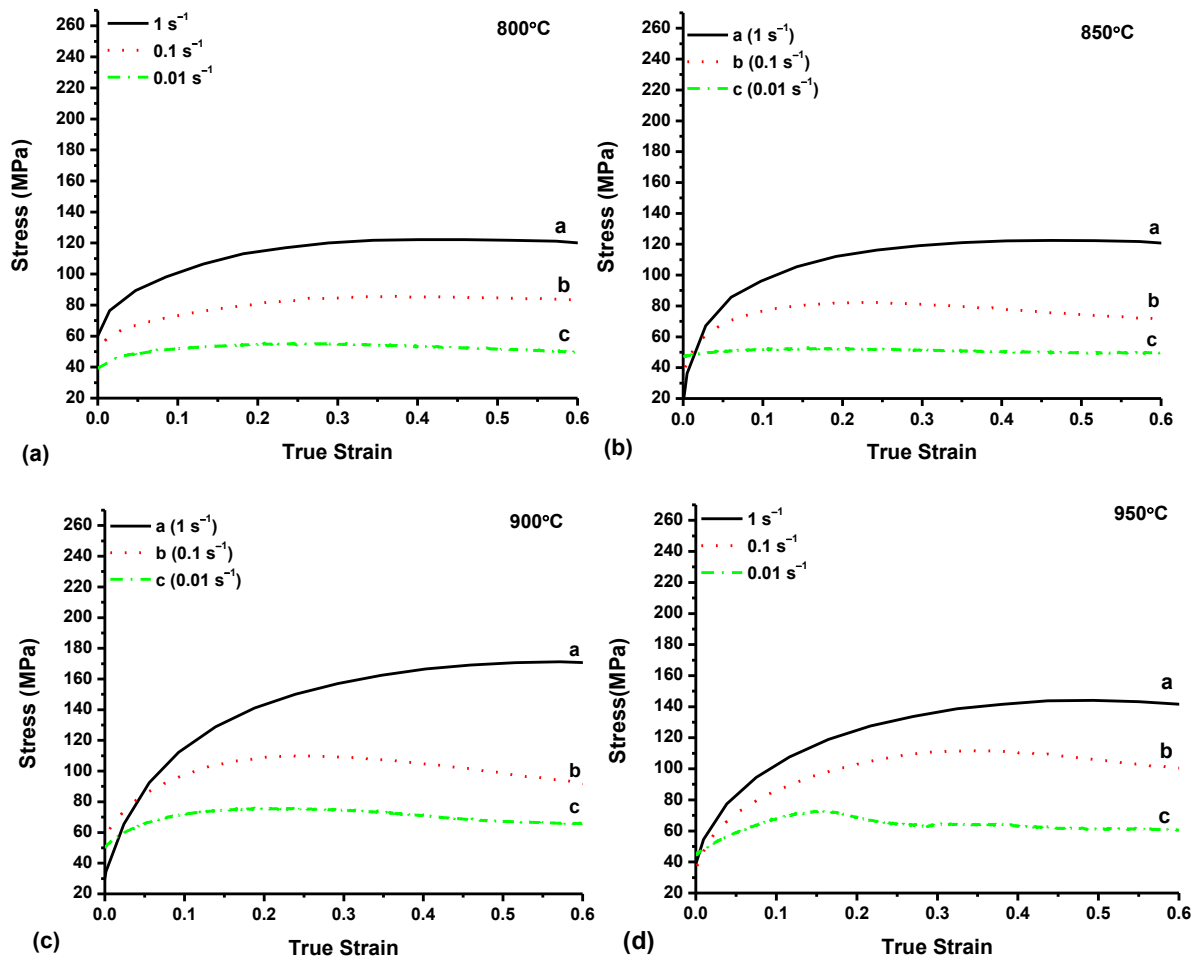


Figure 3. Flow curves of S1 steel hot compressed at (a) 800°C, (b) 850°C, (c) 900°C and (d) 950°C, using different strain rates.

Table 3. Highest augmentation in the temperature for the duration of testing of S1

Deformation Temperature (°C)	<i>dT</i> (°C) at the strain rate of		
	0.01 s ⁻¹	0.1 s ⁻¹	1 s ⁻¹
800	3.8	6.1	14.3
850	1.2	7.2	16.6
900	2.7	5.4	20.7
950	5.8	4.8	8.5

3.1 Influence of the test temperature on microstructure and grain dimension

Figure 4 shows the variation in grain dimension with the modification in temperature and strain rate. The grains at test temperature of 950°C are equiaxed which indicates that they have been obtained after dynamic recrystallization, which was also depicted in figure 3(d). Since the temperature is high, a certain amount of grain growth is also expected. At deformation temperature 900°C, the grains are equiaxed and smaller than those produced at 950°C, because the amount of thermal energy available for diffusion has been diminished. At deformation temperature 850°C, a mix of equiaxed and

elongated or flattened grains are observed. The equiaxed grains are smaller than those obtained at higher temperatures. The degree of dynamic recrystallization depends on the temperature and higher at higher temperatures. The grains found at deformation temperature 800°C show that the dynamic recrystallization has occurred around the larger grains, forming a necklace type structure [1].

In general, the grain size decreases as the deformation temperature is lowered. This is due to the fact that the grain growth phenomenon is more pronounced at higher temperatures. The only exception to this generality, in the present work is the grain size observed at 0.01s^{-1} and deformation temperature 900°C. This is slightly lower than that found at test temperature of 850°C. This might be due to the incidence of large pro-eutectoid α grains observed in the microstructure.

