Developments of hybrid in-situ observation system to study the microstructural change of metallic alloys†

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1. Introduction

Rapid heating and cooling is one of the characteristics of welding conditions. The metallic alloys under such thermal conditions show non-equilibrium behavior of phase transformation (PT). The non-equilibrium behavior of PT results in unexpected microstructures at room temperature. Then understanding such a behavior is essential for controling the microstructure of a weld accurately.

A typical instance of non-equilibrium PT was presented by Vitek et al in the case of solidification process of stainless steel [1]. When the cooling rate increased, the solidification path was changed from delta-ferrite region to austenite region. Then the quantity of delta-ferrite at room temperature was unexpected in the case of rapid cooling. In that case, the solidification path was estimated from the quantity of the delta-ferrite at room temperature. On the other hand, Babu et al [2] has directly confirmed that the primary phase of solidification depended on the cooling rate by using a time-resolved X-ray diffraction (TRXRD) technique. The primary phase of Fe-C-Al-Mn steel changed from delta-phase to austenite phase for rapid cooling conditions. This work shows that the in-situ observation technique is useful to trace the PT under the rapid cooling/heating conditions. The TRXRD technique gives valuable information about PT, such as phase identification and element partitioning during phase transformation. On the other hand, the optical microscope technique is suitable for getting the information about the microstructural morphology and origin of microstructure growing. Especially, laser scanning cofocal microscopy (LSCM) system is appropriate for tracing of PT under the thermal cycle designed [3]. The system consists of LSCM and an infrared furnace. The infrared furnace possesses good response of temperature.

In order to use all the features a described above, our research group has been developing a hybridTRX RD/LSCM system to trace the PT [4]. In the present work, the detail of the hybrid system is described. Furthermore, the application of the hybrid TRXRD/LSCM system to trace the PT of metallic alloys, such as steel alloy is described.

2. Hybrid TRXRD/LSCM system

Figure 1(a) shows a photograph of the experimental setup on the 46XU beamline at SPring-8 in Hyogo, Japan. The infrared furnace was set on the theta axis of a goniometer situated within the hatch of the beamline. In this system, the head of a laser scanning confocal microscope (LSCM) was also set by fitting the theta axis, as shown in the photograph. The focus point of the LSCM is on the surface of the observed sample, which is set in the furnace. A two-dimensional pixel detector (Pilatus2M) was placed. The incident beam, an ultra bright X-ray, was introduced into the furnace and the diffractions were recorded by the pixel detector with high time resolution. Simultaneously, the microstructural changes were observed through the LSCM in situ. The specimens, 5 mm in diameter and 1 mm thick, were placed in a boron-nitride (BN) crucible in which the X-ray absorption is quite small. The crucible was held by a platinum holder, which was inserted in the furnace. The temperature was measured by a thermocouple incorporated into the crucible holder. The specimens were placed at the focal point of a halogen lamp. The temperature controller, which was connected to a personal computer (PC), the

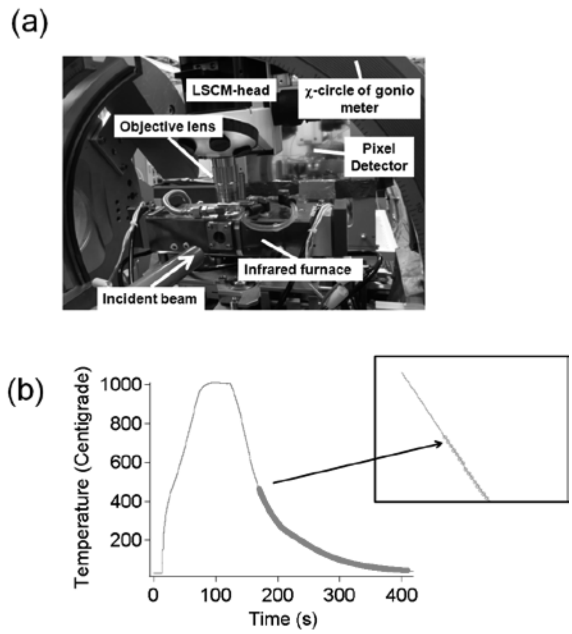

Fig. 1 Hybrid TRXRD/LSCM system at the 46XU beamline of SPring8.

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thermocouple and the halogen lamp in the furnace were placed outside the beamline hatch. When the thermal cycles that simulate welding were programmed on the PC, the profiles were sent to the temperature controller, which reproduced the desired thermal cycles by switching the halogen lamp on and off, based on the measured temperature. Both the maximum heating and cooling rates of the system were 37 K/s. The LSCM head enables in situ observations of microstructural changes at 30 frames/s at high temperatures. A CCD camera was connected to the PC located outside the hatch and the images were stored at a rate of 30 frames/s. The control program could trigger the temperature controller, the X-ray shutter, the x-y-z stages on theta axis, goniometer-axis control and the exposure of the pixel detector.

