Development of *In Situ* Observation Technique on Solidification Structure of Weld Metals by X-ray Diffraction

High-intensity heat sources used for fusion welding create steep thermal gradients in materials as they are rapidly heated and cooled to and from their melting points. The rapid thermal cycling that takes place induces melting and solidification in those parts of the weld where the liquidus temperature has been exceeded, as well as solid-state phase transformation on both heating and cooling during welding. Then, the weld metal suffers from a crack, known as a solidification crack, owing to the thermal strain in the solidification temperature range in which ductility remarkably decreases during welding. The solidification crack is a significant problem of austenite stainless steels during rapid cooling for industrial applications. *In situ* observation is essential for understanding these phenomena. X-ray diffraction analysis is often utilized as an *in situ* technique. In the case of welding, the physical dimensions of the weld depend on the welding conditions and transformation kinetics, and an X-ray probe must have a beam size finer than the dimensions of the transformation region. In this work, the experimental setup for time-resolved *in situ* observation of phase transformation during solidification and cooling (10 to 10^4 K/s) in a welding process using synchrotron radiation and the result for several kinds of steel welds are presented.

In our research group, an *in situ* phase identification system combined with an undulator beam and an imaging plate has recently been utilized at **BL46XU** beamline [1]. Furthermore, a two-dimensional pixel detector that the SPring-8 detector team has been developing in collaboration with the Paul Scherrer Institute (PSI) in Switzerland [2] was used to exclude the influence of preferred orientation as shown in Fig. 1. A photon energy of 18 keV was selected to maximize the number of observable diffraction peaks in the 2θ window. The gas tungsten arc-welding torch was moved in parallel to the upper surface of the specimen, which was set on the anode. These make it possible for phase transformation to be identified in real-time under conditions of one-directional solidification and a spatial resolution of 0.1 × 0.3 mm.

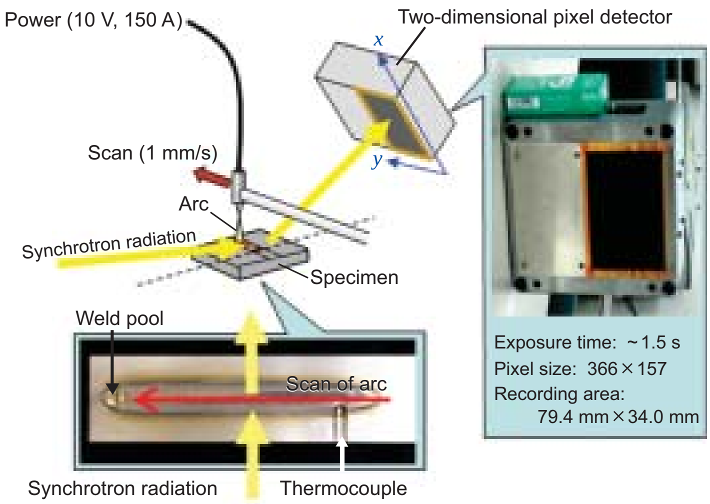

Fig. 1. Experimental setup for time-resolved *in situ* observation.

Figure 2 shows an example of X-ray diffraction pattern on the imaging plate obtained in the welding and an SEM observation of the rapid cooling microstructure of the stainless steel (Fe-20%Cr-14%Ni). The shift of the diffraction peak, mist-like pattern and periodic pattern of the primary γ phase were observed, suggesting an increase in crystallinity, the appearance of the nucleus of dendrites and the rotation of crystallites due to a drop in temperature. The crystal growth of γ phases was observed in detail with a time resolution of 0.1 s or less.

Figure 3 shows the change of the two-dimensional diffraction pattern for the austenite stainless steel (high alloy) (a-d) that is identical with the specimen in Fig. 2 and hypoeutectoid steel (low alloy) (e-h) during welding. In the high alloy, the diffraction patterns of the primary γ phase blink at a high temperature of more than 1400 ºC (a,b), suggesting the rotation of crystallites. The diffraction spots converge with the appearance of the secondary δ phase, suggesting the preferred orientation due to lattice matching. Consequently, the microstructure of the high alloy after solidification also has the preferred orientation normal to the specimen surface. In the low alloy, the δ, γ, and α phases appear at (e) about 1500 ºC, (f) 1450 ºC and (g) 600 ºC, respectively. Consequently, the diffraction peaks of residual γ and α phases mainly coexist. The comparatively random microstructure is formed as shown by the ring pattern (h).

