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Microstructural Evolution of Ultra Fine Grained C-Mn Steel Warm Rolling and Intercritical Annealed

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The attainment of finer ferrite grain structure in low carbon is object of interest because significantly higher yield strengths and lower ductile-brittle transition temperatures can be predicted at the same time with ultra-fine grain sizes. This work verified the microstructural evolution of a low carbon 0.15%C-1.39%Mn steel after an ice brine quenching from 1200 °C followed by warm rolling and intercritical annealing at 800 °C at different times. To compare the final microstructure, a first group of specimens were heated at temperatures in the range from 660 °C to 800 °C during 30 min and quenching in ice brine. After quenching from 1200 °C, a second group of specimens were laboratory warm rolled at 700 °C and annealed at 800 °C, for 1, 60, 120 and 180 min, following air cooling or quenching. The final microstructure of all specimens was analyzed by quantitative metallography using optical and scanning electron microscopy. The initial steel grain size condition was 120 μm. The specimens, after the whole processing cycle achieved a microstructure with ferrite grain size between 1 μm and 1.5 μm. The ferrite grain size values changed until 50% for samples warm rolled and heat treated between the first and last annealing time.

Keywords: *ultra fine grain steel, warm rolling, intercritical annealing*

1. Introduction

The attainment of finer ferrite grain structure in low carbon is object of interest because significantly higher yield strengths and lower ductile-brittle transition temperatures can be predicted at the same time with ultra-fine grain sizes. This method is important as hardening mechanism in these steels when they are used in welding applications and is more inexpensive than employing high alloying steels[1,2].

Controlled rolling with accelerated cooling has been used to microstructure refinement and increase strength in HSLA steels[3-5], whereby the austenite deformation has an important role. However, the limit to which ferrite can be refined by this conventional thermomechanical processing of austenite is about 4 μm[6].

To reach best results, it might be demonstrated from steels submitted to heat treatment with warm rolling and intercritical annealing, the refinement of ferrite grain size to about 2 μm[7]. The objective of this work was to study the ferrite grain refinement during microstructural evolution from a thermomechanical process to obtain ultrafine ferrite grain size at a carbon-manganese steel using quenching, warm rolling laboratory and intercritical annealing.

2. Experimental procedure

The chemical composition of the steel investigated is given in Table 1. The warm rolling process was carried out on a 500 kN laboratory mill having rolls speed of 25 m/min.

To compare the final microstructure, a first group of specimens were heated at one temperature in the range from 660 to 800 °C (660, 680, 700, 720, 740, 760, 780 and 800 °C) for 30 min and quenched. With this procedure it was possible to determine the critical temperatures, Ar1 and Ar3, of the steel investigated.

Table 1. Steel composition (weight %).

C	Mn	Si	P	S	Al	N_2
0.16	1.39	0.39	0.016	0.009	0.039	0.0042

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Specimens of 16.2 mm thick slab commercial steel were reheated at 1200 °C for 30 min and submitted to ice brine quenching with cooling rate around of 300 °C/s. After that, all specimens were reheated at 740 °C for 30 min and submitted to warm rolling at 700 °C, with three equal pass reduction of 22.4% true strain each one. The final thickness specimens were 8.3 mm gauge sheets, resulting in a total reduction of 67.1% true strain. After first and second rolling passes, the samples returned to furnace at 740 °C and after the last rolling pass the specimens were air cooled. Annealing schedule was employed at all deformed specimens at temperature of 800 °C for 1, 60, 120 and 180 min. After the soaking time, some specimens were air cooled (annealing) and others were quenching. Figure 1 describe the scheme of the specimens processing.

Transverse sections of the heat treated and rolled specimens were examined by optical and scanning electron microscopy. Samples were prepared following standard procedures[5]. The austenitic grain size was measured for 1200 °C quenched condition by the linear average intercept method[8] and saturated aqueous picric acid solution was used as etchant. Otherwise, ferritic grain size and volume fractions of the final microstructure constituents were measured by an IMAGE PRO-PLUS™ image analyzer software, as stated by ASTM standards[9]. Nital 2% and Le Pera[10] were used as etchants. All results were established according to mean, standard deviation and relative error values for 95% confidence limit.

3. Results and Discussion

Ice brine quenching of non-deformed samples

The critical temperature for a C-Mn steel calculated according Ouchi *et al.*[11] using eq. 1 is: Ar3 = 726 °C. Figures 2 and 3 show how much the constituents volume fraction change with quenching temperature. It may be concluded from Fig. 2 that the transformation of ferrite and pearlite to austenite start at 740 °C and finish around 800 °C. Although, at 800 °C there still are some ferrite, as can be seen in the Fig. 4b.

