Analysis of Primary Weld Solidification in Stainless Steel using X-ray Diffraction with Synchrotron Radiation †

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Abstract

The phase development through the weld solidification process was observed in order to acquire essential information for controlling weld micro structure. The diffraction pattern produced by synchrotron radiation (SPring-8 BL46Xu) was used to identify the crystallized phase. The austenitic stainless steel with FA mode was quenched in liquid Tin and analyzed. The experimental results showed primary crystallization growing and δ-γ solid transformation or peritectic crystallization between δ and γ phases.

KEY WORDS: (Weld solidification), (Phase transformation), (Synchrotron radiation), (K-S relation), (Peritectic crystallization)

1. Introduction

For controlling weld metal microstructure, phase information throughout the solidification process is essential. Furthermore, the microstructure development during solidification has been recently evaluated using computational thermodynamics and kinetic calculations[1]. In order to evaluate the model, experimental data for the phase development, as a function of temperature must be acquired.

Kou and Le[2] showed the weld solidification structure of stainless steels using a weld metal quenched by liquid Tin. They used optical microscopy and scanning electron microscope for phase observation. Lee[3] analyzed quenched weld beads of stainless steel based on the observation by optical microscopy and scanning electron microscope, and theoretical model. His result gave important information for decreasing the weld cracking of duplex stainless steel.

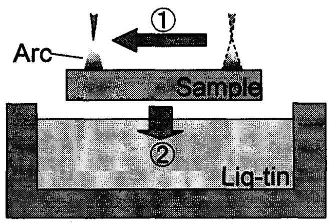

Fig. 1 Schematic illustration of Tin quench

In the present work, the diffraction pattern produced by synchrotron radiation (SPring-8 BL46Xu) was used to identify the crystallized phase. The austenitic stainless steel with FA mode was quenched by liquid Tin and analyzed. The temperature profile during solidification of the weld metal was also measured and the distance

Table 1 Chemical composition of stainless steel

C	Si	Mn	P	S	Ni	Cr	N	O
0.021	0.14	0.12	0.027	0.001	11.33	19.96	0.0007	0.005

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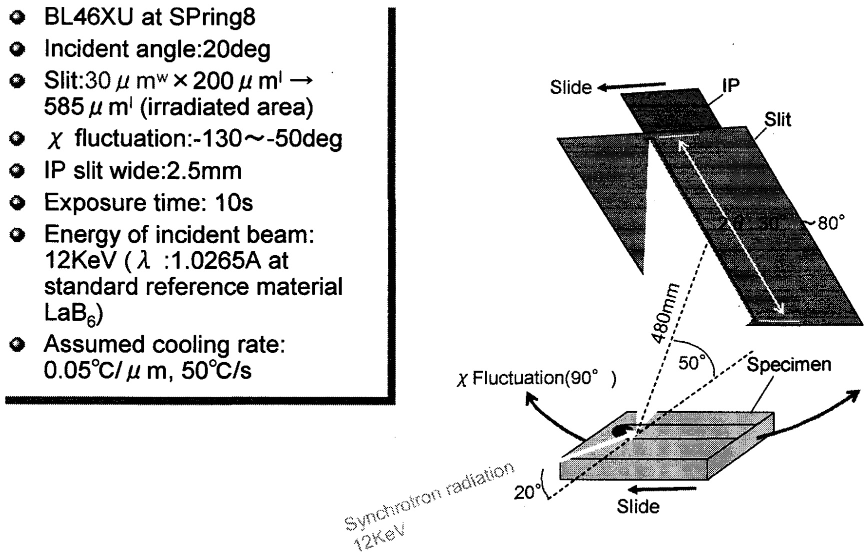

Fig. 2 Schematic illustration of X-ray diffraction measurement

from solid-liquid interface was transformed to temperature drop during solidification. Then the X-ray diffraction patterns for the temperature drop from liquid/solid interface could be acquired during the weld solidification process.

Those diffraction patterns showed primary crystallization growing and δ-γ solid transformation or peritectic crystallization between δ and γ phases.