Before the measurements, the specimen position was adjusted in the manner explained in the following section. Then, the theta axis was tilted to a fixed angle (5° in the present study). The temperature controller was then triggered at a set time, and the exposure of the detector was activated with the time resolution of 0.2 s. TRXRD data and LSCM images under the desired thermal cycles were obtained simultaneously.

The undulator beam was monochromatized by the double Si crystal, and a 30 keV of X-ray energy was used. The X-ray was introduced into the hatch through a mirror; The X-ray beam was shaped by the slit; the dimensions of the beam were 0.5 mm × 0.5 mm. Before the measurements, the position of the sample surface was adjusted. By controlling the z-stage, the sample surface is forced to be in the middle of the beam height. The theta axis was then rotated by 5°. The camera length was 468.79 mm. The detector dimensions were 1475 pixels (one pixel has an area of 0.172 mm^2) × 1679 pixels in the 2θ direction. The PT of low temperature transformation steel (LTT) was analyzed in the present work. Fig. 1(b) shows thermal cycle applied and the circle on the temperature profile means the X-ray exposure point.

3. Results and Discussions

Figure 2 shows snapshots of LSCM images of PT of LTT material under the cooling cycle. The austenite was super-cooled until 300 Centigrade as shown in Fig. 2(a). The austenite had large driving force of PT and changed the transformation mechanism from diffusive to displacive since diffusion is impossible at low temperature. As shown in Fig. 2(b), the clear contrast appeared in the LSCM images. The contrast increase as temperature decreased as shown in Fig. 2(c). The clear contrast corresponds to surface relief of martesitic microstructure. Finally, the martensitic transformation stopped as shown in Fig. 2(d). The displace transformation discretely occurred along the decreasing of temperature. As shown in this instance, the contrast of image included many information about the mechanism of PT.

Figure 3 shows d-spacing, intensity, time and temperature diagram during PT of LTT. The figure shows reflections of γ111, α'110, γ200, α'200, γ220, α'211, γ311, γ222 and α'220 starting from the right. The diffraction data were analyzed using the method and programming described in reference [5]. The mark on the temperature profile corresponds to that in Fig. 2. The 5 diffraction peaks of austenite transformed to diffraction peaks of martensite. The change was compatible to results in LSCM images. For instance, the analyzing the d-spacing change during PT gives the information about element partitioning during PT [5, 6]. Then it could be said that the developed hybrid system enable in-situ observation of PT of metallic alloys in real and reciprocal lattice space.

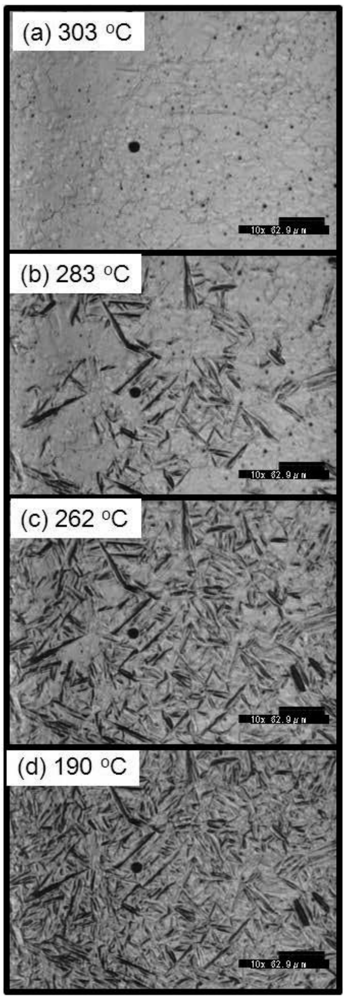

Fig. 2 Snap shots of PT of LTT under the cooling cycle.

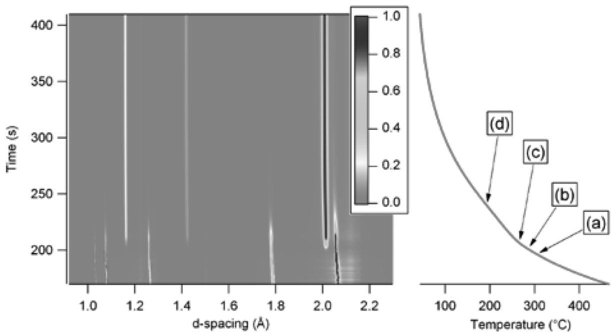

Fig. 3 d-spacing, intensity, time and temperature diagram.

4. Conclusions

The detailed configuration of the hybrid TRXRD/LSCM system was explained. The phase transformation behavior of low-temperature transformation steel was traced by using the system and it was shown that the hybrid system enable in-situ observation of phase trasnformation of metallic alloys in real and reciprocal lattice space.

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