This phenomenon corresponds to the miniaturization of the microstructure due to the solid transformation in the final stage.

This technique is quite useful for verifying experimentally whether ferrite or austenite is the primary phase that solidifies from the melt, and for following the dynamics of phase transformation during cooling in the fusion welds under a steep thermal gradient and non-isothermal heating and cooling conditions.

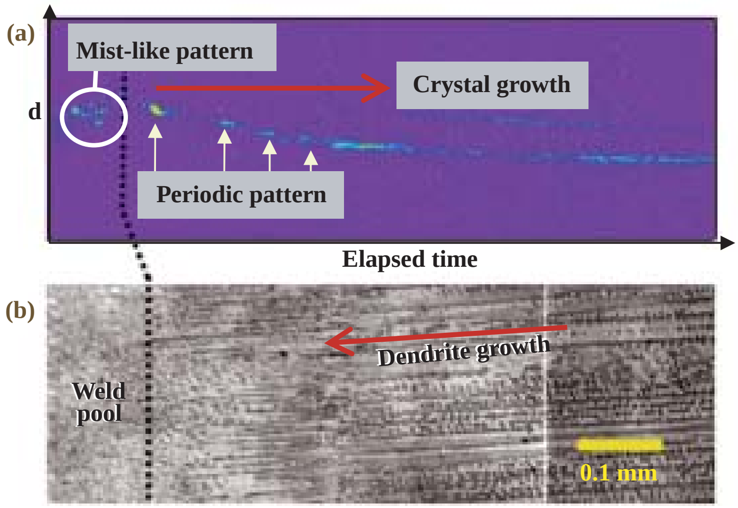

Fig. 2. X-ray diffraction peak on imaging plate and SEM observation of rapid-cooling microstructure.

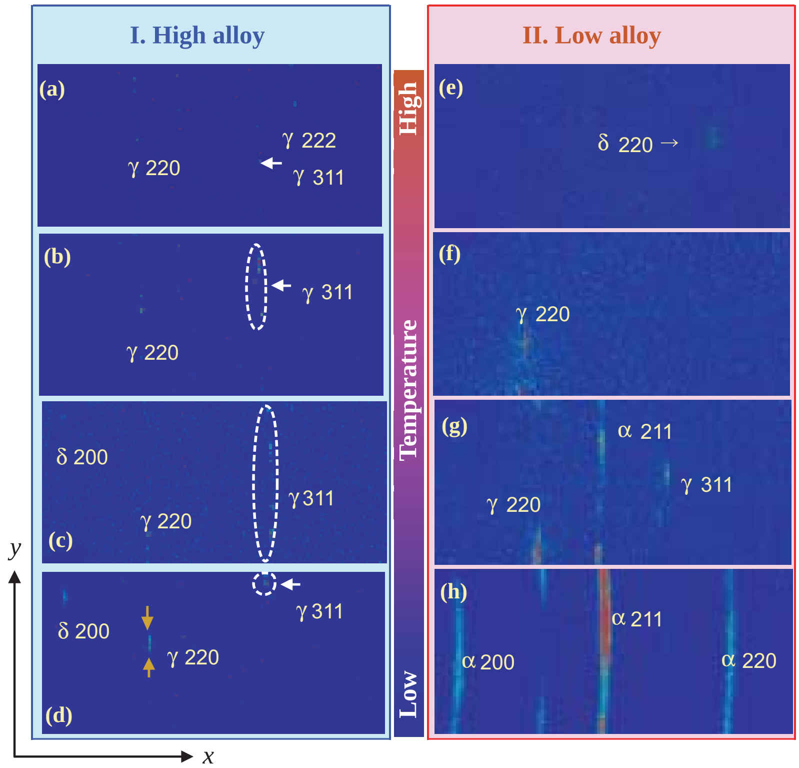

Fig. 3. Time-resolved two-dimensional diffraction patterns. The *x*- and *y*-axis corresponds to those in Fig. 1.

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References

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[2] H. Toyokawa: Abstract of The 8th SPring-8 Symposium (2004) p. 11.