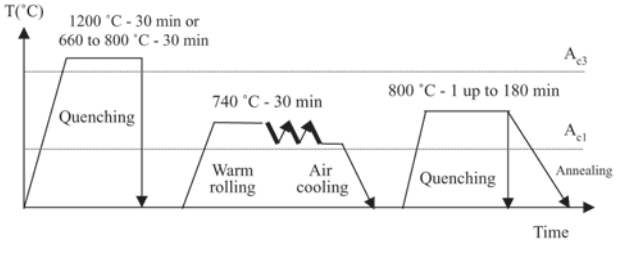

Figure 1. Processing schedule for deformed samples of the steel investigated.

$$Ar3\ (°C) = 910 - 310\%C - 80\%Mn - 20\%Cu - 15\%Cr - 80\%Mo - 0.35(t - 8) \quad (1)$$

where, t = thickness of the plate.

It may be seen from Fig. 3 the increase of ferrite volume fraction with decreasing the quench temperature because less austenite is transformed to martensite. Anyway, for these conditions the microstructure was not so refined and ferrite grain size was around 20 μm.

The microstructure after quenching from 1200 °C is shown in Fig. 4a. In the Fig 4b is shown the microstructure of the sample quenching from 800 °C. A fully martensitic structure was formed in Fig. 4a with distinct blocks evidenced by different etching contrast. These blocks consist of fine martensite laths (low carbon steels) parallels each other and they are delineated by the different orientations of the laths, according to the parent austenite grains[12]. The width of martensite blocks is not uniform, some blocks are 100 μm thin, and others are wider than 200 μm. The average prior-austenite grain size for this condition was 120 μm. The microstructure transformed from quenching temperature of 800 °C is more refined and has the presence of martensite and the ferrite that did not transform to austenite during the heating and soaking time, Fig. 4b.

Annealing after warm rolling

The effects of the deformation on the final microstructure of the processing samples might be seen in Figs. 5a-5d. At short annealing time (1 min), Fig. 5a-5b, the ferritic grain size is very small (~1 μm) but the microstructure is heterogeneous, as can be seen in the upper part of Figs. 5a-5b. Recrystallisation works[13-19] had already well established the annealing time-temperature and total deformation amount

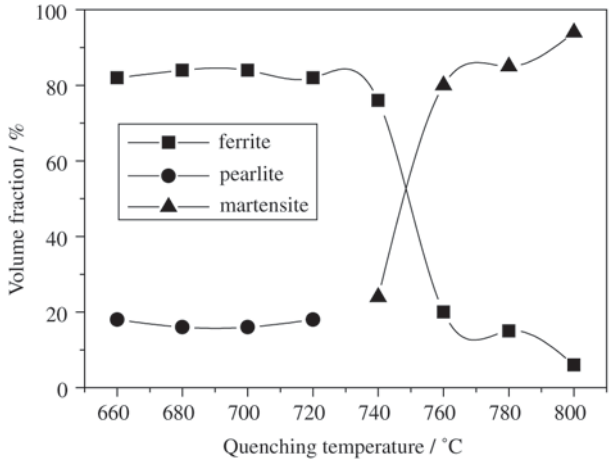

Figure 2. Effect of quenching temperature on the constituents volume fractions of non-deformed specimens.

