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In-situ microstructural evolutions of 5Mn steel at elevated temperature in a transmission electron microscope

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ABSTRACT

The microstructural evolutions of 5Mn steel during various heat treatments have been investigated by in-situ transmission electron microscopy (TEM). The specimen of 5Mn steel was prepared using focused ion beam (FIB) milling, which allowed the selection of specific morphology of interest prior to the in-situ observation. The complete austenization at 800 ℃ was verified at the atomic scale by minimizing thermal expansion and sample drift in a heating holder based on micro-electro-mechanical-systems. During annealing at 650 ℃, the formation of reverted austenite was dynamically observed, while the morphologies of austenite laths of 5Mn steel after in-situ heating were quite similar to that after ex-situ intercritical annealing. During annealing at 500 ℃, the morphological evolution of cementite and associated Mn diffusion were investigated. It was demonstrated that a combination of FIB sampling and high temperature in-situ TEM enables us to probe the morphological evolution and elemental diffusion of specific areas of interest in steel at high spatial resolution.

1. Introduction

Recently, the 0.2 mass% C-5 mass% Mn steels (5Mn steel) have drawn a lot of attentions due to their extraordinary mechanical properties and potential applications in automobile industry[1,2]. In order to understand the structure-property relationship of 5Mn steels, the microstructural evolution and elemental diffusion under different heat treatment procedures have been carried out[3-13]. Moor et al. [3] and Lee et al. [4] reported that Mn partitioning was a key factor in stabilizing a high volume fraction of retained austenite in such steels at room temperature (RT), which improved the mechanical properties of 5Mn steels. Mn diffusion between austenite and ferrite during intercritical annealing[5-8], as well as the precipitation and coarsening behaviors of cementite[9-12], has also been investigated. Nonetheless, in most of the previous studies, ex-situ methods were applied to examine the microstructural evolution of 5Mn steels. Ex-situ experiments cannot dynamically track the phase transformation and elemental diffusion in the 5Mn steel during heat treatments. It is not possible to choose an area of interest containing specific microstructural features like grain boundaries, packet interfaces, lath interfaces, precipitates and see how a specific microstructure evolves at elevated temperature.

In-situ heating experiments in a transmission electron microscope (TEM) allow real-time observations of heat treatments of steels, including grain growth[14], recrystallization[15], dislocation propagation[16] and elemental diffusion[17]. Instead of performing heat treatments outside the microscope and then observing the samples post-mortem, in-situ microscopy allows direct observations of the dynamic behavior of steels under a variety of annealing conditions. In traditional in-situ heating TEM experiments, the spatial resolution was dramatically suppressed due to the sample drift induced by thermal expansion when an entire 3 mm-diameter TEM specimen was heated. Recently, the state-of-the-art in-situ TEM has been developed to study the microstructures at the atomic scale by using micro-electro-mechanical systems (MEMS)-based heating stages[18-20], which generally requires specimen prepared by focused ion

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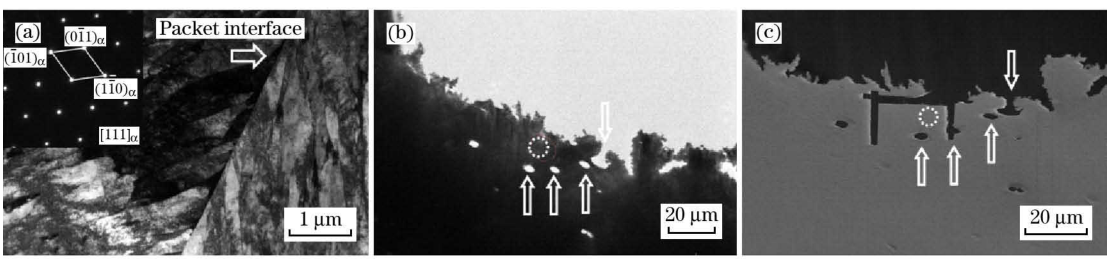
beam (FIB) milling[21-23]. However, the traditional lift-out lamella prepared by FIB cannot precisely choose the area of interest, since the lamella was buried in the bulk materials before lifting out.

