Effect of austenitizing temperature on microstructure formation of quenched X65 micro-alloyed pipeline steel

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Abstract. The effect of austenitizing temperature on the microstructure formation in micro-alloyed X65 pipeline steel was investigated using an optical microscope (OM), transmission electron microscope (TEM) and microhardness measurement. The results show that the austenite grains coarsening temperature of X65 steel reaches 1100°C. When austenitization is carried out below 1000°C, a large number of fine microalloying carbides precipitate during quenching within some α ferrite laths, with a uniform orientation. With austenitizing temperature increasing, the amount of precipitated carbides increases. Such fine carbide precipitates play significant strengthening for the steel. When the austenitizing temperature exceeds 1000°C, no carbide precipitation occurs in α ferrite laths, because microalloying solute atoms have too low mobility to precipitate under such conditions. This results in that X65 steel exhibits the maximum microhardness when quenched from 1000°C.

1. Introduction

Low carbon micro-alloyed high-strength pipeline steel has been recognized as a preferable material and widely used for transporting oil and gas in the deep-water environment, due to its high strength-toughness combination [1-3]. Its excellent mechanical performance results mainly from the grain refinement and precipitation strengthening through the formation of fine and highly stable particles of microalloying carbides. Grain refinement is principally achieved from the fine carbide particles strain induced in austenite during hot deformation, which can inhibit austenite grain growth during reheating and retard the recrystallization of austenite. The particles responsible for precipitation strengthening mainly precipitate during or after γ→α phase transformation on the γ/α interface or in the matrix of ferrite [4-8].

At present, quenching-tempering treatment after rolling is commonly applied in the production of micro-alloyed pipeline steel to obtain optimum comprehensive mechanical properties by grain refining and precipitation hardening. Thus, it is necessary and quite essential to design optimal parameters for the technology of quenching-tempering treatment (QTT). Reheating is the first critical step for QTT because the initial state of prior austenite obtained in this process can influence the development of final microstructure significantly. Many previous studies have been done on the relationship between reheating temperature and the final microstructures after quenching to room temperature, and it was shown that the reheating temperature influences the microstructures and properties of steels mainly through its effect on the size of prior austenite grains [9-11]. For steels with additions of microalloying elements like niobium, vanadium, and titanium, fine sparingly soluble precipitates can efficiently inhibit
austenite grain growth at relatively low temperatures. As heating temperature increases to some level, microalloying carbide particles completely dissolve into austenite, and afterward, sudden abnormal grain growth occurs. Therefore, aiming to obtain fine austenite grains, the high heating temperature is usually avoided during QTT. In previous studies, few researchers paid much attention to the influence of austenitization temperature on the behavior of $\gamma \rightarrow \alpha$ phase transformations during the cooling process to room temperature from the austenite region. However, for micro-alloyed steels, the reheating temperature is a significant factor responsible for the dissolution extent of microalloying atoms into austenite and thus would affect the precipitation behavior of carbide particles as phase transformation occurs in the continuous cooling process.

In the present work, the investigation was carried out on how austenitizing temperature influence the produced microstructure after quenching to room temperature from austenite, using X65 steel, typical micro-alloyed pipeline steel. TEM observations were performed on the obtained microstructures, and furtherly the results were confirmed by microhardness measurement. It is expected to provide a helpful theoretical basis for optimizing the technological parameters for quenching-tempering treatment.

