Formation of Fine Austenite through Static Recrystallization in Low Carbon Micro-alloyed Steels

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The feasibility of further refinement of recrystallized austenite in a low carbon micro-alloyed steel was investigated through warm deformation of the overcooled austenite followed by reheating to the austenite non-recrystallization region. The results showed that static recrystallization of the deformed austenite happened at a higher degree of overcooling due to the increased driving force by warm deformation. The fraction of recrystallized austenite during reheating increased with increasing degrees of overcooling. After warm deformation at 550°C, the fraction of recrystallized austenite reached about 20.9%, and the average grain size was less than 5 μm. In addition, if a second deformation was imposed to the overcooled austenite at 550°C, considerable recrystallization was activated, resulting in a dramatically higher fraction of recrystallized austenite (94.0%) and a finer austenite grain size of 3.3 μm.

KEY WORDS: austenitic refinement; overcooled austenite; warm deformation; reheating; static recrystallization.

1. Introduction

Recently, more and more steels are required a good combination of strength, toughness and ductility.1–3) To realize this combination, refinement of microstructure is an economic and effective way, which can simultaneously improve both strength and toughness of materials.3–5) It is well known that the refinement of final structure can be realized by reducing the initial austenite grain size.6,7) The minimum austenite grain size obtained in high-strength low-alloy (HSLA) steels is normally about 10–20 μm through hot rolling in the recrystallized region of austenite.7) So far, several approaches have been attempted to refine the austenite grain size below 5 μm, such as austenite dynamic recrystallization,8) cyclic transformation of γ→α→γ and austenitization from the tempered and cold-rolled lath martensite.6,9)

Alternatively, austenite can be further refined by increasing rolling reduction or strain rate, or decreasing deformation temperature due to static recrystallization,7) i.e., improving the driving force for static recrystallization of austenite. Accordingly, Adachi et al.10) and Beladi et al.11) proposed a novel thermo-mechanical route to produce ultrafine ferrite grains through warm deformation of the overcooled austenite and reheating to the austenite region. The formation of such an ultrafine structure was ascribed to either dynamic strain-induced transformation in steels with low quench hardenability or static transformation in steels with high quench hardenability. Therefore, we can conclude that the driving force for austenite decomposition and nucleation rate can be extensively improved by imposing warm deformation to the overcooled austenite.

However, it remains unclear how warm deformation (i.e., degree of overcooling) influences the formation of such a fine-grained austenite structure during subsequent reheating in the non-recrystallized austenite region, especially in commercial low carbon micro-alloyed steels. Therefore, the present work aims to investigate the microstructure evolution of warm-deformed austenite during reheating using compression tests of a low carbon micro-alloyed steel. In particular, we put an emphasis on investigating the effect of the deformation temperature in overcooled austenite regions because of the fact that the driving force of austenite decomposition and nucleation of recrystallization is significantly affected by deformation temperature of overcooled austenite.

2. Experimental Procedures

The compression tests were performed on an MMS-200 thermo-mechanical simulator. The chemical composition of the micro-alloyed steel is Fe-0.13C-0.39Si-1.5Mn-0.014P-0.002S-0.034Nb-0.031V-0.016Ti-0.039Als-0.0032N-0.0048O (mass.%). The steel was produced by a continuous casting line. After austenitization at 1 200°C for 2 h, the steel was hot-rolled to 12 mm at 1 150°C, followed by air cooling to room temperature. The cylindrical specimens, whose dimensions are 6 mm diameter and 12 mm length for ultrafast cooling, were machined along the rolling direction of the plate.

According to the following solubility product equation proposed by Irvine,12) the solution temperature of the tested
steel is calculated to be 1197°C. So, the soaking temperature of 1200°C was selected to assure the dissolution of Nb(C,N).

\[
\lg \omega_{\text{Nb}} \left[ \frac{12}{14} \frac{\omega_{\text{C}}}{T} \right] = - \frac{2.26 - 6770}{T} \quad \text{......... (1)}
\]

where \( \omega_{\text{Nb}}, \omega_{\text{C}} \) and \( \omega_{\text{N}} \) are the concentration of niobium, carbon and nitrogen in mass percent, respectively, and \( T \) is the absolute temperature in K.