In this paper, FIB was used to cut out the specific area of interest from a regular 3 mm-diameter TEM sample of 5Mn steel after observing its microstructure in a TEM. Various in-situ observations were carried out on 5Mn steel during different heat treatment procedures in order to understand the phase transformation of 5Mn steel at different temperatures. Benefiting from an advanced MEMS-based heating system, the microstructural evolution in 5Mn steel can be observed at the atomic scale with minimum sample drift, while in-situ elemental diffusion can also be detected. In the example of 5Mn steel, it was demonstrated that the in-situ heating TEM experiments are increasingly able to provide fundamental information for understanding the microstructural evolution at the atomic scale and the elemental diffusion during the phase transformation.

2. Experimental Procedure

The steel with a chemical composition of 0.2 mass% C-5 mass% Mn was investigated. The sample was first prepared under vacuum using an induction furnace, then homogenized at 1250 ℃ for 2 h before forged into rods with diameter of 16 mm. Next, after austenization at 1200 ℃ for 30 min, the forged rods were water quenched to form complete martensite microstructure.

In order to prepare the in-situ TEM specimen with the specific area of interest, a conventional TEM specimen with 3 mm in diameter was first prepared. The sample was mechanically ground to 50 μm in thickness, then the sample foils were twin-jet polished in a solution of 7 vol. % perchloric acid and 93 vol. % alcohol at −20 ℃. After locating the area of interest in a TEM, the in-situ TEM specimen using Zeiss Auriga FIB was prepared. For example, Fig. 1(a) shows the microstructure and the selected area electron diffraction (SAED) patterns of the area of interest in an as-quenched sample, including martensite laths and packet interfaces without any cementite and retained austenite. As shown in the low-magnification TEM image in Fig. 1(b), the area of interest was marked by the white circle. In order to position the area of interest, the holes formed during twin jet thinning and the edge morphology of the TEM specimen were selected as the markers and indicated by the white arrows. As is shown in the scanning electron microscope (SEM) image of the corresponding sample in Fig. 1(c), the area of interest was also marked by the white circle and positioned by the corresponding markers as indicated by the white arrows. Here the relatively thick area was selected for in-situ experiments, since the microstructural evolution in the bulk materials during heat treatments takes place in the relatively large volume. Using FIB milling, the lamella with the size of 25 μm × 10 μm including the area of interest was lifted out and transferred onto the chips of DENS solutions in-situ heating holder. The MEMS-based holder provided an elevated temperature environment without reducing the resolution of a TEM. During in-situ heating TEM experiments, the microstructural evolutions and elemental mapping of 5Mn steel were carried out in FEI cubed Titan 80−300 with an image corrector, FEI Titan G2 80-200 ChemiSTEM equipped with a probe corrector and the SuperX energy dispersive spectrometry (EDS) system with the four Bruker Silicon Drift Detectors, and FEI G2 20.



(a) Bright-field transmission electron microscope images and a selected area electron diffraction pattern from an area of interests including packet interfaces and lath interfaces; (b) Low-magnification TEM images of the interested areas; (c) Scanning electron microscopy images of corresponding interested area cut by focused ion beam milling.

Fig. 1. Locating interested areas of as-quenched 5Mn steel using TEM and FIB milling.

3. Results and Discussion

In order to better understand phase transformation of 5Mn steel at different temperatures and determine the typical temperature for various heat treatments, the phase diagram of 5Mn steel was calculated using Thermo-Calc software, as shown in Fig. 2. Based on the calculated phase diagram, in-situ heat treatments at three annealing temperatures were carried out as marked by three stars in Fig. 2. Firstly, when the annealing temperature was set to