# Microstructural Characterization of Inter-Diffusion Layer between CrAl and Zr alloy after High Temperature Oxidation at 1200°C

Dong Jun Park, Yang Il Jung, Jung Hwan Park, Byoung Kwon Choi, Young Ho Lee, Il Hyun Kim, Hyun Gil Kim
*Korea Atomic Energy Research Institute, 989-111 Daedeok-daero, Yuseong-gu, Daejeon 34057, Korea*
[*]*Corresponding author: pdj@kaeri.re.kr*

## 1. Introduction

Loss-of-coolant accident (LOCA) is one of the design basis accident for light water reactor. In the event of LOCA, Zr based alloy used for nuclear fuel cladding and core component is subjected to a very high temperature over 1000℃ and water vapor environment. As a result, significant oxidation of the Zr alloy occurs and hydrogen generation as by-product of Zr-water reaction may result in explosion as in the case of Fukushima nuclear power plant. Many studies have been devoted to develop accident tolerant fuel cladding which can maintain its integrity in accident conditions for as long as possible. Coating of high-temperature materials with oxidation resistance on existing Zr alloy cladding is the one approach. In a previous study performed at this laboratory, the excellent performance of CrAl alloy in severe condition of high temperature and steam environment was shown, but the inter-diffusion between CrAl and Zr alloy and their microstructural analysis have not yet been investigated systematically.

In this study, we used CrAl alloy as a coating material on the Zr based alloy to improve their high temperature resistance during LOCA scenarios. CrAl layer coated Zr based alloy samples were exposed at 1200 ℃ in steam environment. Microstructure and elemental distribution in the inter-diffusion layer and interface region between coated CrAl alloy and Zr based alloy was analyzed in detail using field emission transmission electron microscopy (FETEM). The results were coincident with high resolution TEM (HRTEM) results.

## 2. Methods and Results

### *2.1 Experimental*

To compare the high temperature oxidation behavior of the coated and uncoated Zr alloy samples under LOCA scenario conditions, the specimens were exposed to a flow of water vapor at high temperature using an infra-red radiation furnace. The samples were enclosed in a quartz tube for steam flow and a water quench. Then the furnace was heated to a pre-test hold temperature of 300 ℃ within 240 s, where the steam flow and sample temperature were stabilized for 500 s. A heating rates of 28℃/s from 300 ℃ to 1000 ℃ and 2.5 ℃/s from 1000 ℃ to 1200 ℃ were used. After exposure at 1200 ℃ for 300 s in steam environment, the sample was cooled slowly to 800 ℃ and then quenched by flooding water from the bottom. Further details of the test equipment and experimental procedures can be found in our previous paper [1].

### *2.2 Results*

To investigate more detailed microstructure and elemental distribution near interface region, the scanning TEM(STEM) and EDS analysis were performed and the results are shown in Fig. 1. Fig. 1(a) shows the bright-field scanning TEM(STEM) image of the interface region.

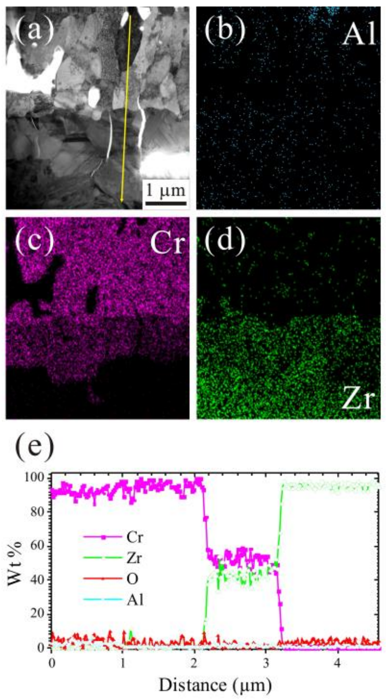


Fig. 1. (a) STEM image of an area close to the CrAl/Zr interface. EDS elemental mappings of (b)Al, (c) Cr, and (d) Zr in same area as in (a). (e) EDS profiles recorded along the yellow line shown in (a).

The elemental distribution maps of Fig. 1(c) and (d), obtained using EDS method, shows clearly distinguished three regions exist in the interface region. They are the upper CrAl alloy coating layer, the middle region formed by inter-diffusion of Cr and Zr, and the lower Zr matrix. Width of the diffusion layer is about 1.115 μm, which contains both Cr and Zr elements. In Fig. 1(e), EDS profiles for each element recorded along the yellow line in Fig. 1(a) and steep variations in the concentration of the Cr and Zr elements were observed at both points of intersections of white dotted line and yellow line. This result also provides a direct evidence for the existence of the inter-diffusion layer. To identify the phase and microstructure of the inter-diffusion layer, SADPs were taken from the inter-diffusion layer in Fig. 1(a), which is shown in Fig. 2. d-spacings (interplanar distance) of independent diffraction spots, nearest the (000) transmitted spot were measured as 3.60 and 1.16 Å, respectively. The angles between diffraction spots were also measured. These d-spacings and angles are consistent with those of the $Cr_2Zr$ having a diamond structure [2]. The final indexing of the SADP of $Cr_2Zr$ along with a zone axis of [011] is shown in Fig. 2.

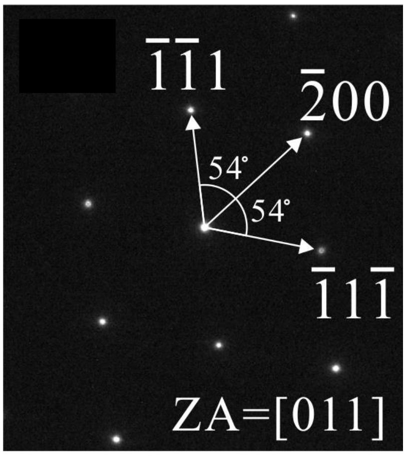

Fig. 2. SADP taken from inter-diffusion layer in Fig 1(a).

## 3. Conclusions

To improve the oxidation resistance of Zr alloy, CrAl alloy was coated on the Zr alloy specimens. The coated sample were oxidized at 1200 ℃ in steam environment for 300 s and showed extremely low oxidation when compared to uncoated Zr alloy specimens (not shown here). Microstructure and elemental distribution of the inter-diffusion layer formed between CrAl and Zr alloy have been investigated. Even after exposure at high temperature of 1200 ℃, inter-diffusion between CrAl alloy and Zr alloy occurred in very limited areas near the interface. Analyzed phase of inter-diffusion layer was $Cr_2Zr$, which is confirmed by HRTEM and EDS measurement.

## REFERENCES

[1] D.J. Park, H.G. Kim, Y.I. Jung, J.H. Park, J.H. Yang, and Y. H. Koo, “Behavior of an improved Zr fuel cladding with oxidation resistant coating under loss-of-coolant accident conditions”, Journal of Nuclear Materials, Vol.482, p.75-82, 2016.

[2] Golden Book of Phase Transitions, Wroclaw, Vol.1, pp.1-123, 2002