# Corrosion and Creep Characteristics of the HANA-4 Alloy with the various Manufacturing Processes

Hyun-Gil Kim<sup>\*</sup>, Byung-Kwan Choi, 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

Zirconium alloys have been used as a fuel cladding material for several decades, since these alloys have revealed a good corrosion resistance and mechanical properties in reactor operating conditions. 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. The alloying element effects of the Nb, Sn, Fe, Cr, Cu etc as well as an optimization of the manufacturing processes such as the reduction ratio and annealing temperatures have been studied to improve the corrosion and creep properties [1]. A high Nb-containing Zr-based alloy named HANA-4 was designed at KAERI and its nominal composition is Zr-1.5Nb-0.4Sn-0.2Fe-0.1Cr in wt.%. For high Nb-containing Zr alloys, their corrosion resistance is very sensitive to their microstructural characteristics which are determined by a manufacturing process [2, 3]. In order to obtain the best manufacturing process for the HANA-4 alloy, various evaluations such as corrosion and creep tests, a microstructural analysis, and a texture analysis were performed on the HANA-4 alloy with various manufacturing processes.

#### 2. Methods and Results

Test samples of HANA-4 alloy were prepared as sheets by applying 4 types of the manufacturing processes which were controlled by a combination of the intermediate annealing temperature at both 570 and 596 °C and the reduction ratio range from 72 to 87% in thickness from the HANA-4 alloy TREX (Tube Reduced Extrusion). And two types of final annealing conditions at both 470 and 510 °C were applied to the manufactured HANA-4 alloy sheets. Therefore, 8 types of sheet samples were prepared from the HANA-4 alloy TREX as shown in Table 1.

Table 1	Manufacturing	processes	of HANA-4 alloy	
1 4010 1		p100000000	01111111111101	

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

A corrosion performance test was conducted at a  $400\,^\circ$ C 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. Creep test were carried out under a constant applied stress of 120 MPa at a temperature of  $380^{\circ}$ °C. The creep strain was measured by using a LVDT (Liner Variable Differential Transformer) extensometer and that strain was collected for up to 240 hours. 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. The microstructural characteristics were analyzed by TEM (Transmission Electron Microscopy) equipped with EDS (Energy Dispersive Spectra).



Fig. 1 Corrosion behavior of the HANA-4 alloy with the manufacturing processes

Fig. 1 shows the corrosion weight gain of the HANA-4 alloy as a function of the manufacturing processes tested at 400  $^{\circ}$ C steam. It was observed that the weight gain was changed by the various manufacturing processes. The corrosion behavior was affected more by the intermediate annealing temperature than by the intermediate reduction ratio from this result. Regarding the annealing effects, the corrosion resistance was increased when the intermediate annealing was performed at a lower temperature of 570 than that of 596℃. However, the weight gain was decreased when the sample was annealed at a high temperature of  $510^{\circ}$ C when compared to a low temperature of  $470\,^{\circ}$ C from the final annealing effect. By a control of the manufacturing processes, the corrosion resistance was increased by up to 15% when comparing the highest and the lowest weight gain of HANA-4 alloy process. Therefore, it is

revealed that a manufacturing process control is an important factor to increase the corrosion resistance of the HANA-4 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 Creep behavior of the HANA-4 alloy with the manufacturing processes

After the creep test, the creep behavior was considerably affected by the final annealing temperature regardless of the intermediate annealing and reduction ratio as shown in Fig. 2. The creep strain for the low final annealing of 470°C was increased by up to 16% when compared to that for the high final annealing of  $510^{\circ}$ C after the 240 hours. Since it is found that the fraction of the recrystallized grains is considerably increased when the samples are annealed at  $510^{\circ}$ C from the microstructural observation by using TEM, it is concluded that the fraction of the recrystallized grains is affected by the creep strain of the HANA-4 alloy. Since many dislocations are removed from the matrix by increasing the final annealing temperature, the mobile dislocation density is decreased. Therefore, the creep resistance was improved for the high temperature annealed HANA-4 alloy.

From the texture analysis by using XRD, the texture of the (0002) basal plane was considerably oriented perpendicular to the rolling direction as shown in Fig. 3, since the crystallographic orientation of that plane is controlled by a twin mechanism, which is favorable for a compressive stress during cold rolling. It was found that the texture was similar in all manufactured HANA-alloy sheets in this work, since the total reduction from the 1<sup>st</sup> to the 3<sup>rd</sup> cold rolling is the same for all manufactured sheet samples.



Fig. 3 Pole figure results of the HANA-4 alloy sheets for the normal direction

#### 3. Conclusions

The corrosion and creep characteristics of HANA-4 alloy were affected by the manufacturing processes. The corrosion resistance was increased when the intermediate annealing was performed at  $570^{\circ}$ C rather than  $596^{\circ}$ C, and also when the final annealing was performed at  $510^{\circ}$ C rather than  $470^{\circ}$ C. The creep property was increased by increasing the final annealing temperature from 470 to  $510^{\circ}$ C regardless of the intermediate annealing temperature as well as the cold reduction rate. So, we need to control the intermediate annealing temperature so that it is decreased to increase the corrosion resistance and to increase the final annealing temperature to increase the corrosion and creep properties.

### REFERENCES

[1] Y.H. Jeong, S.Y. Park, M.H. Lee, B.K. Choi, J.H. Baek, J.Y. Park, J.H. Kim and H.G. Kim, Out-of-pile and In-pile Performance of Advanced Zirconium Alloys (HANA) for High Burn-up Fuel, J. Nuclear Science and Technology, Vol. 43, p. 977, 2006.

[2] Y.H. Jeong, H.G. Kim, T.H. Kim, Effect of  $\beta$  phase, Precipitates and Nb-concentration in Matrix on Corrosion and Oxide Characteristics of Zr-xNb Alloys, J. Nucl. Mater., Vol. 317, p. 1, 2003.

[3] H.G. Kim, S.Y. Park, M.H. Lee, Y.H. Jeong, and S.D. Kim, Corrosion and Microstructural Characteristics of Zr-Nb Alloys with Different Nb Contents, J. Nucl. Mater., Vol. 373, p. 429, 2008.