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The role of Nb in Q-P-T process in martensite/austenite microstructures

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Abstract: Understanding carbon redistribution in steels is crucial in developing advanced high strength steels. For instance, Quenching & Partitioning & Tempering (Q-P-T) processes rely on the partitioning of carbon from martensite into austenite, where at the end of the heat treatment the carbon-enriched austenite shows higher stability at room temperature. The effects of 0.05%Nb addition on the austenite stability and mechanical properties of quenched and partitioned Fe-C-Mn-Si-Cr-Mo steel were investigated by means of microstructure observation, x-ray analysis. The Nb addition lowered the martensite start temperature (from 304 to 291 °C) and the partitioning temperature (from 140 to 320°C) to achieve the maximum retained austenite fraction of about 18%. The increase in Nb content brought about grain refinement and an increase of the retained austenite fraction in the quenched and partitioned specimens. The stability of the retained austenite was increased with the Nb addition, resulting in retarded kinetics of the strain-induced martensite transformation during deformation.

1. INTRODUCTION

The concept of Q&P steel was first proposed by Speer and his coworkers [1]. Some amount of untransformed austenite can be retained at room temperature because of the carbon enrichment in austenite diffused from martensite when the steel sample is held at a temperature between the martensite start and finish temperatures. During deformation, the retained austenite is mechanically transformed to martensite, resulting in a simultaneous increase in both strength and elongation. The stability of the retained austenite is a key parameter to determine the mechanical properties; thus, most prior studies have been focused on the control of the stability and fraction of retained austenite by changing alloying elements such as C, Mn, Si, or Al, and by the partitioning conditions [2-3]. For the Q&P treatment, Rizzo et al. [4] reported the effects of C and Ni additions on the tensile strength of four Fe-C-Mn-Si-Ni-Cr-Mo steels. Their work focused on the variation of the mechanical properties depending on the partitioning temperature in the four steels with different C and Ni contents. With the development of Nb micro-alloying technology, it is well known that the recrystallization of plain carbon steel is influenced by the addition of micro quantity of Nb. The retardation of recrystallization is caused by the solute drag effect of Nb and the pinning effect of fine precipitates such as NbC. By the retardation of recrystallization, strain can be accumulated and the microstructure can be refined after phase transformation.

In the present study, therefore, we investigated the effect of Nb addition on the stability of the retained austenite formed during the Q-P-T treatment with different partitioning temperatures. The relationship between the change of the retained austenite and the microstructure as a function of the Nb content will be discussed in this paper.

2. EXPERIMENTAL PROCEDURE

Steel ingots, weighing 25 kg, were prepared using vacuum induction melting (VIM). Their chemical composition is shown in Table 1. The amount of C and Mn as austenite stabilizing elements was 0.3 and 1.2 wt%, respectively. Si, in the amount of 0.6 wt% was added to suppress cementite formation during isothermal holding at low temperature. To investigate the effect of Nb addition, 0.05 wt% Nb was added to the base composition (Fe-C-Mn-Si-Cr-Mo). The ingots were homogenized at 1200 °C for 1 h and then hot rolled to a final thickness of 10 mm. The heat treatments applied were Q-

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P-T style heat treatments, shown in Fig. 1. The heat treatments were carried out using a DIL805A thermal dilatometer, where cylindrical samples of 4mm diameter and 10mm length were obtained from the middle of the rolled plate, parallel to the rolling direction. The heating rate used was 5°C/s and all quenching processes were done using helium gas.

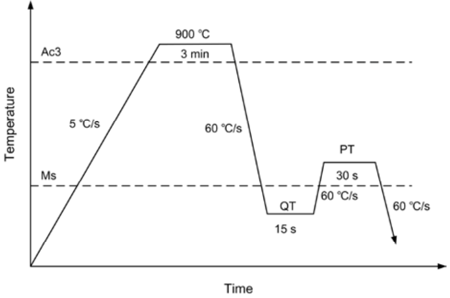

Fig. 1. Illustration of heat treatments applied to alloys

Field emission scanning electron microscope (FE-SEM) was used to observe the microstructure of the heat treated specimens. The samples for microstructural observation were mechanically polished and etched for 5–15 s in nital etchant [3 mL nitric acid (HNO3) +97 mL ethanol]. Electron backscatter diffraction (EBSD) analysis was performed to observe the changes in the fractions of retained austenite and martensite. X-ray diffraction (XRD) analyses were performed to measure the volume fraction of retained austenite; the 2θ scan range was from 40° to 120°. The amount of carbon diffused from the quenched martensite to the austenite during the partitioning process was calculated by analyzing the lattice parameters obtained from the XRD peaks.

