

## Microstructure and Corrosion Characteristics with the Manufacturing Process of HANA Alloy

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### 1. Introduction

The HANA alloy designed in KAERI was one of the newly developed materials having an improved corrosion resistance. It was reported that the corrosion properties of Zr-based alloys were very sensitive to their microstructural properties such as the texture, dislocations and precipitate characteristics [1-2]. The microstructural characteristics of Zr-based alloy were determined by the performed manufacturing process conditions. Therefore, to obtain a good corrosion resistance, the Zr-based alloy as a fuel cladding was applied to the optimized manufacturing process which could be found by different parameter studies. Generally, the manufacturing process of a fuel cladding was divided into two stages. In the first stage, the TREX (Tube Reduced Extrusion), which is a semi-product to make a fuel cladding, is manufactured by a sequence of a melting, beta-forging, beta-quenching, extrusion, pilgering, and annealing. In the second stage, the fuel cladding, which is final product, is manufactured by several pilgering steps containing an intermediate and final annealing from the TREX. So, the quality of the fuel cladding as a final product could be affected by the TREX property as a semi-product. Therefore, the purpose of this investigation is to evaluate the property of HANA alloy during a sequence of the TREX manufacturing process.

### 2. Experimental procedure

The HANA TREX was manufactured by the sequence of a melting, beta-forging, beta-quenching, extrusion, pilgering, and annealing in the Western Zirconium Company. To evaluate the property of HANA alloy during the TREX manufacturing process, the test samples were cut from the mass product during each manufacturing step as shown in Fig. 1. The microstructure with the manufacturing sequence was observed using an optical microscope with a polarized light. The second phase characteristics were analyzed using a transmission electron microscope equipped with energy dispersive spectra. Specimens for TEM observation were prepared by twin-jet polishing with a solution of C<sub>2</sub>H<sub>5</sub>OH (90 vol.%) and HClO<sub>3</sub> (10 vol. %) after a mechanical thinning to about 70µm.

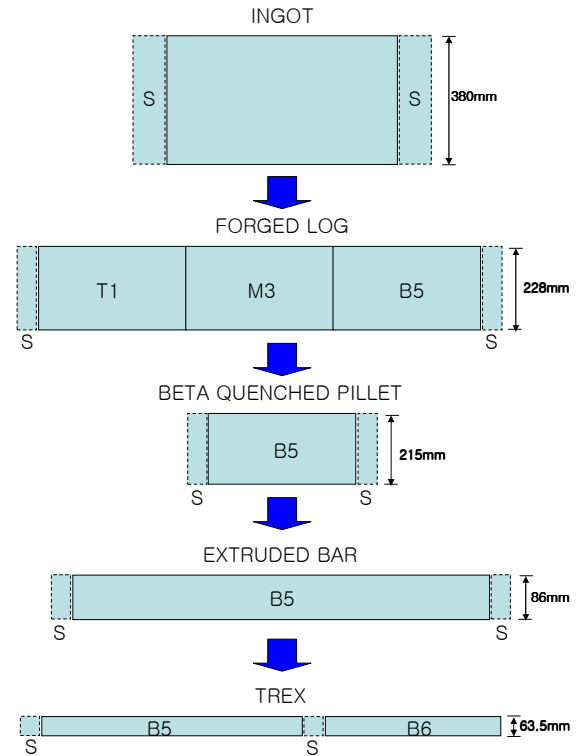


Fig. 1 Sampling plan of the HANA alloy during the TREX manufacturing process

The corrosion test was performed in a static autoclave of 360°C water under a saturated pressure of 18.7 MPa. Corrosion testing specimens of 10mm x 25mm x 1mm in size were cut from the prepared sheets and mechanically ground up to 1200 grit SiC paper. Also, the ground specimens for the corrosion test were pickled in a solution of H<sub>2</sub>O (40 vol.%), HNO<sub>3</sub> (30 vol.%), HCl (25 vol.%) and HF (5 vol.%). The corrosion resistance was evaluated by measuring the weight of the corroded samples after suspending the corrosion test at a periodic term.

