Special Feature: Metallic Materials

Research Report

In-situ Observation of Microstructural Changes of Steel during Hot Deformation

Yasuhiro Yogo, Hirohisa Takeuchi, Kouji Tanaka, Noritoshi Iwata and Takashi Ishikawa

Report received on Jul. 3, 2012

ABSTRACT An in-situ observation method for microstructures at high temperature is developed. The newly developed observation system is composed of two units, a hot deformation type thermo-mechanical simulator, which can control the heating condition, atmosphere, hot deforming condition, and cooling condition, and a confocal scanning violet laser microscope. Two observation results, grain growth at high temperature and dynamic recrystallization, conducted by the developed method are shown. The results show that the new observation system can observe revealed grain boundaries and their dynamic changes at high temperature.

KEYWORDS In-situ Observation, Laser Microscope, High Temperature, Grain Growth, Recrystallization, Steel

1. Introduction

In conventional studies, researchers have measured the relationships between grain size and holding time at various high temperatures to comprehend the microstructural changes.[1,2] In the case of using the conventional observation method for an austenite structure, a specimen is first heated to a predefined temperature and maintained at that temperature for a predefined time. The specimen is then cooled to room temperature by water or He gas. Next, the specimen is cut, and the cut plane is polished to a mirrored surface and etched by an etching agent. Finally, the grain size is quantified. With the conventional observational method, many specimens are required to measure the relationships between grain size and heating condition.

With respect to the conventional observation method, there is a difficulty to measure and understand microstructural changes at high temperatures. It is that dynamic changes of microstructure at high temperature cannot be observed, and observation is usually carried out at room temperature. Therefore, dynamic changes of microstructure are inferred by the observation results at room temperature. In order to overcome the difficulty, a new method for observing microstructural changes at high temperature is developed. In this paper, the developed method is described in detail. In addition, two observation results, grain growth at high temperature and dynamic recrystallization, are shown.

2. New In-situ Observation System

In order to observe microstructural changes at high temperature, the observation method must overcome two difficulties: (1) the way to reveal the grain boundary and (2) the way to observe austenite structure at high temperature.

For revealing grain boundary, the thermal etching method [3, 4] is applied to the new observation method. The thermal etching method can reveal austenite grain boundary at high temperature, because atoms diffuse and vaporize into the atmosphere from the grain boundary at a much higher rate than from the rest of the specimen, which leaves grooves that can be measured to determine the boundary. The quantified results using the thermal etching method are the same as the quantified results using the conventional observation method, which proves that the thermal etching method is reliable.[5]

The new observation system is composed of two units, a hot deformation type thermo-mechanical simulator, THERMECMASTOR-Z, which can control

the heating condition, atmosphere, hot deforming condition, and cooling condition, and a confocal scanning violet laser microscope, VL2000DX.[6,7] **Figure 1** shows the schematic layout of the new observation system. A specimen is heated by joule heating and the temperature is controlled with a type R thermocouple. The distance from the surface of a specimen to the observation window is very close, so that is exposed to a large amount of radiation heat. Therefore, to protect the observation window from extreme heat, the window is cooled by a water-filled tube. In order to obtain clearer images, the surface of each specimen is polished to a mirrored surface, i.e., 0.1 z. **Figure 2** shows the geometry of the specimen.

At high temperature, atoms are diffused and vaporized into the atmosphere in the chamber from the surface of the specimen. The diffusion and vaporization of atoms reveals grain boundaries, but these atoms also obstruct the view through the observation window. Therefore, during the observation Ar gas is blown against the observation window, both to prevent the deposition of diffused and vaporized atoms and to cool the window. These configurations of the new observation system and the atmosphere treatment make it possible to observe the revealed grain boundaries at high temperature.

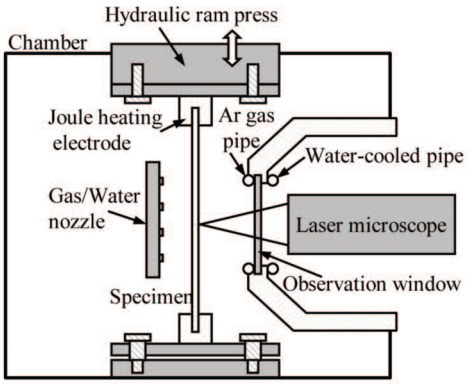
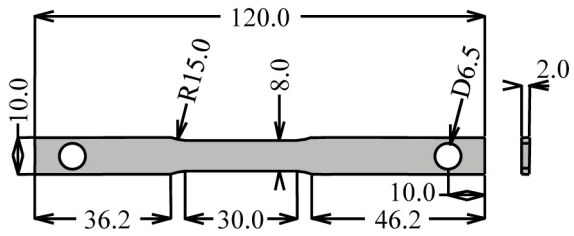


Fig. 1 Schematic diagram of the in-situ observation system.

