In-situ Observation of Steel Weld Solidification and Phase Evolution using Synchrotron Radiation

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Abstract

In order to understand the microstructure changes during welding processes , aiming to design high intensity and tough weld structures, kinetic information about the phase transformation is essential. In our research group, in-situ observation systems consisting of undulator beam and imaging plate and two dimensional pixel detector have recently been used. The welding torch is driven by a stepping-motor in the system. These make it possible that phase transformation can be identified in real-time under the condition of directional-oriented solidification . In the present work, a combination of analyzing method: the in-situ observation system by X-ray diffraction technique using intense synchrotron radiation, and morphological observation by high-temperature laser scanning confocal microscopy is suggested to analyze the phase transformation during the welding process. Using the results acquired by these analysis methods, phase evolution of hypereutectoid carbon steel, during fusion welding was analyzed. The primary phase was directly identified as an austenite phase. Precipitation of pearlite phase was observed , during cooling followed by the martensitic transformation.

KEY WORDS: (Welding), (Phase transformation), (X-ray diffraction),(In-situ observation), (Synchrotron radiation), (Solidification)

1. Introduction

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. In welding processes where steep thermal gradients exist in the material, no direct methods are available for investigating the phase transformations that take place. For example, conventional methods for studying general phase transformation behavior such as calorimetry, dilatometry, and resistivity are indirect in that they measure the response of the sample (changes in enthalpy, length, resistivity, or hardness) to the imposed thermal cycle and do not in any way determine the phases that are present during the test.

Furthermore, these methods only provide phase transformation data for heating and cooling rates of the order of 1 K/s , which is much less than those in welding (10 to 10^4 K/s). In the absence of phase transformation information during welding , the microstructure and integrity of the weld cannot be accurately predicted.
Neutron and X-ray diffraction are two popular direct measurement techniques used to determine the structural factor of materials. Neutron scattering requires large samples(≈10 mm diameter or greater) and extended exposure times(several hours) to a high flux radiation source. On the other hand, X-ray diffraction studies are more suited to smaller specimens and require shorter exposures to the radiation. However, the physical dimensions of the weld depend on the welding conditions and the transformation kinetics, and an X-ray probe must have a beam size finer than the dimensions of the transformation region. Conventional X-ray sources do not have sufficient intensity for real-time measurement in such a small area, whereas synchrotron radiation, by virtue of its intensity and narrow natural collimation, is superior for such experiments. In this work, the experimental setup for time resolved in-situ observation of phase transformation and microstructural evolution during solidification and cooling in welding process, using synchrotron radiation and the result for several kinds of steel welds are presented.

2. Quenching in liquid-tin bath

Problems due to non-equilibrium temperature changes during either heating or cooling, can occur in the use of these materials. The material in the fusion welding process, for example, is heated and continuously cooled through the liquid phase and hot cracking due to segregation of minor elements can occur during

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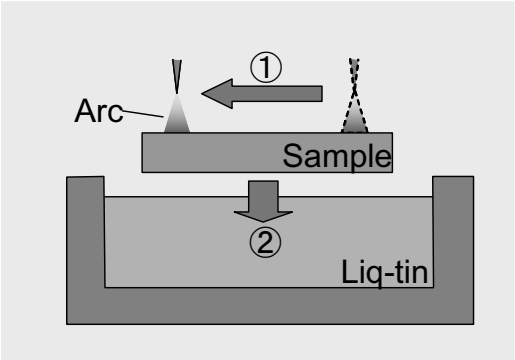

Fig.1 Schematic illustration of tin quench

solidification. Kinetic information about the phase transformations occurring in the solidification process is essential.

In order to acquire kinetic information about the phase transformations occurring in the welding thermal cycle, quenching using liquid-tin has been used.[1-4] This method freezes the welding bead in the liquid-tin bath, during welding and the diffusion phase transformation is stopped, as shown in Fig.1. Figure 2 shows the SEM image of quenched austenitic stainless steel (Fe-20%Cr-11%Ni) with FA mode (solidification occurs as primary ferrite, then austenite forms at the end of solidification) to the temperature drop from liquid/solid interface[5]. It can be observed that dendrites are growing along one direction, the easy growth direction. Analyzing the quenched weld, phase transformation from the liquid phase can be identified. The thermal transformation, however, cannot be stopped. Furthermore, it was difficult to observe the crystallization timing of primary δ phase in the material of FA solidification mode. The experiments become time- consuming if high spatial resolution, i.e. high time-resolved observation for the solidification process, is used for identifying the phase.