### 3. Results and discussion

From the observation of the optical microstructures of HANA alloy with a TREX manufacturing process, massive large grains were observed in the case of a melting, forging, quenching process, and the elongated grains were observed in the case of a pilgering process.

The recrystallized grains were observed in the case of an extrusion and an annealing after a pilgering process.

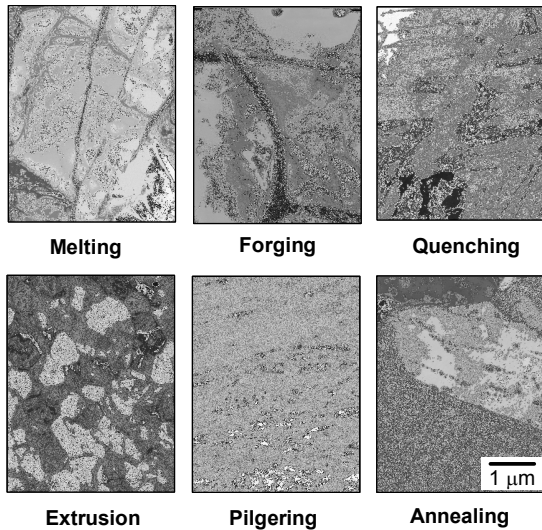


Fig. 2 TEM microstructures of HANA alloy with a TREX manufacturing process

From the result of the second phase analysis using TEM, a thin  $\beta$ -Zr phase was observed at the  $\alpha$ -phase boundary in the melted and forged HANA alloy. However, the alloying elements of HANA alloy were homogeneously supersaturated in the martensitic matrix by a quenching from 1050°C, because precipitates were not observed in the martensite structure. The second phase in the matrix was observed after a hot extrusion process of 650°C. By the pilgering process, the elongated microstructure, which was contained a lot of dislocations, was formed in the matrix. And the equiaxed grains containing small precipitates were formed by a TREX annealing. It was observed that the second phase type from the  $\beta$ -Zr phase to ZrNbFe precipitate was determined by a processing of the manufacturing steps from the TEM-EDS analysis.

Fig. 2 shows the corrosion behavior of HANA alloy with a manufacturing process in an autoclave of a 360°C loop condition up to 120 days. The corrosion resistance of HANA alloy was considerably affected by the TREX manufacturing steps. The corrosion resistance of HANA alloy was decreased when that alloy was taken by the process of melting, forging, and quenching steps, however, the corrosion resistance of that alloy was considerably increased by the next processes of an extrusion, pilgering, and annealing steps.

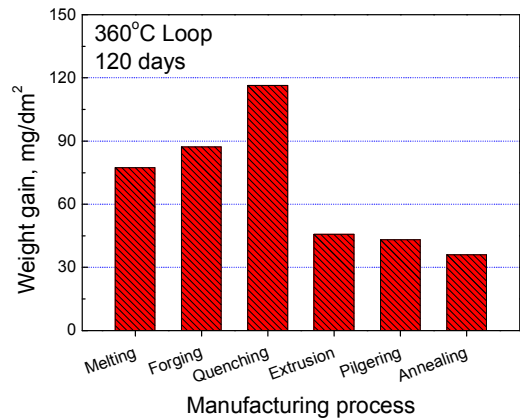


Fig. 2 Corrosion behaviors of HANA alloy with a TREX manufacturing process

From the results of the correlation between the microstructure and corrosion, it was assumed that the corrosion rate was correlated with the microstructural characteristics such as the  $\beta$ -Zr phase, supersaturated amount of the alloying elements, and the ZrNbFe precipitate which were determined by the TREX manufacturing steps.

#### 4. Conclusion

The microstructural characteristics of HANA alloy were largely changed by a TREX manufacturing process. The corrosion resistance of HANA alloy with a TREX manufacturing process was mainly affected by the microstructural characteristics such as the  $\beta$ -Zr phase, supersaturated amount of the alloying elements, and the ZrNbFe precipitate in the matrix.

#### ACKNOWLEDGMENTS

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