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Thermo-stability of ultra-fine non-equilibrium microstructures

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Abstract: The evolution of the microstructures and hardness of a bainitic plate steel during tempering at 650°C has been investigated. The steel was manufactured by RPC (relaxation-precipitation controlling phase transformation) technique. A part of the plate was reheated to 930°C and held for 1 h before quenched into water (RQ). No obvious change was detected by means of optical microscopy in the RPC steel tempering for 0.5 h, while dislocation cells were formed inside the bainite laths, accompanied by an obvious drop of hardness. The bainite laths started to coalesce in some regions, but the sample hardness kept nearly constant during tempering from 1 to 7 h. With further tempering, polygonal ferrite was formed in local regions while the hardness decreased dramatically. The RQ samples softened faster during tempering and finally transformed into the polygonal ferrite completely. These results indicate that the thermo-stability of fine non-equilibrium microstructures is tightly related to their history.

Key words: microalloyed steel; bainite and martensite; tempering; RPC technique

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Refining the effective grain size is one of the basic methods to improve the mechanical properties of bainite steels [1, 2]. Due to high stored energy in the steels, their microstructures will evolve towards equilibrium accompanied with the change of mechanical properties while the steels were tempered. It has been found [3] that the bainite will recover and recrystallize when held above 600°C. In Fe-18Ni alloy, the austenite formed from martensitic reversion will recrystallize as well during reheating [3]. These phenomena are possibly owed to the high density of defects in bainite. New developing RPC (relaxation-precipitation controlling phase transformation) technique is an efficient process to refine the bainite effective grain size in low carbon steels [4-6]. The interface energy will heighten after refinement. This non-equilibrium microstructure stored high free energy in metastable state. General the grain will coarsen during welding or heat treating [3]. So it is important to study the thermo-stability of microstructure. In this paper, the thermo-stability of ultra-fine bainite has been studied.

1 Material and experimental procedures

The steel tested was melted in a vacuum induction furnace. The chemical composition (mass fraction in %) of the steel is: C, 0.04; Si, 0.32; Mn, 1.53; P, 0.009; S, 0.0055; Cu, 0.52; Ni, 0.23; Mo, 0.24; Nb, 0.0046; Ti, 0.042; and B, 0.001. According to **figure 1**, RPC steels were austenized at 1200°C, and rolled at the recrystallization temperature and non-recrystallization temperature range, respectively. The cumulative reduction in each stage is more than 60%. After rolling at 850°C, the plate cooled in the air (relaxation) for 20 s and then quenched in water. A part of the plate was reheated to 930°C and held for 1 h before quenched into water (RQ samples). The RPC samples and RQ samples were tempered at 650°C for 0.5-20 h respectively.

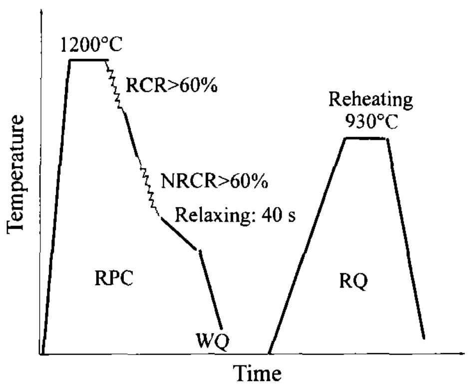

Figure 1 RPC and RQ technique view.

The hardness measurement was carried out with an

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HD9-45 Vickers hardometer, an average over 5 measures was taken for each sample. The samples for optical examination and SEM were etched with a 3% Nital. TEM observation was carried out with an H-800 electron microscope.

2 Results and discussion

2.1 Hardness change during tempering

Figure 2 shows the hardness changed with the tempering time at 650°C for RPC and RQ steel, respectively. In the curve, the hardness change of RPC steel with time at 650°C could be divided into 3 stages. The Vickers hardness decreases from 306 to 294 (tempered for 0.5 h). In the second stage (1-7 h) the hardness does not change obviously. With farther tempering the hardness decreased rapidly again. The hardness-time curve of RQ samples (curve Q) is obviously different from curve P. Despite RQ steel has a lower original hardness, the samples softened faster during tempering. The Vickers hardness decreases dramatically from 291 to 244 after tempering for 0.5 h. After tempering for 7 h, the hardness does not decrease with further tempering. The hardness of RQ samples tempered for 7 h has dropped into the hardness range of polygonal ferrite. It indicates that the evolution of microstructures has nearly reached the equilibrium state at the moment. According to reference [2], the second rapid decrease at the hardness-time curve shows the beginning of recrystallization. The recrystallization of RPC samples starts after 7 h while that of the RQ samples starts at 1 h and finishes at 7 h. This indicates that reheating promotes the recrystallization during tempering.