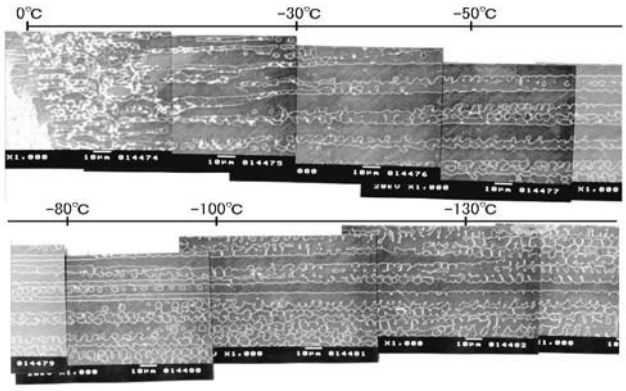

Fig.2 SEM image of austenitic stainless steel quenched in liquid-tin. Figures show temperature drop from liquid- solid interface.

3. In-situ time resolved X-ray diffraction using the imaging plate

In the late-nineties, more developed analysis methods for phase transformations during welding, using synchrotron radiation have been presented by Elmer et al[6]. Phase mapping in the quasi-stationary temperature field of pure titanium welding has been made in the spatial resolution of 250*500 μm. In their research, beta-phase near the solidification front was directly identified. The phase mapping for low-carbon steel[7] and duplex stainless steel[8] also have been presented and analyzed in quasi-stationary temperature fields. The measuring time of phase identification, for one region is 10 second with their experimental setup. So the welding torch and workpiece was translated with respect to the fixed X-ray beam, in order to probe discrete regions around the weld. This also results in time- consumption if high spatial resolution, i.e. high time-resolved observation for continuous cooling process, is used for identifying the phase. Elmer et al also presented the high time resolved X-ray diffraction investigation using synchrotron radiation[9-10], with spatial resolution of 730 μm ϕ and static welding, in which one-directional oriented solidification had not occurred. However, phase selection phenomena of primary phases due to the difference of cooling rate have been clearly shown.[10]

In our research group, in-situ phase identification system consisting of undulator beam and imaging plate have recently been used as shown in Fig.3[11]. Undulator beam line (BL46XU) in the third generation synchrotron radiation source, SPring8 (JASRI), was used. The water-cooled copper anode was set on the 4-axis goniometer set in the beam line, avoiding the heat effect on it. The weld material was set on the anode inclined at 20 degrees to the incident beam. A photon energy of 12 keV was selected to maximize the number of observable diffraction peaks in the 2θ window and to ensure that the photon energy was sufficiently high above the K edges for iron to minimize the K fluorescence contribution from the specimen. The gas tungsten arc-welding torch was moved in parallel to the upper surface of weld, using a stepping-motor driven stage. The torch speed, arc current

