

Nano-structural and Nano-chemical analysis of dissimilar metal weld interfaces

Kyoung Joon Choi, Sang Hoon Shin, Jong Jin Kim, Ju Ang Jung, Ji Hyun Kim*
Ulsan National Institute of Science and Technology(UNIST)
100 Banyeon-ri, Eonyang-eup, Ulju-gun, Ulsan, Republic of Korea 689-798
*Corresponding author: kimjh@unist.ac.kr, kjchoi@unist.ac.kr

1. Introduction

Dissimilar Metal Welds (DMWs) is generally applied to nuclear power plants for manufacturing and machining in structural components such as reactor pressure vessels and pressurizer nozzles. Alloy 152 is used frequently as filler metal in the manufacture of the DMW in light water reactors to join the low alloy steel pressure vessel nozzles and steam generator nozzles to nickel-based wrought alloy or austenitic stainless steel components. However, in recent years cracking phenomena has been observed in the welded joints. Concerns have been raised to the integrity and reliability in the joint transition zone due to the high susceptibility of the heat affected zone (HAZ) and the fusion boundary (FB) to stress corrosion cracking in combination with thermal aging [1].

Since the material microstructure and chemical composition are key parameters affecting the stress corrosion cracking, improving the understanding of stress corrosion cracking at the FB region requires fundamental understanding of the unique microstructure of the FB region in DMW. Despite the potential degradation and consequent risk in the DMW [1, 2], there is still a lack of the fundamental understanding of microstructure in the FB region, in particular the region containing unidentified band structures near the FB.

The scale of the microstructure in modern metallic materials is becoming increasingly smaller. The 3-dimensional atom probe tomography (3D APT) has a truly quantitative analytical capability to characterize nanometer scale particles in metallic materials, thus its application to the microstructural analysis in multi-component metallic materials provides critical information on the mechanism of nanoscale microstructural evolution [3]. The 3D APT is a microscope that allows the reconstruction of 3D "atom maps" [4]. These reconstructions can be interrogated and interpreted to determine the nanoscale chemistry of the material.

Therefore, the current study is aiming at the establishment of detail procedure for the characterization of the dissimilar metal welds and the analysis of results by using 3D APT in order to get a clear understanding of structure and chemistry in the fundamental scale of weld interfaces.

2. Experimental Procedure

Specimens for atom probe analysis were created using a multi-stage process as follows. A representative dissimilar weld mock-up made of Alloy 690/Alloy 152/A533Gr.B fabricated by Argonne National Laboratory (ANL) was acquired. The weld joint of Alloy 690 and A533Gr.B was prepared by the shielded metal arc welding method with Alloy 152 fillers. The chemical compositions of both metals are shown in table 1. The DMW containing the interface and Alloy 152 was cut from the dissimilar weld joint. The samples were polished with ~0.05 μ m colloidal silica. In the previous study [5], the chromium carbides were observed in the FB. Thereby, specimens for 3D APT were prepared near the region containing the FB which was represented in the electron microscope images of Fig. 3. After isolating a 10 μ m \times 1 μ m \times 5 μ m volume containing the FB [6-8], three specimens were fabricated with an isolated volume by focused ion beam from a Helios Nanolab 600 Dual Beam.

Specimens were analyzed using a Cameca LA_WATAP 3D AP for the chemical composition in the region containing the A533Gr.B and the FB. Since the specimen containing some chromium carbides is fragile during the analysis, the laser pulsing method which can alleviate the problem [4] was used to remove ions from various specimens with 343 nm of wave length. For the laser pulsed specimen atom probe specimen, the tips were cooled to a temperature of 60K. The laser pulse was used with a repetition rate of 100 kHz and voltage of 2~14kV.

3. Result

All 3D APT analyses were performed near the FB containing some the chromium carbides as shown in Fig. 1 (b). The three dimensional reconstructions obtained from the needle specimen are shown in Figs. 2. The reconstruction volumes can show Cr and C segregation in the FB and depletion on either region. In addition, the profiles of the specific elements (such as Fe, C, Cr, Mn and so on) are observed in the selected region of the three dimensional reconstructed volumes of these figures. These volumes were expressed as a total and each elemental distributional map including C, Cr, Mn, Mo, Si, Fe and Ti.

Table 1. Chemical Composition

Material	C	Al	Si	P	S	Cr	Mn	Fe	Co	Ni	Cu	Nb+Ta	Mo	Ti
Alloy690	0.03		0.07		<0.001	29.5	0.20	9.9		59.5	0.01			
Alloy 152	0.040	0.240	0.460	<0.003	<0.001	29.04	3.560	9.360	<0.01	55.25	<0.01	1.84	0.01	0.15
A533B	0.220		0.19	0.010	0.012	0.18	1.28			0.51			0.48	

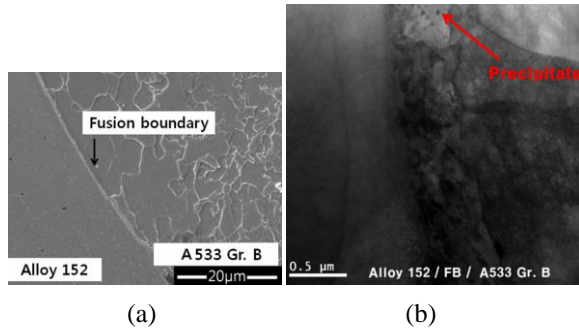


Figure 1. Scanning electron microscope image (a) and transmission electron microscope image (b) in the fusion boundary in the weld root [5]