2. Experimental method

For space-resolved measurement, the synchrotron radiation has suitable characteristics, high brilliance and parallel. In our experiment, X-ray with 12 KeV energy (BL46Xu at SPring8) was used as the incident beam. The beam was introduced to quenched austenitic stainless

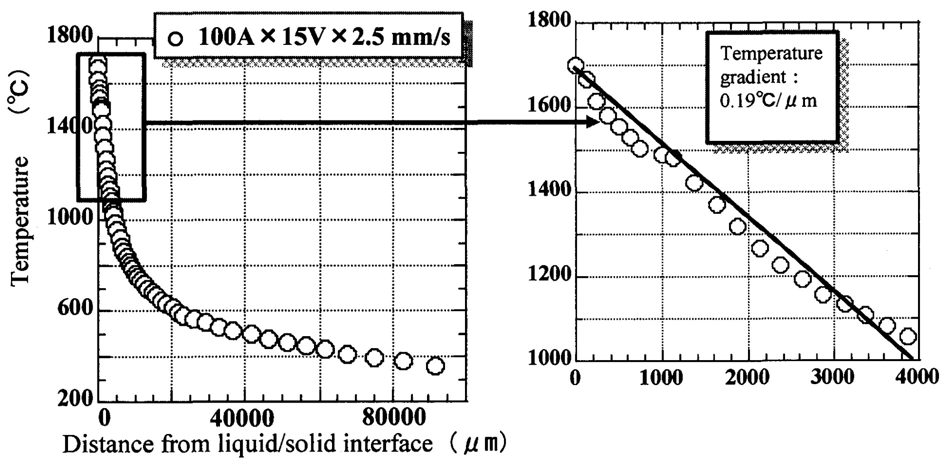

Fig. 3 Measured temperature profile during solidification of weld metal

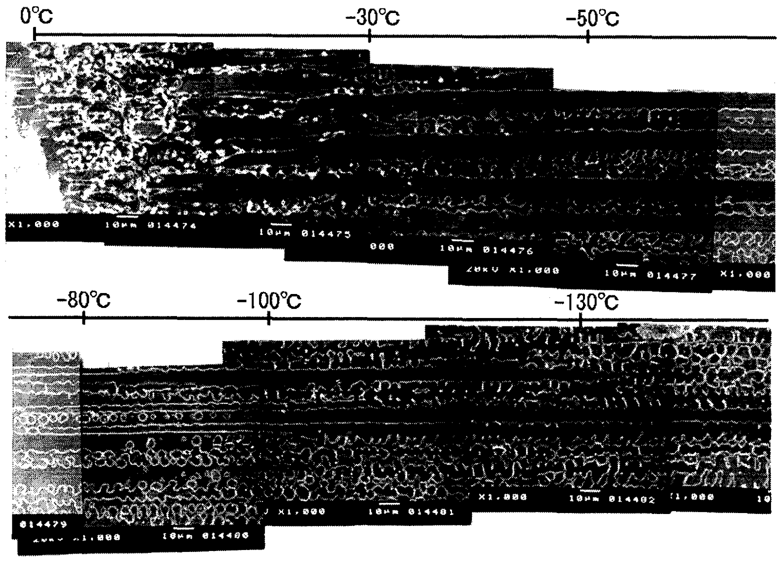

Fig. 4 SEM image of quenched stainless steel to the temperature drop from liquid/solid interface.

steel, with an angle of 20 degrees. The schematic illustration of quenching is shown in Fig. 1. The heat source was a TIG arc with a current of 100 A. The arc voltage and welding speed were 15 V and 15 cm/min, respectively. The chemical composition of the austenitic stainless steel used in this work is shown in Table 1. A schematic illustration of the X-ray diffraction measurement is shown in Fig. 2. The irradiated area is 30×500 μm and measurement spots were selected along the middle line of the weld bead. The measurement interval was 30 μm. Diffraction patterns were recorded onto an imaging plate, which was set with an angle of elevation of 50 degrees. The imaging plate was covered by the slit, that is, the imaging plate was used as a recording line and was slid for each measurement point. The camera length was acquired using 5 data of direct beam plotted on the imaging plate and result in 480.5017 mm. The wavelength of the incident beam was refined by the Rietveld method[4], using the diffraction pattern of LaB_6 (a=0.41569162 ± 0.00000097 nm).