Table 1. Chemical composition of the steels used in the present study(in wt.%).

Steel	C	Si	Mn	Cr	Mo	B	Nb	Fe
Nb-0	0.34	0.66	1.18	1.22	0.52	0.005	-	Bal.
Nb-1	0.36	0.68	1.21	1.22	0.54	0.005	0.044	Bal.

3. RESULTS AND DISCUSSION

The calculated evolution of the phase fractions during Q-P-T processing is presented in Fig. 2. Fig. 2 shows the calculated equilibrium fraction of retained austenite corresponding to each alloy composition. Fig. 2 shows the calculated phase fractions as a function of T_Q, assuming full C partitioning into austenite prior to the final quenching, as proposed by Speer et al [1]. The martensite start (M_S) temperature was determined by dilatometry tests. The measured M_S temperature decreased from 304 to 291 °C as the Nb content added. The austenite fraction after initial quenching to a certain T_Q was predicted by applying the Koistinen-Marburger relationship. The austenite volume fraction is seen to increase with increasing T_Q. The C content of the retained austenite is gradually reduced with increasing T_Q. At $T_{Q,max}$ a maximum volume fracture of retained austenite is obtained in the final microstructure. The Nb added specimen, containing 0.05 wt% Nb, shows the lower Ms temperature and larger volume fraction of retained austenite. The temperature for achieving the maximum retained austenite fraction (T_{max}) was 204 and 230 °C for the Nb-0 and Nb-1 specimens, respectively. The partitioning treatment was performed at four different temperatures: 140 °C, 200 °C, 260 °C and 320 °C.

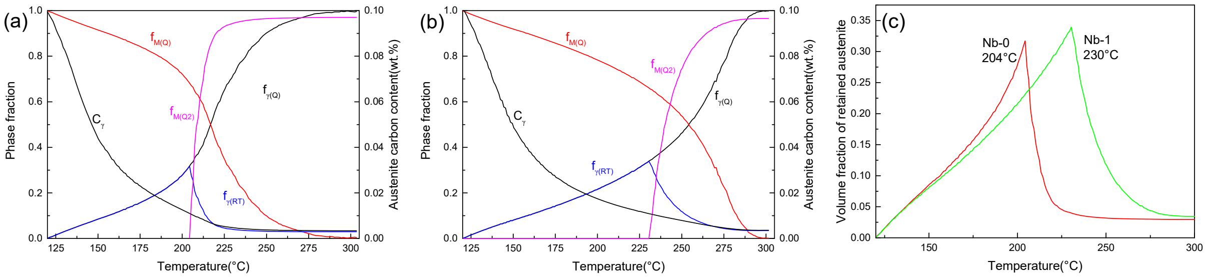

Fig. 2. Calculated TQ-dependence of the phase fraction of austenite and secondary martensite, assuming full C partitioning into austenite prior to the final quenching: (a) Nb-0, (b) Nb-1, (c) comparison of the calculated results. The designations $f_{\gamma(Q)}$, $f_{M(Q)}$, $f_{\gamma(RT)}$, $f_{M(RT)}$, and C_γ indicate the calculated fractions of initially quenched austenite and martensite at the quench temperature, and of finally quenched austenite and martensite at the room temperature, and the calculated carbon concentration in austenite.

Fig. 3 and Fig. 4 show the SEM images and image quality (IQ) maps of the specimens partitioned at different quenching temperature. A mostly martensitic mirostructure was observed regardless of composition and partitioning temperature. It was confirmed that the grain size decreased drastically as a result of the Nb addition. The average grain sizes were 12 μm and 6 μm for the Nb-0 and Nb-1, respectively. The refinement of the austenite grain size strongly increases the stability of austenite [5]. IQ maps of the specimens show the grain size distribution and the retained austenite distribution.

