Manufacturing Parameter Study on Corrosion and Microstructural Characteristics of HANA-6 Alloy

Hyun-Gil Kim*, Byung-Kwan Choi, Hai-Dong Cho, Jeong-Yong Park, Yong-Hwan Jeong
Fusion Technology Development Division, KAERI, Daedeokdaero 1045, Yuseong, Daejeon 305-353, Korea
**Corresponding author: hgkim@kaeri.re.kr*

1. Introduction

The development of an advanced Zr-based alloy with an improved corrosion and creep resistance is necessary for the high burn-up operating conditions in PWRs. Many parameter studies have been performed to increase the corrosion resistance of Zr-based alloys [1, 2]. They have reported that the corrosion kinetic of Zr-based alloys was affected by the alloying elements as well as the heat-treatment conditions. It is considered that the manufacturing parameter is a very important factor in the new alloy system to increase its corrosion resistance. So, it is necessary to perform a parameter study, which contains cold working steps and intermediate annealing temperatures, to obtain the optimum manufacturing parameters.

A high Nb-containing Zr-based alloy named HANA-6 was designed at KAERI and its nominal composition is Zr-1.1Nb-0.05Cu in wt.% [3]. In order to obtain the best manufacturing parameter for the HANA-6 alloy, various evaluations such as a corrosion test, a microstructural analysis, and a texture analysis were performed on the HANA-6 alloy with various manufacturing parameters.

2. Methods and Results

Test samples of HANA-6 alloy were prepared as sheets by applying 4 types of manufacturing parameters which were controlled by a combination of the intermediate annealing temperature at both 570 and 596℃ and the reduction ratio range from 72 to 87% in thickness from the HANA-6 alloy tube shell. And two types of final annealing conditions at both 470 and 510℃ were applied to the manufactured HANA-6 alloy sheets. Therefore, 8 types of sheet samples were prepared from the HANA-6 tube shell as shown in Table 1.

Table 1 Manufacturing parameters of HANA-6 alloy

Process		P1	P2	P3	P4
TREX		10.9mm			
1st rolling		3.5mm		4.955mm	
1st annealing		570C x 2h	596C x 3.5h	570C x 2h	596C x 3.5h
2nd rolling		1.65mm		1.83mm	
2nd annealing		570C x 2h	596C x 3.5h	570C x 2h	596C x 3.5h
3rd rolling		0.57mm		0.57mm	
Final annealing	A2	470C x 8h			
	A4	510C x 8h			

A corrosion performance test was conducted at a 400℃ steam condition under a saturated pressure of 10.8 MPa. The corrosion behavior was evaluated by measuring the weight gain of the corroded samples during the corrosion test at a periodic time interval.

The microstructure with various manufacturing processes was observed by an optical microscopy with a polarized light. The precipitate characteristics were analyzed by TEM equipped with EDS. Specimens for the TEM observation were prepared by a twin-jet polishing method with a mixed solution of C_2H_5OH (90 vol. %) and $HClO_4$ (10 vol. %) after a mechanical thinning to about 70 μm.

To define the crystallographic texture with the various manufacturing processes, XRD analysis was performed for all the specimens by using the Bruker AXS 5005 model with a 4 cycle Goniometer.

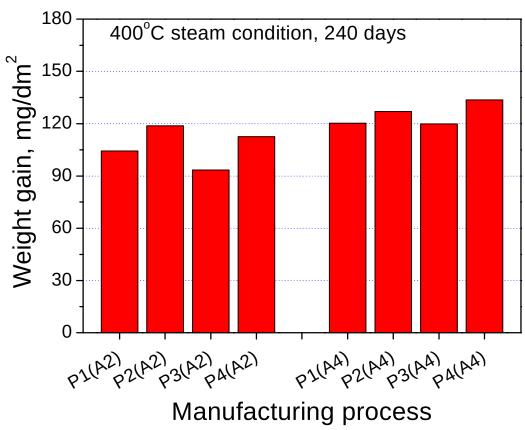

Fig. 1 Corrosion behavior of the HANA-6 alloy with the manufacturing parameters

Fig. 1 shows the corrosion weight gain of the HANA-6 alloy as a function of the manufacturing parameters tested at 400℃ steam. The corrosion weight gain was affected more by the intermediate annealing temperature than by the intermediate reduction ration from this result. Regarding the intermediate annealing effects, the corrosion resistance was increased when the intermediate annealing was performed at a lower temperature of 570℃ than that of 596℃. Also, the weight gain was decreased when the sample was annealed at a low temperature of 470℃ when compared to a high temperature of 510℃ from the final annealing effects. By a control of the manufacturing parameters, the corrosion resistance of the HANA-6 alloy was increased by up to 25% when compared with the highest and the lowest weight gain of the manufacturing parameters. Therefore, it was revealed that a manufacturing parameter control is an important factor to increase the corrosion resistance of HANA-6 alloy. The corrosion resistance is affected by the microstructural characteristics such as the grain size and precipitate type, since the microstructural characteristics are determined by the annealing and reduction control during each manufacturing step.