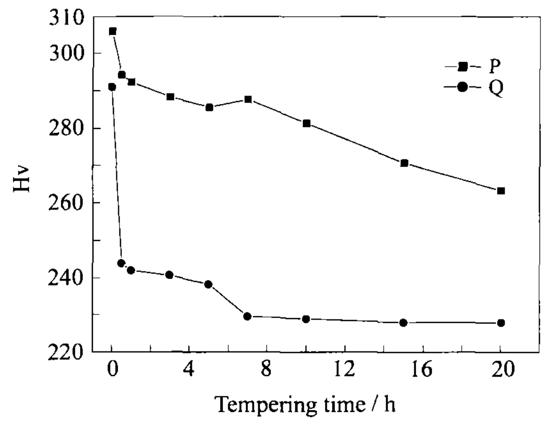

Figure 2 Hardness change of the samples tempered at 650°C with time.

2.2 Microstructure evolution during tempering

Figure 3 shows that both RPC samples and RQ samples consist of ultra-fine bainite along with little martensite by means of SEM before tempering. The difference between the two types of samples is that bainite is finer in RPC sample than in RQ sample. The microstructures of the samples tempered for different time are exhibited in **figure 4**. By optical microscopy, no obvious distinction was detected between the original RPC samples and the samples tempered for 0.5 h (figure 4(a)), while the hardness of the latter was remarkably lower. Thus, the evolution must begin inside the bainitic laths. A part of the bainite lath boundaries have disappeared and the bainite lath started to coalesce in some regions in the samples tempered for 1-7 h (figures 4(c), (e)). With further tempering, the polygonal ferrite started to occur in the samples (figure 4(g)). According to figure 4(i), until tempering for 20 h, only about half of the bainite laths transformed into ferrite.

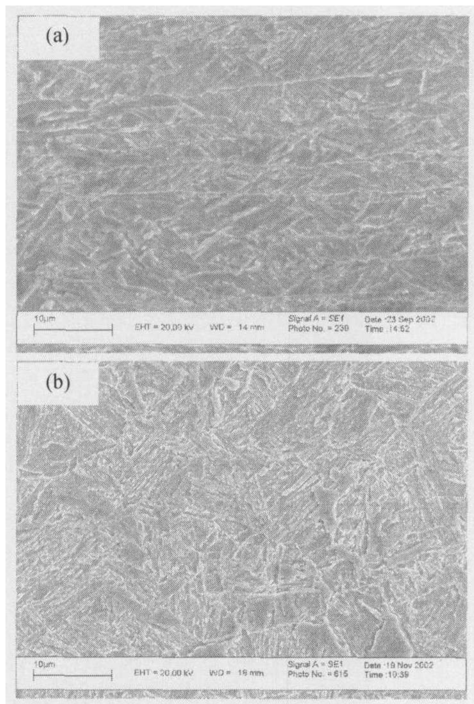

Figure 3 Microstructure of RPC (a) and RQ (b) samples before tempering.

The microstructures of RQ samples for different time at 650°C are given in figures 4(b), (d), (f), (h), (j). Comparing with the original samples, the evolution in RQ samples is far faster. After tempered for 0.5 h, the polygonal ferrite started to occur in the samples (figure 4(b)). Half of the microstructures have transformed into polygonal ferrite after tempering for 1 h (figure 4 (d)). With farther tempering, the evolution has finished in fact (figures 4(f), (h), (j)).

2.3 Discussion

Ultra-fine bainite along with little martensite are non-equilibrium. They trend to evolve towards equilibrium microstructures. In fact, this kind of evolution progresses by recovery and recrystallization [3, 7]. In the evolution of bainite towards equilibrium, dislocation motion plays the role of precursor. The rate of the evolution is determined by the movability of dislocations inside the bainite [3, 8].