2. Experimental materials and procedures
The nominal composition of the investigated X65 pipeline steel is (in weight percent): 0.09C, 1.29Mn, 0.29Si, 0.15Ni, 0.18Mo, 0.06Cr, 0.034Al, 0.13Cu, 0.05V, 0.03Nb and 0.001Ti. Heat treatments were conducted in a Muffle furnace with a thermocouple for detecting the temperature. The samples with a dimension of 10mm×10 mm × 10mm were mechanically cut from an X65 hot-rolled steel pipe. To determine the effect of austenitizing temperature on the microstructure, the samples were heated to eight different austenitization temperatures respectively (850, 900, 950, 1000, 1050, 1100, 1150, and 1200°C), held for 10 min, and then quenched into the water at room temperature. Heat-treated samples were subjected to microstructural examination via an OLYMPUS optical microscope and JEM-100CX II transmission electron microscope. Prior austenite grain boundaries were displayed by etching metallographic specimens with a saturated picric acid solution after mechanical polishing. Austenite grains sizes were determined by mean linear intercept method. Foil specimens for TEM were prepared by mechanical grinding to about 100µm in thickness, followed by dual jet electrolytic polishing using an electrolyte of 10% perchloric acid and 90% ethanol. To determine the hardness of the quenched samples, microhardness measurements were performed on a Vickers microhardness tester, using a load of 200g.

3. Results and Discussion

3.1. Austenite grain size
Figure 1 presents the typical optical micrographs of X65 pipeline steel specimens quenched from different austenitizing temperatures, revealing prior austenite grain boundaries. With the austenitizing temperature increased from 900°C to 1200°C, the size of austenite grains grows markedly. The average sizes of austenite grains of the specimens were measured, and the results are shown in figure 2, as a function of austenitizing temperature. As can be seen, austenite grains grow slightly (from 10 to 20µm) in relatively low-temperature range (850~1050°C), and then a sudden grain coarsening occurs (above 1100°C), up to 110µm as the austenitizing temperature increased to 1200°C. Thus the critical grain coarsening temperature of this steel is about 1050°C. The weight percent of Nb dissolved in austenite at various austenitizing temperatures can be calculated according to the following empirical equation (1), and then the undissolved Nb weight percent were also obtained [12]. The calculation results were plotted in figure 2, together with the austenite grain size values:

$$\log_{10}[\text{Nb}] = 2.26 \left( \frac{12}{14} \right) C - \frac{6770}{T} + 2.26$$

(1)
Figure 1. Typical optical micrographs displaying prior austenite grains in X65 pipeline steel specimens austenitized at different temperatures for 10min, (a) 900°C, (b) 1000°C, (c) 1100°C, (d) 1200°C

Figure 2. The average size of prior austenite grains and undissolved content of Nb in X65 pipeline steel specimens, as a function of austenitization temperature.

From figure 2, the undissolved Nb content decreases monotonously with increasing austenitizing temperature. It is noted that the decrease ratio below 1050 °C is lower than that above 1050 °C. At 1150°C, all added Nb dissolved into austenite. At temperatures below 1050°C, more than half of the added Nb keeps out of solution and exists in the form of carbide particles, which can contribute to restraining austenite growth. Therefore, austenite grains keep slow growth as the austenitizing temperature does not exceed 1050°C. This indicates that the percent of undissolved micro-alloy carbide particles is a critical factor for prior austenite grains size.
TEM microstructural examinations were carried out on the specimens quenched from different austenitizing temperatures, as shown in Figure 3. It was found that the produced microstructure consists of aggregates of α ferrite laths, parallel to each other. A high density of dislocations exists within the α ferrite laths. Some adjacent α ferrite clusters angled each other at about 60°, as shown in figure 3b. The widths of α ferrite laths are not uniform, in the range of 0.1–1μm. Detailed observations indicate that a lot of fine needle-like particles are distributed in relatively wide α ferrite laths. It is also noted that such wide laths with the distribution of fine particles can only be observed in the specimens austenitized below 1000°C. No fine precipitates were found within the α ferrite laths in the specimens austenitized at 1100 and 1200°C. Figure 3c is a micrograph with higher magnification, in which the particles are shown more clearly. The needle-like particles are about 100 nm in length, and most of them are distributed in a common direction. This indicates that the carbide particles are precipitated coherently from the parent austenite, but not like the auto-tempering process of martensite. Precipitates in auto-tempered martensite are usually round in shape and without preferred orientation [13]. Previous studies also proved these oriented carbide particles to be M₃C phase [14]. The formation of coherent interfaces between M₃C precipitates and austenite matrix phase can reduce the interfacial nucleation energy. These fine carbide particles contribute much to the strengthening of the steel, named precipitation hardening. It should be noted that such fine precipitates were not observed in the specimens austenitized at higher temperatures (1100 and 1200°C), as shown in figure 3d.

