In-situ Observation of Solidification Behavior of 14Cr-Ni Steel Weld*

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The solidification-mode during GTA welding of martensitic stainless steel was directly judged by using TRXRD system for welding. The X-ray detector with wider area was applied and its usefulness for the monitor of solidification process of welding was presented in the point of accuracy of the mode-judgments. The solidification behavior observed was related to the result of the trans-varestraint test to relate the sollicitation phases and the hot-cracking phenomena. Furthermore, the retaining behavior of delta-ferrite from the temperature at solidification process to the room temperature was chased in-situ.

Key Words: Weld metal, Solidification, TRXRD, stainless steel.

1. Introduction

Martensite stainless steel such as 14Cr-Ni steel is candidate for the weld metal of ultra-high strength steel due to the high-strength nature of martensite. However, in order to secure the weldability with efficient way, the further technological developments is demanded and the researches for the solidification and solid-state transformation during welding should support the evolution to acquire the weldability. Avoiding hot cracking is one of the important factors in order to secure the weldability. It is well-understood that the solidification behavior during welding affects on the degree of ductility deterioration and investigating the behavior is important to avoid the hot-cracking problem.

Recent progress of analyzing method made it possible that the solidification behavior during welding is observed in-situ¹⁻⁵⁾ and the effect of phase sequence during solidification process could be directly related to the hot-cracking problem. When the ultra-bright X-ray would be used, the time to identify the phase could be few seconds. Then, phase evolution all over the welding process could be chased in-situ. During the solidification process of welding, the number of crystal is not enough to make a ring pattern of diffraction for each reflection. Furthermore, the constraint growth during welding makes texture. Then wider area of X-ray detector should be used to access the phase evolution during solidification process of welding.

In the present work, solidification behavior of 14Cr-Ni steel is analyzed in-situ during Gas Tungsten Arc welding. Time-Resolved X-ray Diffraction system used by our research group^{4, 5)} is applied for the observation. In this time, the X-ray

detector is enlarged along ring direction of diffraction pattern to catch the scattering from the small amount of crystal during solidification process. The solidification behavior observed is related to results of the trans-varestraint test to relate the sollicitation behavior and the hot-cracking phenomena. Furthermore, the retaining behavior of delta-ferrite from the temperature at solidification process to the room temperature is chased with the ultra-bright X-ray irradiation.

2. Experimental procedure

14Cr-Ni steel with different nickel contents was prepared. The chemical composition in mass% was 0.045 C-0.33 Si-0.83 Mn-14.05 Cr-9.3 Ni-0.0042 O and balance Fe (referred as M1), 0.045 C-0.32 Si-0.83 Mn-14.10 Cr-7.15 Ni-0.0047 O and balance Fe (referred as M2) and 0.047 C-0.32 Si-0.86 Mn-14.20 Cr-5.2 Ni-0.0045 O and balance Fe (referred as M3). The TRXRD experiments were performed in undulator beam line (BL46XU) in the third generation Synchrotron Radiation source, SPring-8 (JASRI). The schematic illustration of the TRXRD system is shown in Fig. 1. At undulator beam line, the X-ray was monochromatized with double Si crystals and the ultra-bright X-ray irradiated the fixed point on the welding bead. The welding torch position was the controlling parameter of the system. When torch arrived at decided positions, the shutter of X-ray in front of

Fig. 1 TRXRD system for welding (at 46XU SPring-8).

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Table 1 Conditions for TRXRD experiments

theta (incident angle)	10.0
2-theta (Fixed angle of Camera)	13.3
2-theta range	10.50-16.90
Camera length	300.129

the X-ray slits was opened. After that, the exposure starting and exposure finishing signals could be set at the any point of torch positions. The slit size was 0.5×0.1 mm square. Taking into accounts the spatial dimensions, the wavelength used was 30 keV. For the other experimental conditions are summarized in Table 1. The time resolution was 0.01 seconds with two-dimensional X-ray camera. For two-dimensional camera, each pixel could count the photon count⁽⁶⁻⁷⁾. The S/N ratio, dynamic range and read-out time for data is excellent with which halo pattern of liquid phase during welding could be detected in the time-resolution of 0.01 seconds.

3. Result and discussion

Figure 2 shows diffraction patterns at room temperature (before GTA welding). The reflection for $\alpha 110$, $\alpha 200$, $\gamma 111$ and $\gamma 200$ could be recorded on the camera. The number of crystals is enough to make ring pattern before welding. On the other hand, the number of crystal during solidification process was small and then the diffraction pattern changed to spot patterns. Figure 3 shows diffraction patterns on the solidification process of M1 (9.3 Ni) sample during GTA welding. The spot for the austenite phase

Fig. 3 The diffraction patterns for solidification process of M1 **Fig. 2** Diffraction patterns at room temperature

was circled with solid line. The primary phase was gamma austenite phase. The broad and weak intensity pattern (Halo pattern) corresponds to liquid phase was also observed as shown in Fig. 3 (a). As shown in Fig. 3 (b), the solidification finished with austenite phase. Then the solidification mode was A-mode. The diffraction spot in this case was periphery of camera windows. It suggested the importance of wide-camera usage when small number of crystal was observed. Figure 4 shows diffraction patterns on the solidification process of M2 (7.15 Ni) sample during GTA welding. The broken line circled spot diffraction pattern for delta-ferrite phase. The primary phase during solidification was austenite and after 0.5 seconds, delta-ferrite phase was as a secondary phase with halo pattern as shown in Fig. 4 (b). It corresponded to peritectic reaction. The solidification finished with dual phase (delta and gamma) as shown in Fig 4 (c). Then the solidification mode was A-F mode. The retained behavior of delta ferrite is interesting. The delta phase is known as a detrimental phase to the toughness. Furthermore, it is difficult to identify it after the welding. The martensite has a almost same crystal structure and it is hard to distinguish between retained delta-ferrite and martensite in reciprocal lattice space. Then change of diffraction patterns was

Fig. 4 The diffraction patterns for solidification process of M2

Fig. 5 The diffraction patterns after solidification process of M2 sample showing retained behavior of delta-ferrite.