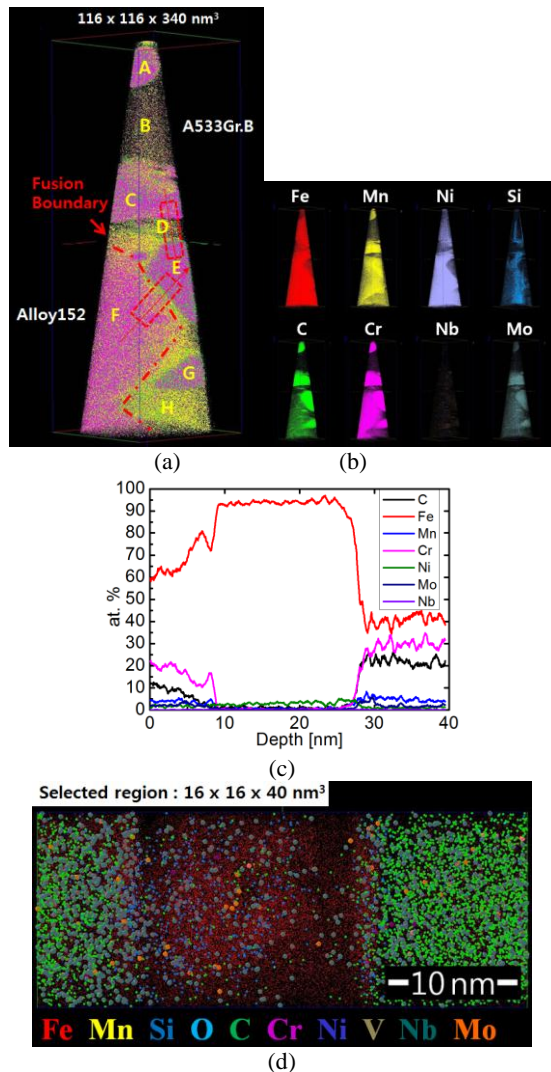


Figure 2. 3-dimensional reconstruction atomic map (a) of the DMW atom probe specimen, the elemental distributional map (b) of C, Cr, Mn, Mo, Si, Nb, Fe and Ti distributions, elemental profiles (c) and atomic map (d) in the volume of 'C-D-E'

In this study, three of the specimens prepared by the FIB equipment were successfully analyzed by the 3D APT. Through the maps, some chromium carbides were observed in A, C, E and G spaces in Fig. 2. These

spaces show that the carbon and chromium concentrations are higher, but the iron is lower than the others. On the part including 'C-D-E' of the maps, there are the specific volume's profiles and atomic map shown in Figs. 2 (c) and (d). As can be seen from the profiles of Fig. 2 (c), it is similarly observed that the carbon and chromium concentrations are relatively high in C space and E space. Fig. 2 (d) also shows that some precipitates exist in the light (C space) and the left (E space) showing the particularly high concentration of chromium and carbon. As seen from the 3D APT results, these regions can be considered as chromium carbides which are formed in Alloy 152 weld metal during the welding process. In addition, through that B and D spaces have higher Fe and lower Ni than F space, the FB can be also distinguished from other regions as shown in Fig. 2 (a).

In short, the region including some precipitates have higher carbon and chromium concentrations than the other regions, which is similar to what was observed in secondary ion mass spectrometry and transmission electron microscope analyses performed in the previous study [5]. These results can help to distinguish the chemical difference between the precipitates and the others. Thereby, there are the chromium carbides which are formed in Alloy 152 weld metal during the welding process. A detail procedure for the 3D APT analysis including sample preparation and analysis condition, and the results will be published in a separate article in the future.

REFERENCES

- [1] J. Hou, Q.J. Peng, Y. Takeda, J. Kuniya, T. Shoji, J.Q. Wang, E.H. Han and W. Ke, "Microstructure and mechanical property of the fusion boundary region in an Alloy 182-low alloy steel dissimilar weld joint," *J. Mater. Sci.*, vol. 45, pp. 5332 (2010)
- [2] T.W. Nelson, J.C. Lippold and M.J. Mills, "Nature and Evolution of the Fusion Boundary in Ferritic-Austenitic Dissimilar Metal Welds —Part 2: On-Cooling Transformations," *J. Weld.*, vol. 79, pp. 267 (2000)
- [3] K. Hono, "Atom probe microanalysis and nanoscale microstructures in metallic materials" *Acta Mater.*, vol. 47, pp. 3127 (1999)
- [4] D. Hudson and G.D.W. Smith, "Initial observation of grain boundary solute segregation in a zirconium alloy (ZIRLO) by three-dimensional atom probe", *Scripta Mater.*, Vol. 61, pp. 411, (2009)
- [5] K.J. Choi, J.J. Kim and J. H. Kim, "Nano-structural and nano-chemical analysis of Ni-base alloy/low alloy steel dissimilar metal weld interfaces", Submitted to *Nuclear Engineering and Technology* (2012)
- [6] S. Lozano-Perez, "A guide on FIB preparation of samples containing stress corrosion crack tips for TEM and atom-probe analysis", *Micron*, vol. 39, pp. 320, (2008)
- [7] M.K. Miller, K.F. Russell, "Atom probe specimen preparation with a dual beam SEM/FIB miller", *Ultramicroscopy*, vol. 107, pp. 761, (2007)
- [8] M.K. Miller, K.F. Russell and G.B. Thompson, "Strategies for fabricating atom probe specimens with a dual beam FIB", *Ultramicroscopy*, Vol. 102, pp. 287, (2005)