The thermo-mechanical processes are schematically shown in Fig. 1. After austenitization at 1200°C for 180 s, the specimens were cooled to various deformation temperatures (700, 650, 600 and 550°C) at an ultra-fast cooling rate of 50°C/s to investigate the influence of degree of overcooling. After holding for 1 s, a deformation of engineering strain of 40% and strain rate of 10 s\(^{-1}\) was exerted followed by rapidly reheating to 900°C at a reheating rate of 60°C/s. Subsequently, these specimens were water-quenched immediately to room temperature to reveal the original austenitic grain boundaries (OAGBs). A water-quenched specimen was also conducted to confirm the presence of a fully austenitic microstructure just before deformation. The tests were conducted in a nitrogen atmosphere as to prevent oxidation, and temperature was recorded using a thermocouple welded at the center of the surface.

Microstructure observations were performed using a LEICA optical microscope, at about 1 mm away from the location of welded thermocouple paralleled to the compression direction. After standard mechanical polishing, some specimens were etched in saturated picric solution containing 2–3 drops of hydrofluoric acid and appropriate paste shampoo for 60–120 s at 60–65°C to reveal the OAGBs, and other specimens were etched by 4% nital. The average grain size of recrystallized austenite was determined from measuring 100–200 grains, and the fraction of the recrystallized austenite was measured using an area method.

3. Results and Discussion

Figure 2 shows the microstructure and the OAGBs of the water-quenched specimen just before deformation at 550°C. The microstructure is fully martensitic. The \( M_s \) temperature of the tested steel is calculated to be 400°C using the following equation.\(^{13}\)

\[
M_s(K) = 764.2 - 302.6\omega_{\text{C}} - 30.6\omega_{\text{Mn}} - 16.6\omega_{\text{Ni}} - 8.9\omega_{\text{Cr}} + 2.4\omega_{\text{Mo}} - 11.3\omega_{\text{Cu}} + 8.58\omega_{\text{Co}} + 7.4\omega_{\text{W}} - 14.5\omega_{\text{Si}} \quad \text{..... (2)}
\]

where \( \omega_{\text{C}}, \omega_{\text{Mn}}, \omega_{\text{Ni}}, \omega_{\text{Cr}}, \omega_{\text{Mo}}, \omega_{\text{Cu}}, \omega_{\text{Co}}, \omega_{\text{W}} \) and \( \omega_{\text{Si}} \) are the concentration of carbon, manganese, nickel, chromium, molybdenum, copper, cobalt, wolfram and silicon in mass percent, respectively. The quenching temperature was higher than the \( M_s \) temperature of the tested steel, so the martensitic transformation happened in the water-quenching process. Therefore, there is no decomposition of austenite during cooling to 550°C and holding for 1 s due to the high cooling rate of 50°C/s, so the microstructure before deformation is austenite. And the original austenitic grain size is approximately 58.3 μm.

Figure 3 shows the true stress-true strain curves for specimens at various temperatures. This indicated that as the degree of austenitic overcooling increased, the flow stress increased, so is the stored energy induced by deformation.\(^{14,15}\)

Figure 4 shows the OAGBs with various degree of austenitic overcooling, i.e., deformation temperature, indicating a pancaked morphology. The grain boundary widths of pancaked austenite are 32.9, 32.8, 34.4 and 33.8 μm respectively, which correspond to the width of deformed austenite under 40% reduction, i.e., 34.9 μm. The OAGBs are smooth after deformation at 700°C (Fig. 4(a)). As deformation temperature dropped to 650°C, the bowing out...
of OAGBs appeared (Fig. 4(b)). With decreasing deformation temperature to 600°C, a few small grains about 3.2 μm were observed, mainly at the OAGBs (Fig. 4(c)). When the overcooled austenite was deformed at 550°C, more small grains about 3.8 μm were mostly located along the OAGBs, and a few fresh grains were formed in the deformed original austenite grains (Fig. 4(d)).

The microstructure before deformation in the temperature range of 550–700°C is austenite due to the high cooling rate of 50°C/s, which inhibits the decomposition of austenite. The as-reheated microstructure at 900°C is also austenite after warm deformation at various deformation temperatures. The austenite grain boundaries at 900°C are characterized by a pancaked morphology, which results from warm deformation at low temperature. Meanwhile, the change in austenite grain boundaries at 900°C with different deformation temperature is in accordance with the typical recrystallization characteristics of austenite. Therefore, the austenite experienced restoration rather than decomposition during reheating process after warm deformation, which may involve meta-dynamic recrystallization, static recovery or static recrystallization.