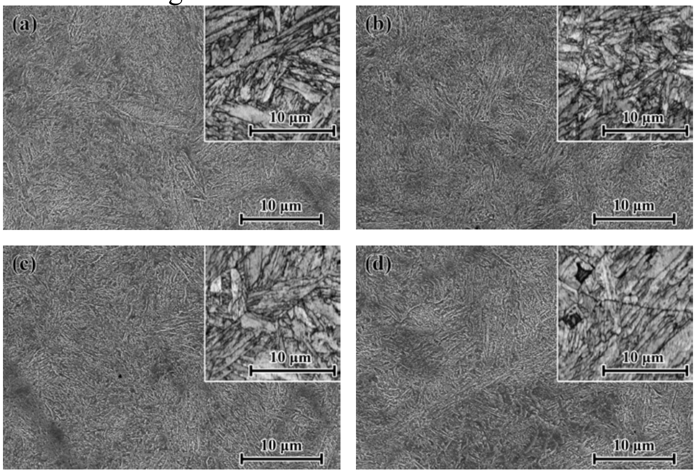

Fig. 3. SEM images and IQ map (EBSD analysis results) for the Q-P-T Nb-0 sample treated by different temperatures: (a) 140 °C, (b) 200 °C, (c) 260 °C, (d) 320 °C.

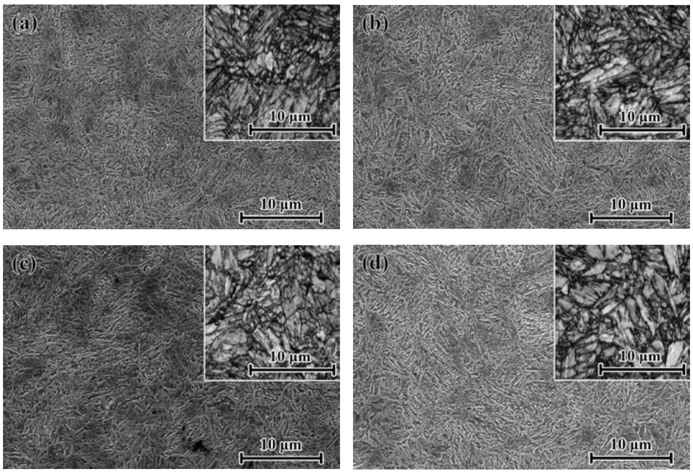

Fig. 4. SEM images and IQ map (EBSD analysis results) for the Q-P-T Nb-1 sample treated by different temperatures: (a) 140 °C, (b) 200 °C, (c) 260 °C, (d) 320 °C.

Fig. 5 shows the XRD peaks of the samples measured after Q-P-T process. The changes of the retained austenite fraction were calculated by XRD peak analysis. The quantitative fraction of retained austenite as follows [6]:

$$V_\gamma = \frac{\dfrac{I_{\gamma(200)}+I_{\gamma(220)}+I_{\gamma(311)}}{3}}{\dfrac{I_{\alpha(200)}+I_{\alpha(211)}}{2}+\dfrac{I_{\gamma(200)}+I_{\gamma(220)}+I_{\gamma(311)}}{3}} \qquad (1)$$

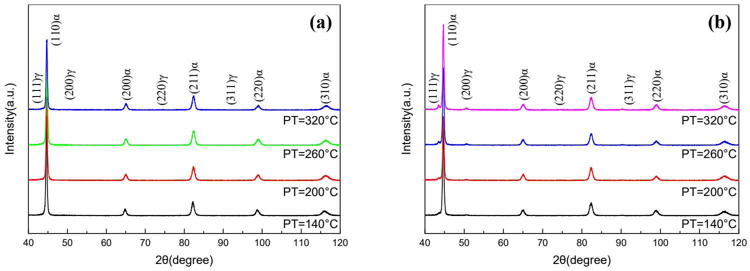

Fig. 5. XRD analyses of the Q-P-T specimens: (a) Nb-0, (b) Nb-1

The austenite peaks of the spectra for the specimens without Nb addition can not be observed, which mean that the contents of retained austenite are too small to be detected. However, the peaks of the Nb-added specimens can be observed, especially in higher partitioning temperature.