![Figure 3](attachment:tem_images.png)

Figure 3. TEM images of water-quenched X65 pipeline steel specimens austenitized at different temperatures for 10min, (a) 900°C, (b) 950°C, (c) 1000°C, (d) 1100°C

### 3.2. Microhardness

Figure 4 shows the Vickers microhardness results of the quenched specimens from different austenitized temperatures. It was shown that the microhardness value (HV) increases rapidly with austenitizing temperature increasing from 900 to 1000°C, and then decreases significantly. The specimen austenitized at 1000°C exhibits the maximum microhardness. It was known from figure 2 that austenite grains size increases monotonously with increasing austenitizing temperature. The width of α ferrite laths formed
within coarse austenite grains would be relatively wide and long, and the steel with such microstructure will exhibit low hardness. Thus, from the point of grains size, microhardness value should decrease monotonously with increasing austenitizing temperature. The significant decrease of microhardness as the austenitizing temperature exceeds 1000°C agrees with this case, as shown in figure 4. But the increasing trend of hardness value in a temperature range below 1000°C is in consistent with this and should be attributed to some other factor. With the TEM examination results being fully considered, the unusual increase below 1000°C should be attributed to the precipitation of fine carbide particles within α ferrite laths. In the process of quench cooling, micro-alloying solute atoms in prior austenite precipitate finely as carbides, which are uniformly distributed within lath-like α ferrite. These fine carbides supply significant precipitation strengthening for the steel. With the austenitizing temperature increasing, more micro-alloying carbides decompose, and more solutes go into austenite, and thus more fine needle-like precipitates form within α ferrite laths. Therefore, higher austenitizing temperature leads to higher microhardness. At the early stage that micro-alloying atoms enter the austenite matrix, they aggregate mainly at sites near dislocations. When precipitation occurs, the micro-alloying carbides nucleates preferentially at the dislocations. Once austenitizing temperature increases to a high enough extent, the micro-alloying atoms, such as Nb, Mo, have sufficient energy to leave dislocations and dissolve into austenite matrix stably. More time and energy are needed for the micro-alloying atoms stably existing in austenite matrix to diffuse and nucleate as carbides, and thus the solute atoms tend to keep within austenite after the specimen is quenched into water. This can explain why precipitated carbides were not observed in specimens austenitized at 1100 °C and above.

Figure 4. Vickers hardness values of water-quenched X65 pipeline steel specimens austenitized at different temperatures for 10min, with a load of 200g.

From the above analysis, it can be concluded that there exists an optimal quenching temperature for microalloyed steels, at which a lot of fine carbide particles can form within lathlike α ferrite and provide high strength for steels. The optimum austenitizing temperature for the present X65 pipeline steel is about 1000°C.

4. Conclusions
Austenitizing temperature has a considerable effect on the precipitation behavior of microalloying carbides. The investigated X65 microalloyed pipeline steel has a critical coarsening temperature of austenite grains of 1050°C. The quenched specimens subjected to austenitization at about 1000°C exhibit the maximum hardness, which is attributed to the significant precipitation of fine carbides at this temperature. If austenitized at lower temperatures, there would be fewer microalloying solute atoms into austenite, and thus fewer precipitates form during the subsequent cooling process, leading to less precipitation strengthening. On the contrary, if austenitization proceeds at too high temperatures,
microalloying solute atoms would be dissolved stably into austenite matrix, and it would be more difficult for carbide precipitates to nucleate.

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