Fig. 6 The diffraction patterns for solidification process of M3 sample (5.2 Ni).

chased until room temperature. Figure 5 shows diffraction pattern of M2 sample (7.15 Ni) after solidification process. The delta ferrite retained until room temperature and after the 64.5 seconds from the primary phase, diffraction pattern corresponded to martensite phase appeared as shown in Fig. 5 (c).

Figure 6 shows diffraction pattern for the solidification process of M3 sample (5.2 Ni). Nickel is the elements of austenite former. Then it is expected the Further decreasing of nickel cause the solidification mode approached to F-A mode. The primary phase was delta phase as shown in Fig. 6 (a). After 0.08 seconds

Fig. 7 The diffraction patterns after solidification process of M3 sample showing retained behavior of delta-ferrite.

from the primary phase, austenite phase nucleate and growth as the secondary phase. The delta phase, gamma phase and broad halo pattern was clearly observed in Fig. 6 (b). It corresponded peritectic reaction and the solidification mode was F-A mode as expected. If the camera width was half of present set-up, the solidification mode could be misjudged as A-mode or F-mode. As the same reason described above, the retained behavior of delta-phase was investigated. Figure 7 shows diffraction pattern of M3 sample (5.2 Ni) after solidification process. The delta ferrite retained until room temperature and after the 46.63 seconds from the primary phase, diffraction pattern corresponded to martensite phase appeared as shown in Fig. 7 (c). In Fig. 5 and Fig. 7, the distribution of diffraction spot for delta-phase slightly moved within same reflection. It reflected the deformation during welding.

Table 2 shows results of trans-varestraint test with 4% strain. In the case of A-mode solidification, that is M1 sample, the crack length was huge. On the other hand, when the delta-phase existed, the crack-length was little. The delta-ferrite phase prevent the hot-cracking due to its high solubility of minor elements such as

Table 2 Results for trans-varestraint test (4% strain)

	Max. crack length (mm)	Total crack length (mm)
M1	1.2	5.96
M2	0	0
M3	0.27	0.7

phosphorus and sulfur. It means that liquid film was retained until low temperature in the case of M1 sample. Unfortunately, the behavior of liquid film was not able to detect due to little amount of it in the present work.

From the results of TRXRD experiments and trans-varestraint test, it could understand the importance of the behavior of delta-phase for the weld metal of high strength steel. The ideal behavior of delta phase are:

1) Existing during solidification process of welding to absorb the minor element and prevent the formation of liquid film.

2) Disappearing the after solidification because the existing after the welding is detrimental for the toughness of weld metal of high strength steel.

In this view, the design of chemical composition of M2 and M3 sample should be upgraded with taking into account the other factor of weldability. The TRXRD technique for welding could serve for the upgrading the chemical design of weld metal of high strength steel.

4. Conclusion

Solidification-mode during GTA welding was directly judged by using TRXRD system for welding. The X-ray detector with wider area was applied and its usefulness for the monitor of solidification process of welding was presented in the point of accuracy of the mode-judgments. The relation between solidification mode and results for transvarestraint experiment enhanced the importance of existing of delta-ferrite during solidification in order to secure the weldability. Furthermore, the upgrading point of chemical design of weld metal for high

strength steel was discussed. TRXRD system for welding is useful to check the design of weld metal with allover assessment of phase evolution during welding.

Reference

- 1) J. W. Elmer, J. Wong, M. Froba, P. A. Waide and E. M. Larson, *Metall. Mater. Trans. A*, 27, pp. 775-783 (1996).
- 2) J. W. Elmer, J. Wong and T. Ressler, *Metall. Mater. Trans. A*, 32A, pp. 1175-1187 (2001).
- 3) S. S. Babu, J. W. Elmer, J. M. Vitek and S. A. David, *Acta Mater.*, 50, pp. 4763-4781 (2002).
- 4) M. Yonemura, T. Osuki, H. Terasaki, Y. Komizo, M. Sato and H. Toyokawa, *Materials Transactions*, 47, pp.2292-2298. (2006).
- 5) Y. Komizo, H. Terasaki, M. Yonemura and T. Osuki, *Quarter. J. of JWS*, 24, pp. 57-64 (2006).
- 6) Ch. Bronnimann, R. Baur, E.F. Eikenberry, P. Fischer, S. Florin, R. Horisberger, M. Lindner, B. Schmitt and C. Schulze, *Nucl. Instr. Meth.*, A477, 531-35 (2002).
- 7) E.F. Eikenberry Ch. Bronnimann, G. Hulsén, H. Toyokawa, R. Horisberger, B. Schmitt, C. Schulze and T. Tomizaki, *Nucl. Instr. Meth.*, A501, 260-66 (2003)

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