A Microstuctural Study on Accelerated Zirconium Alloy Oxidation

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1. Introduction

It has been reported that the effect of thermal redistribution of hydrides across the zirconium metal-oxide interface, coupled with thermal feedback on the metal-oxide interface, is a dominating factor in the accelerated oxidation in zirconium alloys cladding PWR fuel. Basically this influence determines characteristic of oxide layer. Influence estimation for corrosion oxide layer due to hydrogen / hydride carried out because of investigation on the kinetic on accelerated oxidation due to hydride precipitation was preceded. Generally, it is known that ZrO_2 tetragonal layer structures play an important role as a barrier layer. So analysing the ZrO_2 monoclinic and tetragonal structure distribution is our main aim. Especially, this study focused on the hydride effects. In other words, the difference of crystal structure distribution between pre-hydrided and without hydrided specimen is just expected results. Experimental results of microstructure at zirconium metal-oxide interface through TEM and EBSD analysis was confirmed.

2. Methods and Results

TEM analysis were performed on the specimens that have 0.916 ㎛ and 2.15 ㎛ oxide layer, respectively. The specimen has 2.15 ㎛ oxide layer was pre-hydrided(607ppm). Twin Autoclave System and Multi-purpose Apparatus were used to produce oxide film and pre-hydrided specimen. Hydrogen content was analyzed with the hydrogen determinator (RH-404) from LECO Corp. The specimen was cut, ground, polished, and dimpled to a thin cross section specimen 20 ㎛ in thickness. Ion milling was performed to finalize the foil. And the oxide layers were examined in cross section using JEOL JEM2100F TEM and 1MeV HR-TEM instruments in KBSI(Korea Basic Science Institute). FFT patterns and lattice images were obtained. EBSD specimen was 10 ㎛ surface oxided zirconium. It was prepared by ion milling procedure to have flat and clean surface. And the metal-oxide interface was analyzed using JSM 6500F with EBSD & EDS systems.

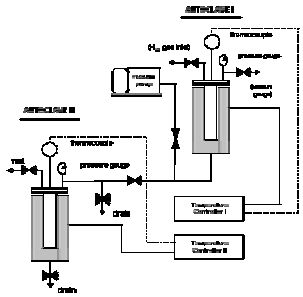

Figure 1. High pressure and temperature Twin Autoclave System

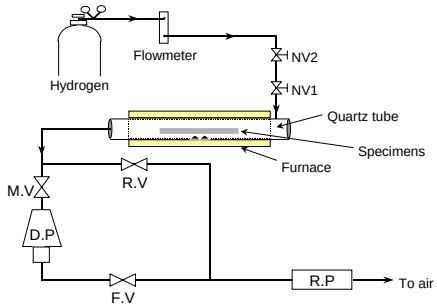

Figure 2. Multi-purpose Apparatus (Hydrogen pre-charging)

2.1 TEM Investigations

Lattice image and FFT pattern analysis of oxide layer was carried out with HR-TEM. Figure 3 shows lattice image of interface region of 0.916 ㎛ zirconium specimen.

And figure 4 shows lattice image of interface region of 2.15 ㎛ zirconium specimen(607ppm hydrided).

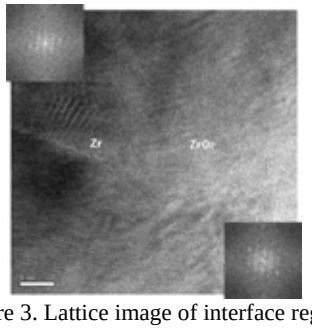

Figure 3. Lattice image of interface region

(0.916um, w/o hyd.)

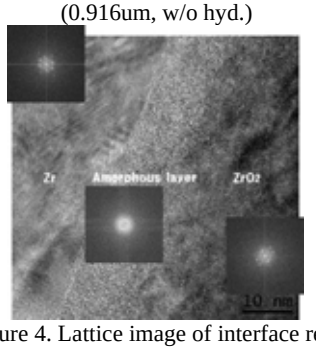

Figure 4. Lattice image of interface region
(2.15um, 607ppm hyd.)

2.2 Crystal structure distribution analysis using EBSD

EBSD system of Oxford INCA Crystal was used to analyze the crystal structure distribution of metal-oxide interface. Figure 5 shows SEM image and crystal structure of interface. It is very important to have perfectly polished surface of the sample for the reasonable results of EBSD analysis. So more careful and thorough examination of the characteristics of oxide interface must be proceeded.

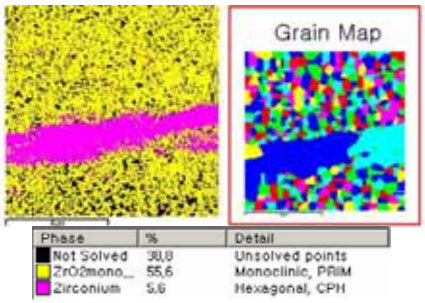

Figure 5. Grain map of interface using EBSD
(10㎛, w/o hyd.)

3. Conclusion

The microstructure of ZrO_2 grown on zirconium metal was investigated by HR-TEM and EBSD system. From the lattice image of interface, pre-hydrided specimen has more rough image than the specimen of without hydrided specimen. And about 10㎚ amorphous layer was founded at the interface region of pre-hydrided specimen. At the interface region of without hydrided specimen, there wasn't it. Crystal structure analysis by FFT patterns is proceeding . And the results of crystal structure analysis at the interface region will be got. Crystal structure distribution analysis at the interface region by EBSD system is very interesting because it shows the whole distributions by easily visible color indexing. But the mapping procedure requires very well polished specimen. Though the results from EBSD system were not perfectly good, more trial and error will be done for the expected results. And then, various zirconium alloys will be analyzed.

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