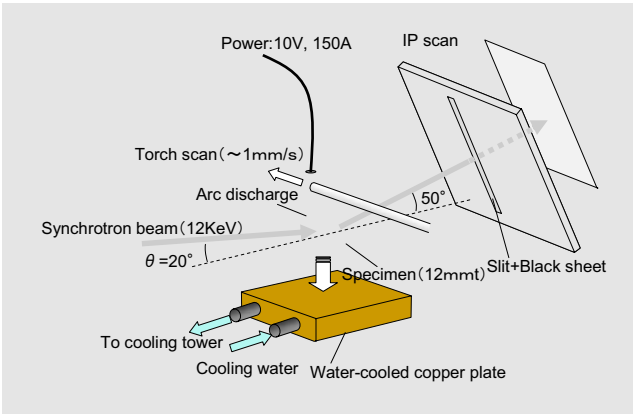

Fig.3 Schematic illustration of X-ray diffraction measurement

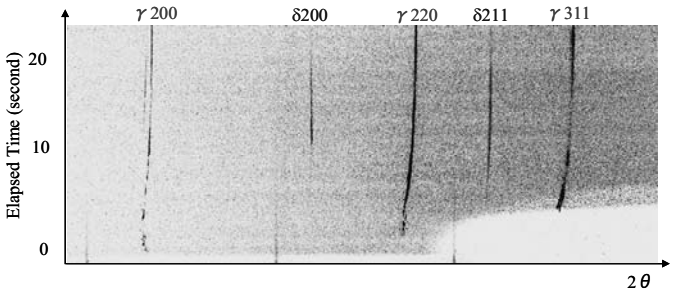

Fig.4 X-ray diffraction patterns from AF solidification mode

and voltage were set to 1 mm/s, 150 A and 10 V, respectively. These make it possible that phase transformation can be identified in real-time under the condition of one-directional oriented solidification and the spatial resolution of 100*500 μm. Figure 4 shows an example of X-ray diffraction peaks on the imaging plate obtained in the welding of the stainless steel (Fe-20%Cr-14%Ni) of AF solidification mode. The diffraction peaks of γ200、γ220 and γ311 show high intensities in the first stage of weld solidification followed by the formation of δ200 and δ211. This result showed that the austenite was the primary phase to solidify from the melt. As the weld cooled further, the austenite peaks shifted toward higher 2θ values, indicating a decrease in lattice spacing due to a drop in temperature, suggesting the enhancement of the crystallinity. This phenomenon was not observed in the diffraction profile from the specimen after liquid-tin quenching. It is impossible to catch the crystallinity in the microstructure after the solidification like liquid tin quenched metal as suspected. In contrast, the curvature of the diffraction profiles is not observed in the secondary δ phase though the crystallinity of the γ phase is enhanced greatly even after the appearance of δ211. The crystal growth of γ and δ phases was observed in detail with a time resolution of 0.1s or less. Displacement of crystallographic information (i.e. lattice constants and crystallite size) was observed quantitatively. However, it was difficult to exclude the influence of preferred orientation.

4. In-situ time resolved X-ray diffraction using a two dimensional pixel detector compared to the morphological observation in real space

A two dimensional pixel detector that the SPring-8 detector team has been developing in collaboration with Paul Scherrer Institut (PSI) in Switzerland [12] was used with elevation angle of 35 deg to exclude the influence of preferred orientation. The energy of a fixed X-ray beam was set to 18 keV. The gas tungsten arc-welding torch was moved in parallel to the upper surface of weld, using a stepping-motor driven stage.

Furthermore, a Laser Scanning Confocal Microscopy (LSCM) with infrared image furnace (IIF) was employed to make the in-situ real space observation of phase transformation. A similar thermal cycle to the TIG

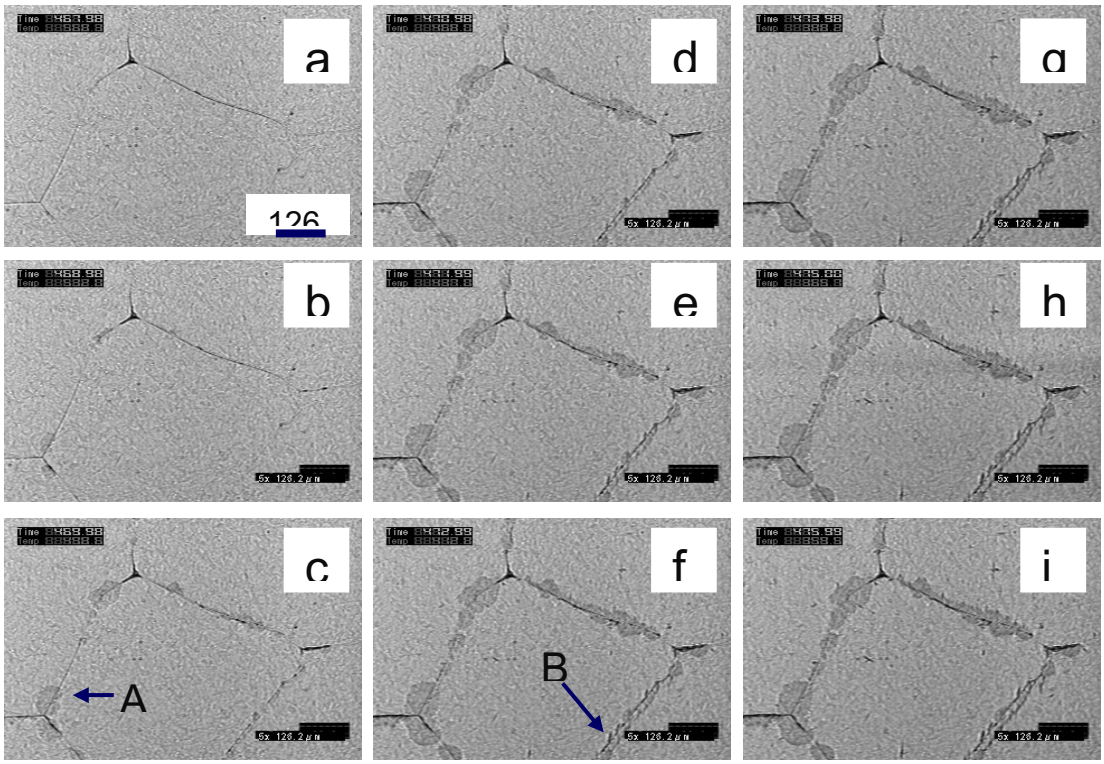

Fig.5 Observed images of hypereutectoid carbon steel by LSCM, in continuous cooling process showing that second phases nucleated and grew at triple points and grain boundaries.