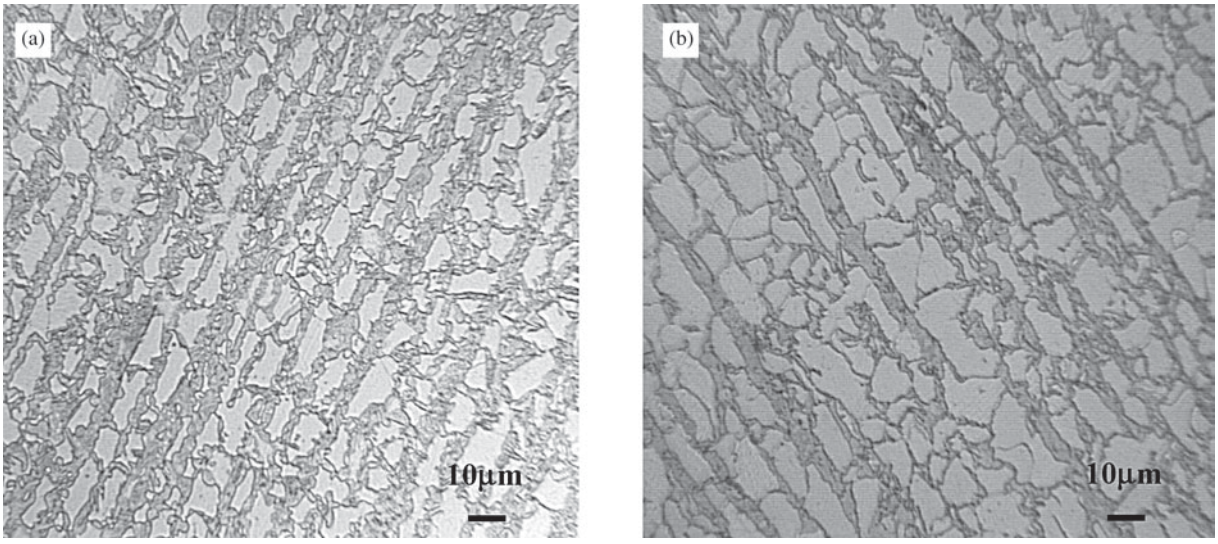

Figure 3. Optical micrographs of non-deformed samples quenching from different temperatures. a) from 760 °C, and b) from 740 °C. 2% Nital etched.

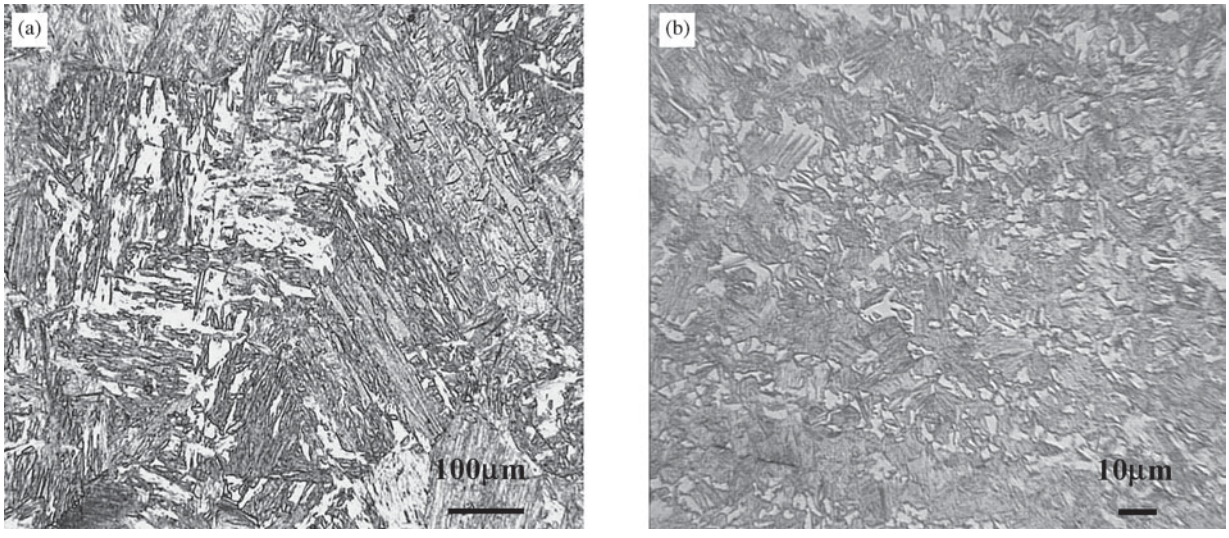

Figure 4. Optical micrographs of non-deformed samples quenching from different temperatures. a) from 1200 °C, and b) from 800 °C. 2% Nital etched.

dependence. The nucleation and growth of new grains have no meaning unless the time is greater than a critical value. This value is dependent of the total deformation amount, been smallest at high deformation temperature. Therefore, for this work, one minute at 800 °C was not enough time to recrystallise the microstructure uniformly. On the contrary, for longer annealing times (180 min) as shown in the Fig. 5c-5d, the microstructure was more homogeneous and its refinement was almost preserved, althought farrritic grain size had increased (1.5 μm).

For deformed samples submitted to quenching after heating and soaking time at 800 °C, the final microstructure was composed of a mixture of ferrite, martensite and traces of bainite. The Fig. 6a shows the results of the constituents volume fraction changes. The soaking time was not very important for modifications on the constituents volume fractions at these conditions.