Figure 3 shows that there is no decrease in flow stress with increasing true strain, regardless of deformation temperatures, implying no dynamic recrystallization during warm deformation. Thus, it can be inferred that the deformed austenite underwent static recovery or static recrystallization during subsequent reheating process. After warm deformation at 700°C, static recovery of the deformed austenite occurred during reheating (Fig. 4(a)). With decreasing temperature to 650°C, static recrystallization was activated (Fig. 4(b)). The fraction of recrystallized austenite increased with increasing degrees of austenitic overcooling as plotted in Fig. 5. It is noteworthy that after warm deformation at 550°C, the fraction of recrystallized austenite could reach 20.9%, and its average grain size could be refined to 3.8 μm.

\[ T_{nr} = 887 + 446\omega_C + 890\omega_Ti + 363\omega_{Al} - 357\omega_{Nb} + 644\sqrt[3]{\omega_{Nb}} - 644\sqrt[3]{\omega_{Nb}} + 732\omega_V - 230\sqrt[3]{\omega_V} \]  

where \( \omega_C, \omega_Ti, \omega_{Al}, \omega_{Nb}, \omega_{V} \) are the concentration of carbon, aluminum, titanium, silicon, niobium and vanadium in mass percent, respectively. Below the \( T_{nr} \), no recrystallization was expected in deformed austenite due to the Nb (C,N) precipitation and Nb in solution for Nb micro-alloyed steels. Vervynckt et al. also demonstrated that this phenomenon was the result of the smaller driving force of recrystallization than the Zener pinning force of small precipitates. However, the \( T_{nr} \) value was obtained under the condition that deformation occurs in the austenite stable region and reduction per pass was relatively small. Bai et al. reported the effect of deformation parameters on the no-recrystallization temperature in Nb-bearing steels. They found that \( T_{nr} \) would be reduced with increasing reduction per pass and strain rate. This variation of \( T_{nr} \) with the pass reduction can be explained by the difference in the kinetic between recrystallization and precipitation, i.e., the former is more sensitive to strain than the latter. In the present work, as the deformation temperature was lowered to 650°C, the austenite static recrystallization took place during reheating to 900°C, which is lower than the \( T_{nr} \) of the tested steel. With further decreasing to 550°C, the fraction of recrystallized austenite at 900°C after warm deformation at various temperature and reheating.
austenite reached 20.9%. Decreasing deforming temperature, the flow stress would increase (Fig. 3), so was the deformation-induced stored energy. The increased stored energy accelerated both recrystallization and precipitation kinetics; however, the recrystallization kinetics was more likely to be sensitive to deformation in this case. Therefore, static recrystallization of the deformed austenitic took place and fine grains were produced during reheating to the temperature below the Tnr.

To further promote recrystallization of the deformed austenite, after first deformation at 550°C and reheating to 900°C, a second cooling and deformation at 550°C was followed, by subsequent a second reheating to 900°C, as shown in Fig. 6, and the OAGBs are shown in Fig. 7. In this case, the fraction of recrystallized austenite reached as high as 94.0%, and grain size was about 3.3 μm. Therefore, we can conclude that an additional deformation to the overcooled austenite improved the density of intragranular defects, and thus significantly promoted nucleation of intragranular austenite during static recrystallization. Meanwhile, the flattening of more austenite grains due to the additional deformation corresponded to smaller distance between adjacent austenite grain boundaries, which limited the growth of recrystallized austenite.

This process exhibits great potential for hot rolling mill applications where ultra-fast cooling and reheating equipment were implemented in the midst of rolling. According to the above work, the austenite can be dramatically refined, so that ultrafine-grained structures with a good combination of high strength and toughness can be achieved by controlling transformation of the refined austenite.

4. Conclusions

The present work suggests that further refinement of the recrystallized austenite was feasible by imposing warm deformation to the overcooled austenite followed by reheating to the austenite non-recrystallized region. The increased degree of overcooling improved the driving force for austenite recrystallization after warm deformation. When the driving force for austenite recrystallization reached to a certain degree, static recrystallization of the deformed austenite took place during reheating process. When an additional deformation was imposed to the overcooled austenite, considerable recrystallization was introduced, leading to a dramatically higher fraction of recrystallized austenite (94.0%) and the finer austenite grain size of 3.3 μm.

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