(Dimensions are in mm.)



Surface roughness on observation side: 0.1 z
Material: S 25 C

Fig. 2 Specimen geometry used for the in-situ observation of hot deformation.

3. Experimental

3. 1 In-situ Observation of Grain Growth

Observations of austenite structure were performed using the new observation system in order to observe austenite grain boundaries and their dynamic changes at high temperature. The specimen dimension was the same as that in Fig. 2, and the material was S 25 C (Fe-0.22 mass% C-0.19 mass% Si-0.51 mass% Mn). The specimen was first heated to 1523 K at a rate of 5 K/s, and then maintained at this temperature for 10 min. In-situ observation was carried out while maintaining the temperature, and observed image data were recorded at 15 Hz. **Figure 3** shows the changes of grain size at 1523 K. The figure shows that the new observation system can observe revealed grain boundaries and their dynamic changes at high

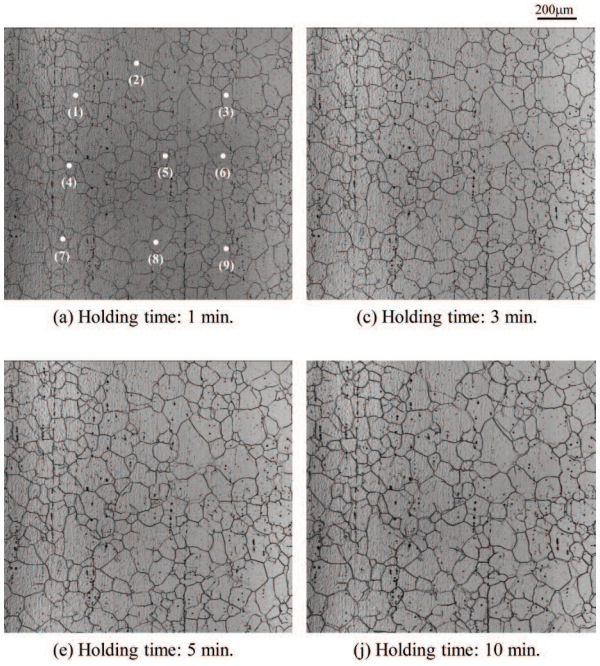


Fig. 3 Images of grain growth at 1523K.

temperature.

Figure 4 shows the average grain size and the sizes of specified grains. The specified grains, which are selected randomly, are shown in Fig. 3(a). The grain sizes quantified by the conventional method are also plotted in the figure. Concerning the conventional method, the experiments were carried out by the following procedure. The specimen was a cylindrical shape with a height of 12.0 mm and a diameter of 8.0 mm. The specimen was heated by induction heating to 1523 K at a rate of 5 K/s and was maintained at the temperature for 1 min and for 5 min. The specimen was then quenched by water to stop grain growth. The linear intercept method was applied to the quenched specimen to quantify the average grain size. Both the average grain size of the new observation method and that of the conventional method show good correspondence. The thermal etching method is expected to require time for the primary grain boundaries to disappear and to reveal the new grain boundaries. However, the correspondence of average grain size between the new observation system and the conventional method indicates that the velocity of the thermal etching method can follow the velocity of grain rearrangement in in-situ observation. The good correspondence also shows that the difference between the surface energy on a specimen surface and that in a specimen do not strongly influence grain growth of austenite. The average grain size increases gradually, even though the size of some of the specified grains increase or decrease drastically with time. These drastic grain size changes indicate shrinking and expansion of grains. The feature, that makes it possible to observe the grain size change of the same specimen in real time, is also able to analyze the shrinking and expansion of grains in greater detail.

The grain size changes, more than two hundred of grains were quantified by image processing, and a histogram of the grain size range and the count was generated (**Fig. 5**). Figure 5 does not contain the error derived from the difference in initial structure because only one specimen was used for the observation. From Fig. 5, the count under 40 microns decreases drastically, whereas the count over 100 microns increases gradually. In general, smaller grains have higher internal pressure. Therefore, in order to lower the Gibbs free energy, smaller grains would be absorbed by neighboring larger grains. This phenomenon is explained by the Gibbs-Thomson effect. Figure 5 supports the existence of this phenomenon.