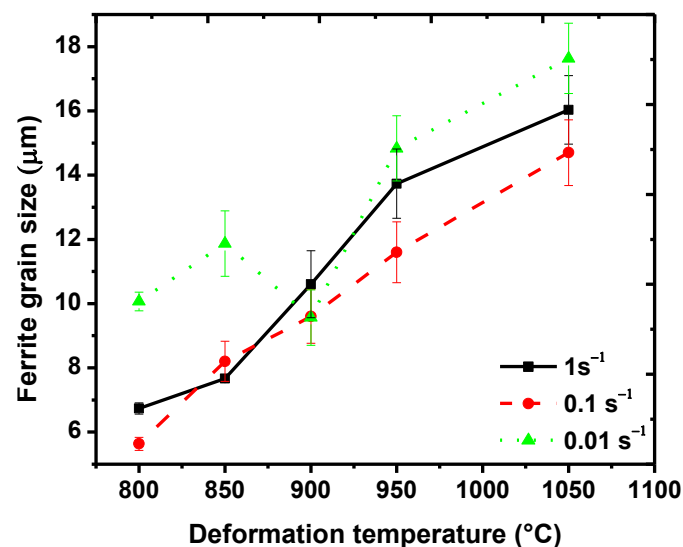


Figure 4. Influence of strain rates & temperatures on the α grain size of S1 steel

3.2 Influence of strain rate on the microstructure and grain dimension

Restoration phenomenon (DRX, DRV) rate declines with the rise in strain rate [1]. At higher temperatures, more deformation energy is available for facilitating recovery and recrystallization. Therefore, at lower temperature and higher strain rates more sub-structures should be formed in the original grains. This will give rise to more nuclei per unit volume of the grains which causes the formation of smaller grains. At lower strain rates and higher temperatures, the grains observed are of larger size, this is due to the more time available during deformation. The grains observed in the present work at a strain rate of 0.01s^{-1} are bigger than those obtained at 0.1s^{-1} and 1s^{-1} strain rates. The grains obtained at strain rate of 1s^{-1} are somewhat bigger than those obtained at strain rate of 0.1s^{-1} . This could be ascribed to the larger values of adiabatic rise in temperatures during the hot compression at strain rate of 1s^{-1} as recorded in table 2.

4. Conclusions

The influence of strain rate and temperature were studied by hot compression for high phosphorous steel (S1). It was observed that generally, the grain sizes decreased with an escalation in strain rate and a reduction in test temperature. The adiabatic rise in temperature caused deviations from the general trend.

5. References

- [1] Y. C. Lin, M. Chen, and J. Zhong, "Micro structural evolution in 42CrMo steel during compression at elevated temperatures," *Mater. Lett.*, vol. 62, pp. 2132–2135, 2008.
- [2] R. Balasubramaniam, "On the corrosion resistance of the Delhi iron pillar," *Corros. Sci.*, vol. 42, no. 12, pp. 2013–2129, 2000.

- [3] R. Balasubramaniam, *Delhi iron pillar—New insights*. Shimla: Indian Institute of Advanced Study, 2002.
- [4] S. K. Rajput, G. P. Chaudhari, and S. K. Nath, “Characterization of hot deformation behavior of a low carbon steel using processing maps , constitutive equations and Zener-Hollomon parameter,” *J. Mater. Process. Technol.*, vol. 237, pp. 113–125, 2016.
- [5] S. K. Rajput, M. Dikovits, G. P. Chaudhari, C. Poletti, F. Warchomicka, V. Pancholi, and S. K. Nath, “Physical simulation of hot deformation and microstructural evolution of AISI 1016 steel using processing maps,” *Mater. Sci. Eng. A*, vol. 587, pp. 291–300, 2013.
- [6] S. K. Rajput, G. P. Chaudhari, and S. K. Nath, “Physical simulation of hot deformation of low-carbon Ti-Nb microalloyed steel and microstructural studies,” *J. Mater. Eng. Perform.*, vol. 23, no. 8, pp. 2930–2942, 2014.
- [7] B. Eghbali and A. Abdollah-zadeh, “The influence of thermomechanical parameters in ferrite grain refinement in a low carbon Nb-microalloyed steel,” *Scr. Mater.*, vol. 53, pp. 41–45, 2005.
- [8] B. Eghbali and A. Abdollah-zadeh, “Effect of strain rate on the ferrite grain refinement in a low carbon Nb-Ti microalloyed steel during low temperature deformation,” *J. Mater. Sci. Tech.*, vol. 21, no. 6, pp. 851–855, 2005.
- [9] B. Eghbali and A. Abdollah-zadeh, “Influence of deformation temperature on the ferrite grain refinement in a low carbon Nb – Ti microalloyed steel,” *J. Mater. Process. Technol.*, vol. 180, pp. 44–48, 2006.
- [10] R. Vogel, “On the system iron-phosphorus-carbon,” *Arch Eisenhüttenwes*, vol. 3, pp. 369–81, 1929.
- [11] K. B. Gove and J. A. Charles, “The high temperature hardness of various phases in steel,” *Met. Technol.*, no. June, pp. 279–283, 1974.
- [12] N. Tsuji, Y. Matsubara, and Y. Saito, “Dynamic recrystallization of ferrite in interstitial free steel,” *Scr. Mater.*, vol. 37, no. 4, pp. 477–484, 1997.