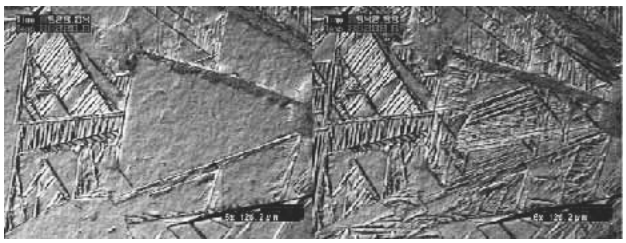

Fig.6 Observed images of martensitic transformation by LSCM. The packets, blocks and laths suddenly appeared in the grains.

welding was applied to the specimen. The system of high-temperature LSCM consists of LSCM and Infrared Image Furnace (IIF). Details of the LSCM with IIF and experimental procedure have been described elsewhere. [13] A sample, hypereutectoid carbon steel, was set in the IIF and continuously cooled along a heat history. The heat history set on the IIF-controller consists of two steps:

1. Heating up to 1400 centigrade (just below the melting point), at 200 K/min.
2. Continuous cooling along the heat history obtained in real welding conditions (measured by thermo-couple), from 1400 centigrade.

The grain boundary and triple point could be observed. In the continuous cooling process, it was observed that the second phases nucleated and grew at triple points and grain boundaries, as showed in Figure 5. Of course the crystal structure of the primary phase and the second phase cannot be identified by LSCM. In the further cooling process, the packets, blocks and laths suddenly appeared within the grain as showed in Figure 6. It can be considered Martensite transformation occurred. From these morphological observations, it was clear that the second phase nucleated and grew from the grain boundary of the primary phase, followed by martensite transformation, during the cooling process of weld.

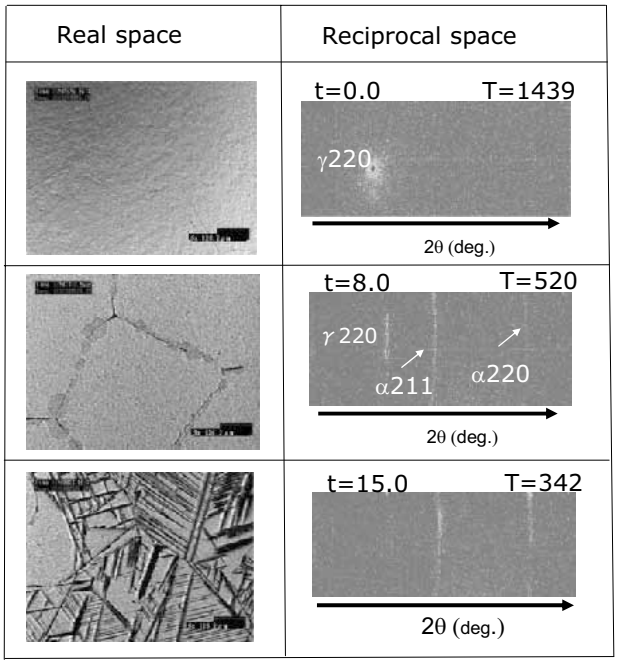

Fig.7 Precipitation of secondary phase in (a) real space and (b) reciprocal space

A combination of analyzing methods: the in-situ phase identification system, morphological observation with laser scanning confocal microscopy with infrared image furnace and observation of microstructure at room temperature by SEM and X-ray in the laboratory, is suggested.

Phase transformation of 0.8wt%C steel, during welding was analyzed as an example of these combined observations as shown in Fig. 7[13,14]. Precipitation of α phase from the primary γ phase was observed during cooling followed by the martensitic transformation. In the reciprocal space the γ220 was observed as a primary phase from the melt at the time of t=0. As the weld cooled further, the α phase was observed at the temperature of around 520 centigrade (t=8 sec), followed by the disappearance of the γ peak which indicates the martensitic transformation . It was difficult to distinguish the diffraction peaks of the cementite from those of ferrite and austenite because of the diffraction condition.

5. Concluding remarks

An in-situ time resolved X-ray diffraction technique using intense synchrotron radiation compared to the morphological observation in real space was established to investigate solidification and phase transformations during welding. This technique is quite useful to verify experimentally whether ferrite or austenite is the primary phase to solidify from the melt, and to follow the dynamics of phase transformation during cooling in the fusion welds under a steep thermal gradient and non-isothermal heating and cooling conditions.

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