In other way, when air cooling (annealing) was applied after soaking of deformed samples, pearlite, bainite and MA constituent were formed in the microstructure, Fig. 6b. They

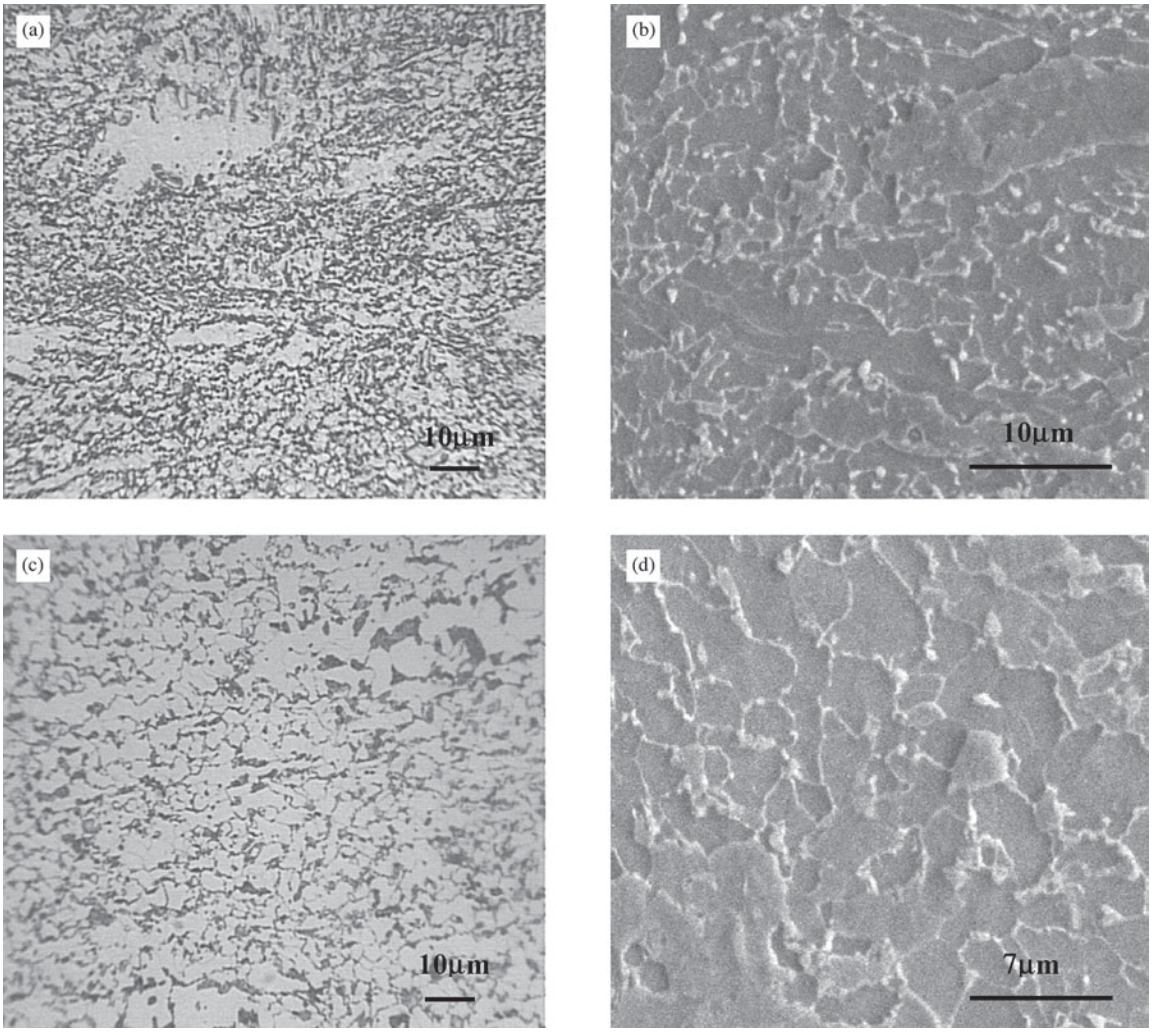

Figure 5. Photomicrographs of samples quenched from 1200 °C, deformed, annealed at 800 °C in different times and air cooled. a) and b) 1 min, c) and d) 180 min. 2% Nital. a) and c) O M; b) and d) SEM.