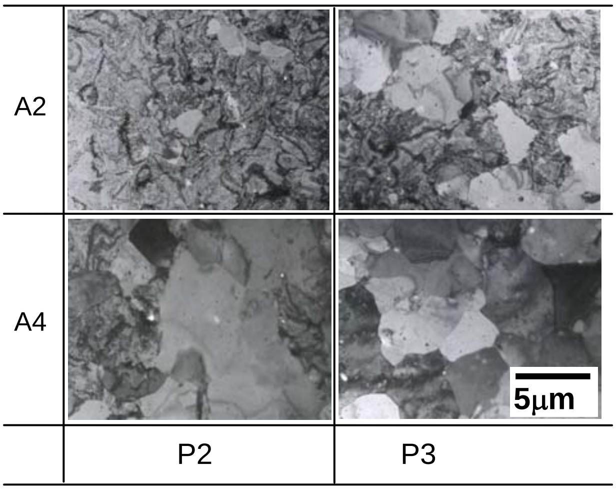

Fig. 2. TEM micrographs of the HANA-6 alloy with different manufacturing parameters after the final steps

Fig. 2 shows the TEM micrographs of the HANA-6 alloy for the final manufacturing steps. From this figure, it is observed that the fraction of the recrystallized grain is changed with the applied manufacturing parameters. The fraction of the recrystallized grain was considerably increased with an increasing final annealing temperature from 470 to 510℃, however, it was difficult to elucidate the effect of an intermediate manufacturing parameter on the microstructure of the HANA-6 alloy. Regarding the precipitate analysis, it is impossible to obtain all of the precipitate information such as the number density, and area fraction with the manufacturing parameters, since the microstructure was not fully recrystallized after each manufacturing parameters. However, from the precipitate observation in the recrystallized region, it is observed that the major precipitate is the β-Nb phase with a BCC structure. A small content of Cu about 1~2 at.% is contained in the β-Nb phase. The minor precipitate is the ZrNbFeCu-type phase with a HCP structure and this phase consisted of Zr (40~70 at.%), Nb (20~40 at.%), Fe (15~25 at.%), and Cu (1~5 at.%). Two types of precipitates were observed in all the manufactured samples, but the composition and mean size of the observed precipitates were changed with the manufacturing parameters.

From the texture analysis by using XRD, the texture of the (0002) basal plane was considerably oriented perpendicular to the rolling direction, since the crystallographic orientation of that plane is controlled by a twin mechanism, which is favorable for a compressive stress during cold rolling. The pole figure results with the manufacturing parameters were summarized in Table 2. The f_n value was increased by increasing the final annealing temperature from 470℃ to 510℃, regarding the effect of the final annealing temperature on the texture.

Table 2. Pole figure results of the HANA-6 alloy sheets for the normal direction

Manufacturing parameters		P1	P2	P3	P4
Final annealing	A2	0.671	0.664	0.688	0.685
	A4	0.709	0.730	0.711	0.736

3. Conclusions

The corrosion and microstructural characteristics of HANA-6 alloy were affected by the manufacturing parameters.

The corrosion resistance was increased when the intermediate annealing was performed at 570℃ rather than 596℃, and also when the final annealing was performed at 470℃ rather than 510℃. Therefore, the corrosion characteristics of the HANA-6 alloy were considerably affected by the manufacturing parameter controls which is affected to the precipitate characteristics in the matrix.

A crystallographic texture was developed more by increasing the final annealing temperature from 470℃ to 510℃.

REFERENCES

[1] R.J. Comstock, G. Schoenberger, G.P. Sabol, "Influence of Processing Variables and Alloy Chemistry on the Corrosion Behavior of ZIRLO Nuclear Fuel Cladding," *Zirconium in the Nuclear Industry*, **ASTM STP 1295**, 710 (1996)

[2] H. Anada, B.J. Herb, K. Nomoto, S. Hagi, R.A. Graham, T. Kuroda, "Effect of Annealing Temperature on Corrosion Behavior and ZrO2 Microstructure of Zircaloy-4 Cladding Tube," *Zirconium in the Nuclear Industry*, **ASTM STP 1295**, 74 (1996)

[3] Y.H. Jeong, S.Y. Park, M.H. Lee, B.K. Choi, J.H. Baek, J.Y. park, J.H. Kim, H.G. Kim, Out-of pile and In-pile Performance of Advanced Zirconium Alloys (HANA) for High Burn-up Fuel, J. Nucl. Sci & Tech., **43**, 977 (2006)