3. 2 In-situ Observation of Dynamic Recrystallization

The experiment was carried out using the following procedure.[7] A specimen was first heated to 1473 K by joule heating at 5 K/s and then held at this temperature for 2 min. The specimen was then tensiled to 22.4% at

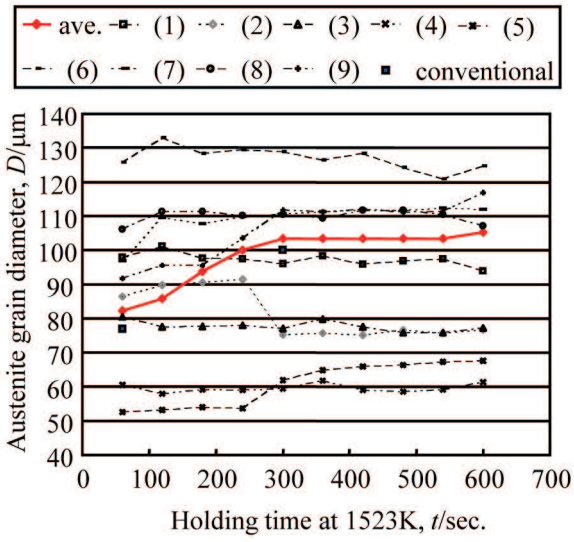


Fig. 4 Grain size change at 1523K.

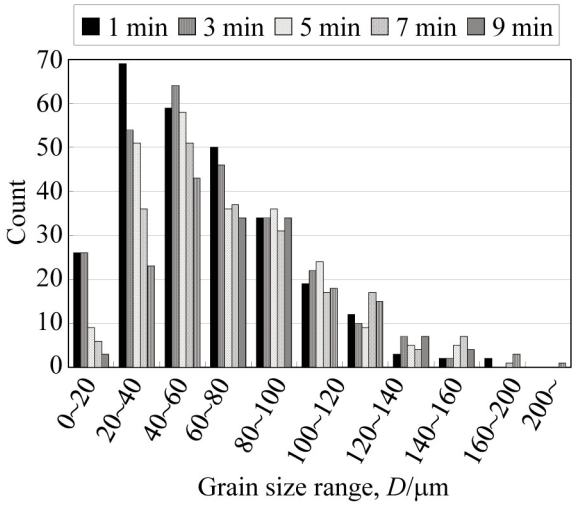


Fig. 5 Grain size histogram at 1523K.

the rate of 0.25 mm/min. In-situ observations were made during this process and images were recorded at 15 Hz. During the in-situ observation, the distance of the ram movement is also recorded, so the strain of observed images can be measured from the recorded distance.

Figures 6(a) and (b) show the microstructure before and after deformation under strain to 22.4%, respectively. The crosses are used as guide marks to identify the initial structure. Grain deformations are apparent by looking at the guide marks. The circled area in Fig. 6(b) is magnified in **Fig. 7** to highlight the microstructural changes.

Under tensiling at 1473 K, curved lines as deformed structures were spreading over the grain (Fig. 7(a)). Figure 7(b) shows the appearance of a new grain boundary. Figures 7(c) and (d) show that the new grain boundary has a sweeping motion inside the grain. In the area where the new grain boundary is swept, the deformed structures disappeared. The disappearance of the deformed structures would be caused by the dynamic recrystallization.

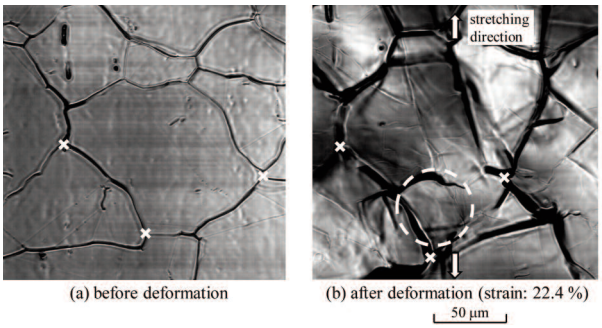


Fig. 6 Microstructural change under strain at 1473 K.

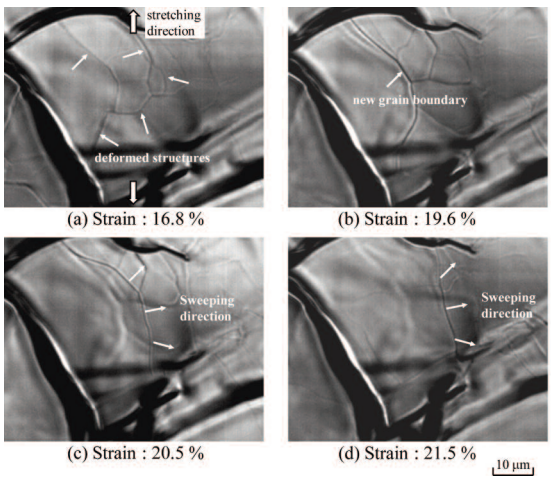


Fig. 7 Observations of deformed structures and the formation of a new grain boundary.

