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In-situ TEM Observation of Cementite Coarsening Behavior of 5Mn Steel during Tempering

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Abstract: The cementite formation and coarsening behaviors of 0.2 mass% C-5 mass% Mn steel during tempering at 500 ℃ were investigated by in-situ transmission electron microscope (TEM). In-situ TEM observation showed uniform distribution of cementite particles at the early stage of tempering in the rapidly heated (500 ℃/s) sample. Elemental analysis confirmed that the cementite growth was dominated by Mn diffusion. During the cementite growth, the coarsening behavior of intragranular cementite was significantly controlled by the matrix diffusion, while that of the intergranular cementite was mainly governed by the boundary diffusion. The in-situ TEM observation revealed that the dislocation pipe diffusion of Mn took place during tempering, which accelerated the Mn diffusion between cementite particles. The coarsening rates of individual cementite particles were calculated based on the in-situ TEM observation.

Key words: in-situ observation; cementite; coarsening behavior; Mn diffusion; 5Mn steel; tempering

The cementite precipitation and coarsening behaviors during tempering of martensite have been widely investigated[1-8]. Both theoretical predictions and experimental results have shown that the coarsening behavior is governed by a power law $d = kt^m$, where d is the particle diameter at time t, k is the coarsening rate constant, and m is a constant related to the controlling coarsening mechanism. The coarsening mechanism includes matrix diffusion, boundary diffusion and dislocation pipe diffusion[6,7], and has been shown to vary from intragranular cementite particles to intergranular ones[8]. Besides, the addition of Mn remarkably retards the cementite growth, due to the smaller diffusion rate of Mn compared to that of C[9-11]. However, most of the previous studies used ex-situ method to study the coarsening behavior of cementite, and only averaged experimental data was obtained, which was unable to track the coarsening behavior of individual cementite particles. As for cementite formation behavior, Furuhara et al.[12] reported more uniformly dispersed cementite precipitates at a rapid heating rate than at a low heating rate. It suggested that higher heating rate led to more nucleation sites and a better distribution of cementite. Recently, the 0.2 mass% C-5 mass% Mn steels after intercritical annealing treatment have drawn a lot of attention due to their extraordinary mechanical properties and their great application potential in automobile industry[13-17]. In this work, the cementite formation and coarsening behaviors of 0.2 mass% C-5 mass% Mn steel during tempering at 500 ℃ at two heating rates of 500 and 10 ℃/min were investigated by in-situ transmission electron microscope (TEM).

1 Experimental

Steels with a chemical composition of 0.2 mass% C-5 mass% Mn were prepared in an induction furnace

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under vacuum in this study. The ingots were homogenized at 1250 ℃ for 2 h, and then forged into rods with diameter of 16 mm. The forged rods were austenized at 1200 ℃ for 30 min, and water quenched to get martensite structure.

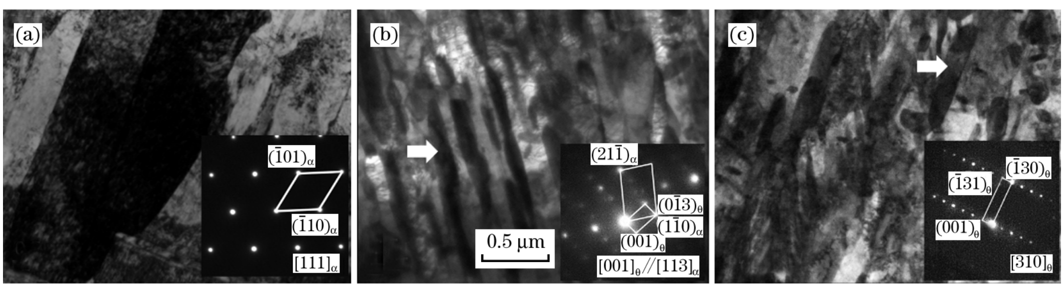
The in-situ TEM specimen preparation included two steps. First, prepare the samples as traditional TEM foils with 3 mm in diameter. Second, cut the foils, and transfer the cut foils onto in-situ TEM heating chips using focused ion beam (FIB). During the traditional TEM foils preparation, the samples were mechanically ground to about 60 μm, and twin-jet polished in a solution of 7 vol. % perchloric acid and 93 vol. % alcohol at about −20 ℃. The thin foils were characterized by TEM before being transferred onto in-situ heating chips. Several areas of interest, such as interfaces and defects, were selected before being further studied during the in-situ TEM experiment.

The microstructures of the as-quenched and heated samples were investigated by TEM. The in-situ microstructural evolution and Mn distributions were experimentally observed by scanning transmission electron microscopy (STEM) and energy-dispersive X-ray spectroscopy (EDS) using FEI Titan G2 80-200 ChemiSTEM with a probe corrector.

In this experiment, the in-situ samples could be heated at a large heating rate, since the size of the specimens was 30 μm×5 μm, which was very small compared to the whole heating area. In order to simulate bulk condition, the thickness of the in-situ samples was prepared to be around 0.5 μm. The maximum heating rate could reach 200000 ℃/s, and the heating process could be programmed. In this work, two different heating rates, 500 ℃/s and 10 ℃/min, were applied. One sample was tempered at 500 ℃ for 22 min at a heating rate of 500 ℃/s, and the other sample was tempered at 500 ℃ for 40 min at a heating rate of 10 ℃/min. During tempering at 500 ℃, the samples were quenched three times to acquire Mn mapping information.

2 Results and Discussion

The microstructures of 0.2 mass% C-5 mass% Mn steel before and after tempering at 500 ℃ were characterized by using TEM. Fig. 1(a) shows that the initial microstructure of as-quenched sample was martensite lath without any cementite, indicating that the cementite in the tempered sample was formed during tempering process. The microstructure of the sample tempered at 500 ℃ for 40 min at the heating rate of 10 ℃/min is shown in Fig. 1(b), and the precipitate marked by the arrow was identified as the cementite phase based on the selected area electron diffraction (SAED) patterns. The SAED pattern of the cementite and the adjacent matrix, as shown in the inset in Fig. 1(b), demonstrates that the orientation relationship between cementite and the matrix ferrite was $[001]_\theta // [113]_\alpha$ and $(0\bar{1}3)_\theta // (1\bar{1}0)_\alpha$. Fig. 1(c) shows the microstructure of the sample tempered at 500 ℃ for 22 min at the heating rate of 500 ℃/s. The cementite was also detected in this sample and verified by the SAED patterns.



(a) As-quenched sample; (b) Sample tempered at 500 ℃ for 40 min at a heating rate of 10 ℃/min; (c) Sample tempered at 500 ℃ for 22 min at a heating rate of 500 ℃/s.
Arrows in (b) and (c) point to the cementite corresponding to the inset SAED patterns.

Fig. 1 Microstructure of 5Mn steel

Fig. 2 shows the microstructural evolution and Mn partitioning during in-situ tempering at 500 ℃ at the heating rate of 10 ℃/min. Since the EDS detectors do not work at 500 ℃ due to the signal saturation, the Mn mapping cannot be achieved at 500 ℃. In order to acquire the Mn distribution of the sample at 500 ℃, the sample was quenched to room temperature right after tempered at 500 ℃ for a certain amount of time. Due to the very slow diffusion rate of Mn at room temperature (RT), the distribution of