The sample shown in Fig. 2 was rolled between –130 deg. to –50 degrees along the X axis, in order to decrease the effect of crystal face orientation. This is important because the imaging plate is used as one-dimensional recorder in the present experiment. The exposure time for each point was set to 10 seconds.

The temperature profile during solidification of weld metal was also measured using a thermo-couple.

Table 2 Diffraction angle and plane indices of crystallized δ and γ phase

2θ (deg.)	δ	γ
	a=2.86 Å	a=3.58 Å
28.876		111
29.518	110	
33.450		200
42198	200	
48.031		220
52.303	112	
56.992		311
59.807		222
70.267		400

3. Results

Measured temperature profile is shown in Fig. 3. The result was linearized in the vicinity of the liquid/solid interface and the temperature gradient was set to the value of 0.19 K/μm.

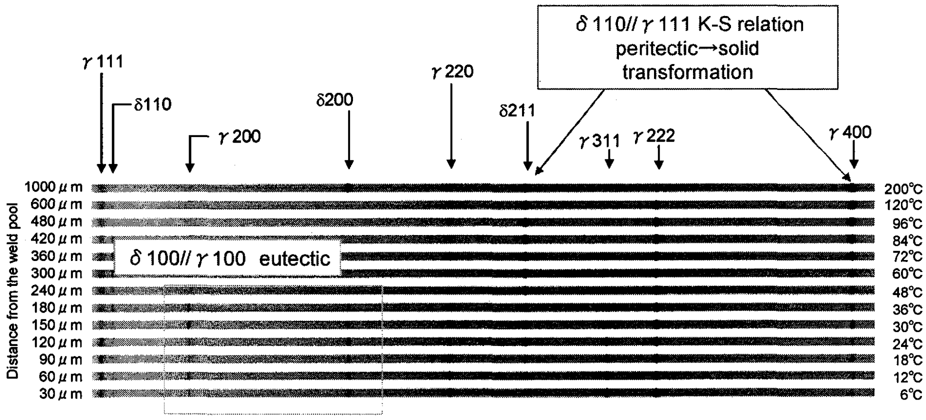

Fig. 5 Changes in X-ray diffraction patterns for distance from liquid/solid interface on quenched austenitic stainless steel.

The SEM image of quenched stainless steel to the temperature drop from liquid/solid interface is shown in Fig. 4. It can be observed that dendrites are growing along one direction, easy growth direction[5].

Diffraction angles and plane indices of crystallized δ and γ phase, which could be recorded in the imaging plate, are summarized in Table 2. A measured X-ray diffraction pattern on the imaging plate is shown in Fig. 5, converting the distance from liquid/solid interface to the temperature drop. The diffraction peaks of δ110 and δ200 show high intensity in the first stage of solidification. These planes are vertical to the easy growth direction <100>. This suggests that the primary δ phase grows along <100> directions. Furthermore, The diffraction peaks of γ200 and γ220 also show high intensity in the first stage of solidification. These planes are also vertical to the easy growth direction <100> and suggest that γ phase is crystallized in divorced eutectic growth, along <100> directions. This means that the parallel relation, δ[100]//γ[100], is established in the first stage of solidification.

On the other hand, the peaks of γ111, γ222 and γ311 which are also observed, are not vertical to the <100> direction, in the first stage of weld solidification. It is estimated that these peaks can be observed due to rotational growth of dendrites and the rolled X circle of experimental set up. So these are also γ scattering peaks, which grow along <100> directions. As solidification progresses, however, the intensity of these scattering peaks decreases. Instead of these peaks, the intensity of δ112 and γ400 peaks increases. These diffraction peaks have a K-S relation[6], δ(110)//γ(111). So it is supposed that these peaks correspond to the stage of δ-γ solid transformation or peritectic crystallization between δ and γ.

4. Conclusions

The high space-resolved diffraction pattern during solidification process, for austenitic stainless steel with F-A solidification mode, was measured using synchrotron radiation. It was cleared that γ phase was crystallized in divorced eutectic growth in the first stage of solidification. After that, a K-S relation could be observed, reflecting the δ-γ solid transformation or peritectic crystallization between δ and γ.

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