4. Summary

In order to conduct in-situ observation at high temperatures, the new observation method was developed. By using the in-situ observation method, two observations were performed. The first observation was carried out under a stable condition at 1523 K to observe grain growth. The second observation was carried out for observing microstructural changes under hot tensiling. In both observations, microstructural changes were clearly observed by the developed in-situ observation method.

References

(1) Sellars, C. M. and Whiteman, J. A.,"Recrystallization and Grain Growth in Hot Rolling", *Met. Sci.*, Vol.13, No.3-4 (1979), pp.187-194.

(2) Senuma, T., Yada, H., Matsumura, Y. and Futamura, T., "Structure of Austenite of Carbon Steels in High Speed Hot Working Processes", *Tetsu to Hagane* (in Japanese), Vol.70, No.15 (1984), pp.2112-2119.

(3) Mullins, W. W., "Theory of Thermal Grooving", *J. Appl. Phys.*, Vol.28, No.3 (1957), pp.333-339.

(4) Mullins, W. W., "The Effect of Thermal Grooving on Grain Boundary Motion", *Acta Metall.*, Vol.6 (1958), pp.414-427.

(5) De Andres, C. G., Caballero, F. G., Capdevila, C. and San Martin, D., "Revealing Austenite Grain Boundaries by Thermal Etching: Advantages and Disadvantages", *Mater. Charact.*, Vol.49 (2002), pp.121-127.

(6) Yogo, Y., Takeuchi, H., Tanaka, K., Iwata, N., Nakanishi, K. and Ishikawa, T., "MRT Letter: In Situ Observation Method for Microstructural Changes of Steel During Hot Deformation", *Microsc. Res. Tech.*, Vol.72 (2009), pp.899-901

(7) Yogo, Y., Tanaka, K. and Nakanishi, K., "In-Situ Observation of Grain Growth of Steel at High Temperature", *Mater. Trans.*, Vol.50, No.2 (2009), pp.280-285.

Figs. 1, 2, 6 and 7
Reprinted from Microscopy Research and Technique, Vol.72 (2009), pp.899-901, Yogo, Y., Takeuchi, H., Tanaka, K., Iwata, N., Nakanishi, K. and Ishikawa, T., MRT Letter: In Situ Observation Method for Microstructural Changes of Steel During Hot Deformation, © 2009 Wiley-Liss, with permission from John Wiley and Sons.

Figs. 3-5
Reprinted from Materials Transactions, Vol.50, No.2 (2009), pp.280-285, Yogo, Y., Tanaka, K. and Nakanishi, K., In-Situ Observation of Grain Growth of Steel at High Temperature, © 2009 The Japan Institute of Metals, with permission from the Japan Institute of Metals.

Yasuhiro Yogo
Research Fields:
- Metal Forming
- Heat Treatment
- Physical Metallurgy
- Computer Aided Engineering

Academic Degree: Dr.Eng.
Academic Societies:
- The Japan Society for Technology of Plasticity
- The Iron and Steel Institute of Japan
- The Japan Society for Heat Treatment

Hirohisa Takeuchi
Research Fields:
- Heat Treatment
- Computer Aided Engineering

Academic Societies:
- The Iron and Steel Institute of Japan
- The Japan Society for Technology of Plasticity

Kouji Tanaka
Research Fields:
- Physical Metallurgy
- Steels
- Metals and Alloys

Academic Degree: Dr.Eng.
Academic Societies:
- The Iron and Steel Institute of Japan
- The Japan Society for Heat Treatment
- The Minerals, Metals & Materials Society

Awards:
- The Japan Institute of Metals Technical Development Award, 1996
- R&D100 Award, 1998
- 11th GTSJ Dissertation Awards, 2002

Noritoshi Iwata
Research Field:
- Metal Forming

Academic Degree: Dr.Eng.
Academic Society:
- The Japan Society for Technology of Plasticity

Takashi Ishikawa*
Research Field:
- Metal Forming

Academic Degree: Dr.Eng.
Academic Societies:
- The Japan Society for Technology of Plasticity
- The Iron and Steel Institute of Japan
- The Japan Institute of Metals
- The Japan Society for Precision Engineering
- The Japan Institute of Light Metals

Awards:
- JSTP Best Paper Awards, 1978
- JSTP AIDA Awards, 1990
- ISIJ Nishiyama Awards, 1996
- JSTP AIDA Medal, 2004
- ISIJ Academic Achievement Awards, 2006
- JSHT Best Paper Awards, 2011

*Nagoya University