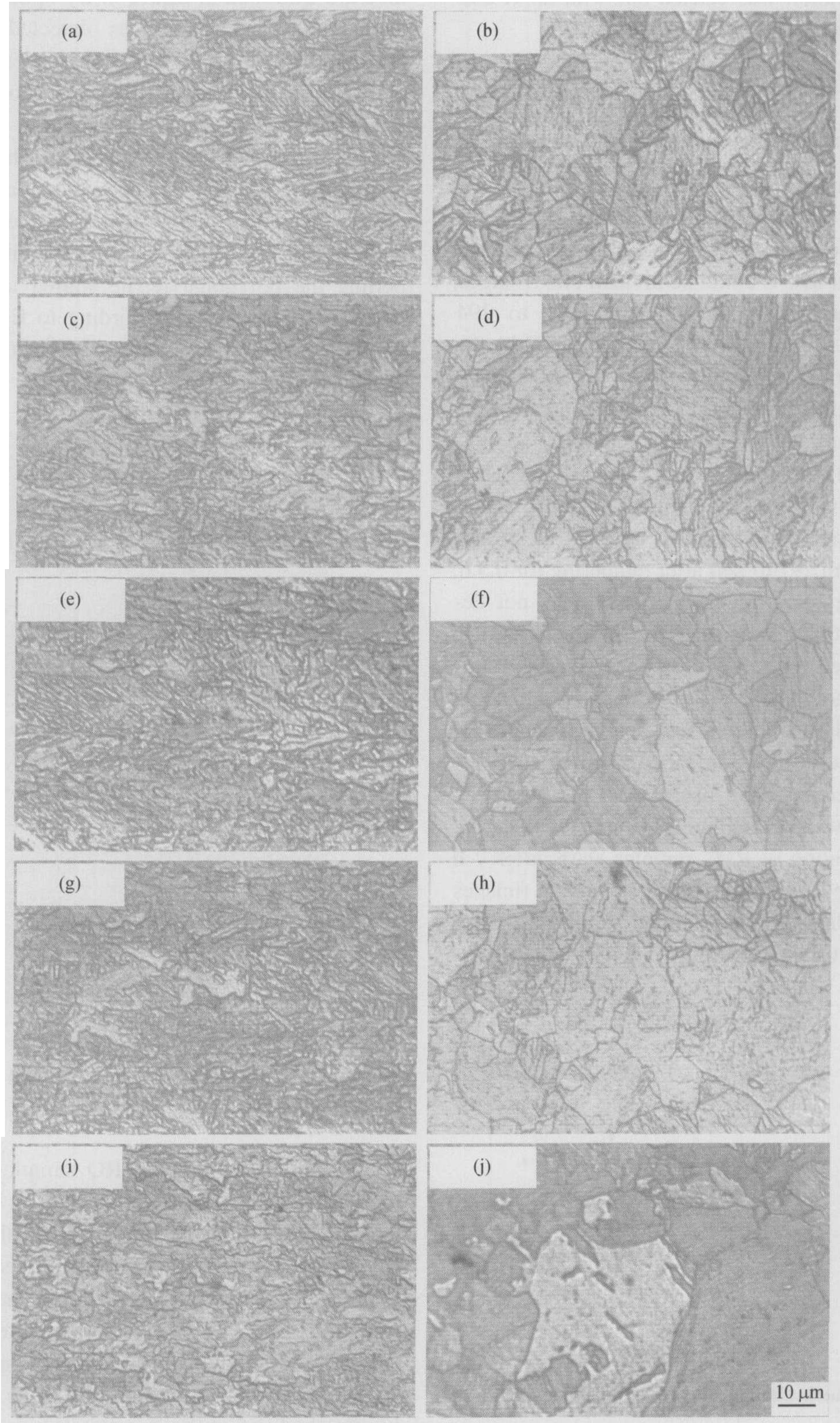

Figure 4 Microstructures of RPC and RQ samples tempered at 650°C for different time: (a), (b) 0.5 h; (c), (d) 1 h; (e), (f) 7 h; (g), (h) 10 h; (i), (j) 20 h.