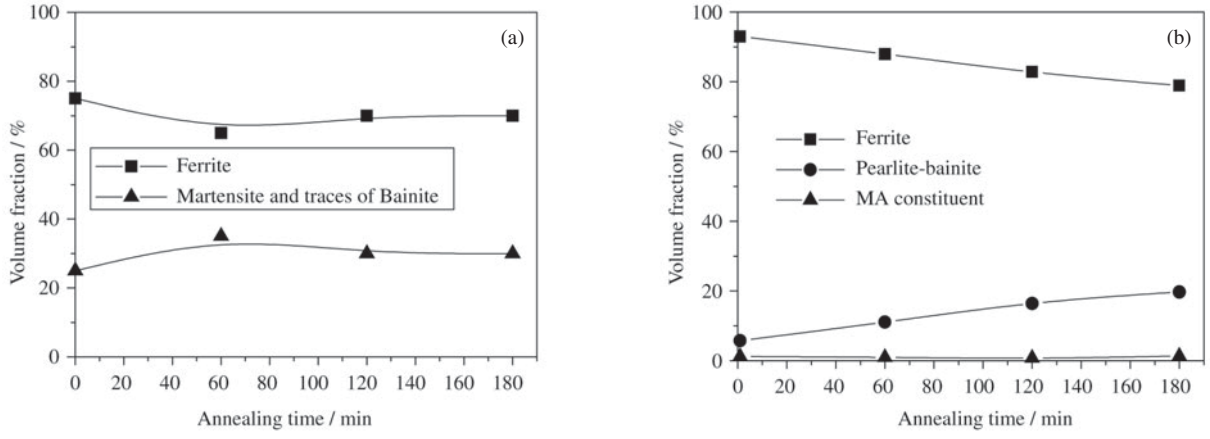

Figure 6. Effect of soaking time on the constituents volume fraction of the steel. a) samples quenched from 1200 °C, deformed, soaking at 800 °C and quenched again, b) the same schedule but air cooled (annealing).

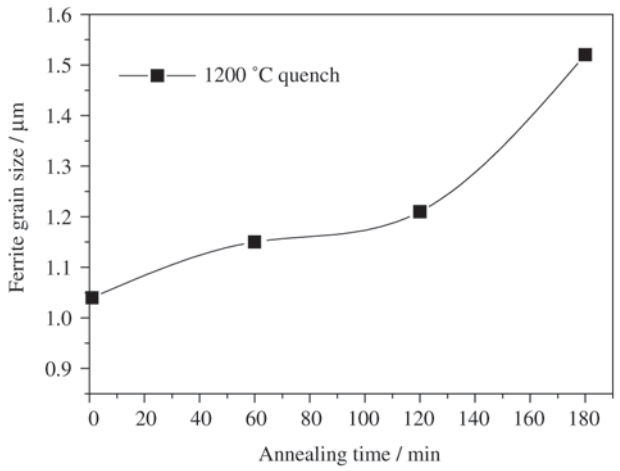

Figure 7. Effect of 800 °C annealing time on the ferritic grain size after warm rolling samples for initial quenching condition of 1200 °C.

are diffusional controlled transformation constituents and their volume fractions increased with annealing time as the grain growth. The MA constituent volume fraction shown in the Fig. 6b is almost constant with annealing time. In addition with large prior-austenite grain (120 µm), the high Mn content of the steel had produced some austenite more stable which had transformed to MA constituent during annealing heat treatment.

Figure 7 shows the strong effect of annealing time of heat treated processing samples on the final ferritic grain size. The ferritic grain refinement was better when sample had submitted to annealing during one hour because a great refinement with a more uniform structure was reached. Meanwhile, short annealing times promote a finer grain but with non-homogeneous microstructure.

From Fig. 7 can be shown that the ferritic grain size increased almost 50% when the annealing time change from 1 min (1.04 µm) to 180 min (1.52 µm) for 1200 °C initial quenching condition. Even so, for the longer annealing time the microstructure was very refined as can be seen in Fig. 5c-5d.

4. Conclusions

- The heat treatment and warm rolling conditions employed on the 0.15%C-1.39%Mn steel investigated in this work conducted to a large ferritic microstructural refinement. In the best processing condition, ferritic grain size was 1.2 µm, considering the initial 120 µm prior-autenite grain size, 1200 °C initial quenching and annealing at 800 °C for 60 min, after warm rolling followed by air cooling. According to the annealing time interval applied, a ferrite grain size maximum variation of 50% was observed.
- Short annealing times (1 min) at 800 °C produced a heterogeneous microstructure. In this case, total deformation, temperature and initial quenching from 1200 °C were not enough to a complete recrystallization of the microstructure.
- For air cooling after warm rolling condition, the final microstructure was formed by ferrite, pearlite and MA constituent. The volume fractions of pearlite constituent increase with annealing time as stated by diffusional controlled constituents transformation. Meanwhile, MA constituent was kept almost constant for all annealing times.
- For samples quenched after warm rolling and soaking condition, the final microstructure was formed by a mixture of ferrite, martensite and traces of bainite. The soaking time did not have eminent effect on the constituents volume fraction changes.

Acknowledgments

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