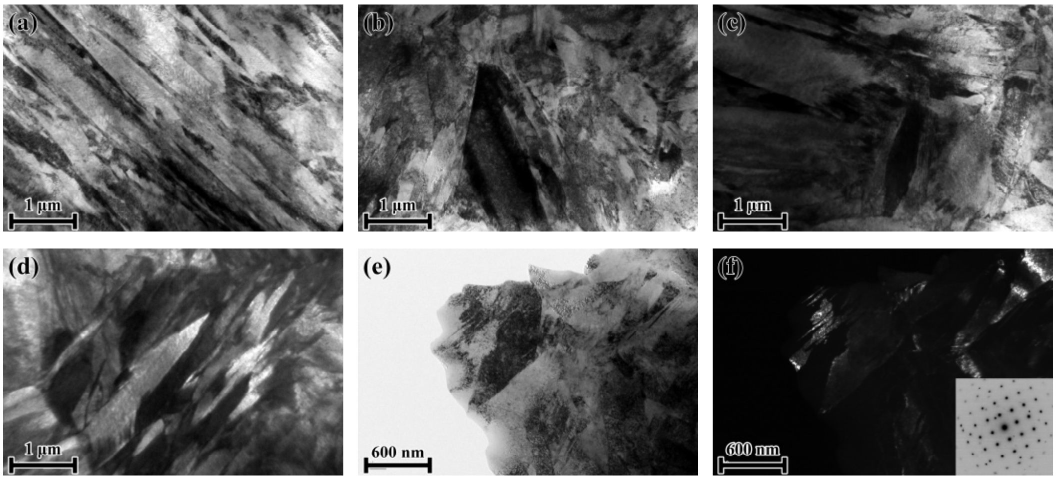

Fig. 6. TEM images show martensite and retained austenite in Nb-0 samples (a-c) and Nb-1 samples (d-f) at quenching temperature 260 °C: (a) coarse martensite plates, (b-c) blocky retained austenite, (d) fine martensite plates, (e) bright field image, (f) dark field image and SAED pattern showing twinned martensite structure.

TEM observation was further employed to identify the microstructure details. Fig. 6 presents the typical TEM microstructure of Q-P-T processed specimens (partitioning temperature = 400 °C) that consists of lath martensite containing blocky and film-like retained austenite located between martensite laths. Fig. 6e and 6f show the corresponding bright field image, dark field image and selected area electron diffraction (SAED) pattern for twinned martensite structure. The specimens of Nd-added have more twinned martensite structure than the Nd-free specimens. This film-like retained austenite and twinned martensite structure can play an important role of strength and ductility.

4. CONCLUSION

In the present study, we investigated the effect of Nb addition on Q-P-T treated Fe-C-Mn-Si-Cr-Mo steel. In the Nb-added Q-P-T treated steels, the Nb addition increased the austenite stability due to the grain refinement and decreased the M_S temperature and increased T_{max} temperature whereas the partitioning temperature controlled the fraction of retained austenite and the austenite stability related to C enrichment in austenite diffused from martensite. The microstructure of Nb-free sample consists of coarse and long martensite plates and the microstructure of Nb-added sample consists of fine and short martensite plates. Retained austenite is mainly in the form of blocky for the Nb-free sample, and in the form of film for the Nb-added sample. Microstructure of Nb-added sample has more twinned martensite structure than the Nb-free sample. With the Nb-added, the sample will have finer microstructure and more twinned martensite. Then this microstructure can be easy to get a good combination of strength and ductility.

REFERENCES

[1] J. Speer, D.K. Matlock, B.C. De Cooman, J.G. Schroth, Acta Mater. 51 (2003) 2611–2622.
[2] A.J. Clarke, J.G. Speer, M.K. Miller, R.E. Hackenberg, D.V. Edmonds, D.K. Matlock, F.C. Rizzo, K.D. Clarke, E. De Moor, Acta Mater. 56 (2008) 16–22.
[3] A.J. Clarke, J.G. Speer, D.K. Matlock, F.C. Rizzo, D.V. Edmonds, M.J. Santofimia, Scr. Mater. 61 (2009) 149–152.
[4] S.J. Lee, C.J. Van Tyne, Metall. Mater. Trans. A 43 (2012) 422–427.
[5] S.J. Lee, S. Lee, B.C. De Cooman, Int. J. Mater. Res. 104 (2013) 423–429.
[6] ASTM E975-00, vol. 03. 01, ASTM International, W. Conshohocken, PA, 2003.