The deformation of austenite not only causes the dislocation density to increase, but also induces fine (Nb, Ti) (C, N) to precipitate along dislocations and pin dislocation during slow cooling or isothermal holding after deformation [3, 9, 10]. After the bainitic transformation, dislocations formed in deformed austenite remain to be pinned by precipitates so that they are difficult to move during tempering. However, the dislocations induced by bainitic transformation are not pinned by strain-induced precipitates so that they are easier to move. These two types of dislocations in the original RPC samples can be seen in **figure** 5(a). A kind of dislocations mainly originates from the deformation of austenite are twisted. The other dislocations occurred in the bainitic transformation during water cooling are straight as arrow. According to figure 5(b), after tempering for 0.5 h the twisted dislocations were pinned by the precipitates. While some straight dislocations have vanished in substance due to their movability. So the hardness of the sample obviously decreased while its microstructures display no obvious change by the moment. The dislocations pinned by the precipitates are difficult to move during the subsequent tempering as figure 5(c) shows, so that the hardness of the sample has no obvious change. Until tempering for 7 h, the dislocations gradually get rid of pinning of the precipitates, possibly owing to the coarsening of the precipitates. As shown in figure 5(d), after tempering for 20 h, the dislocations have disappeared in local regions. In the final stage, the microstructures and hardness of the sample change obviously.

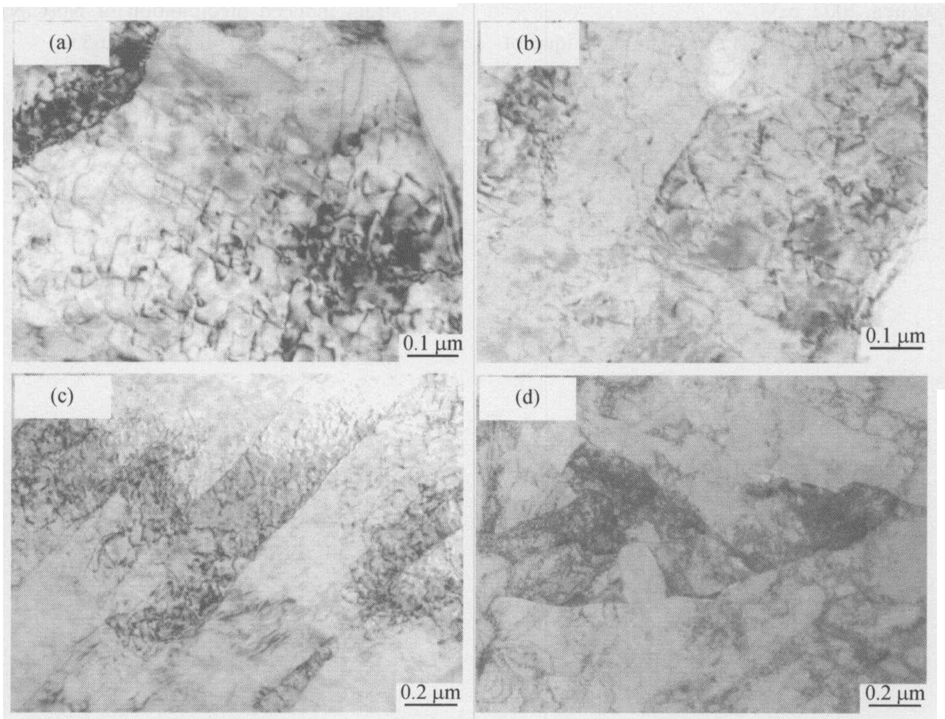

Figure 5 Dislocation configuration in RPC samples before and after tempering for different time at 650°C: (a) 0 h; (b) 0.5 h; (c) 7 h; (d) 20 h.

Though the reheating stage at 930°C reduces the dislocation density and the driving fore of bainite towards equilibrium, it promotes dislocations to get rid of pinning by thermal-excitation so that the resistance of recovery and recrystallization during tempering is lowed. So the microstructures and hardness of RQ samples change rapidly during tempering.

3 Conclusions

(1) The microstructures of the steel manufactured by a recently developed RPC technique possess high thermo-stability during tempering. While the steel was reheated to 930°C and held for 1 h before quenched into water (RQ) it evolves dramatically during tempering.

(2) The evolution of the microstructures of RPC steels is similar to the recovery and recrystallization in deformed metal. It can be divided into three stages, *i.e.* dislocation cells forming, laths coalescing and polygonal ferrite occurring subsequently.

(3) When the dislocations are pinned by strain-induced precipitates in deformed austenite, they remain to be pinned after bainitic transformation. The evolution speed of the microstructures towards equilibrium during the tempering of microalloyed steel is determined by pinned degree of dislocation rather than dislocation density.

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