Determination of Chemical Structure for Oxidized Titanium layer by Micro-XRD

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1. Introduction

Micro-XRD system is being developed in our laboratory to analyze the chemical structure in a narrow region of nuclear materials and nuclear fuels. In this study, inside oxidation of titanium metal occurring due to oxygen uptake from the air at high temperature was investigated. Titanium alloys have been considered as the candidate materials for the heat-exchange tubes in the steam generators in a small-sized advanced integral pressurized water reactor (PWR) due to their excellent corrosion resistance, good mechanical properties and suitable durability characteristics [1-3].

The determination for the chemical structure of oxidized titanium specimen is needed at 30 – 50 μm intervals because the thickness of the oxide layer is below some hundreds μm. The x-ray micro beam of the line type is effective to increase the intensity of the diffraction peaks in a narrow region [4]. The chemical structure for the cross section of oxidized titanium specimen was determined by the micro-XRD developed [5] with a micro beam alignment system and a sample micro translation system. The application of the micro-XRD for the determination of chemical structure for a radioactive material in a narrow region was evaluated.

2. Experimental and Results

2.1 Micro-XRD system

The micro-XRD was consisted of a micro beam alignment system and a sample micro translation system. The beam size through the micro beam concentrator was 4,000 × 20 μm. The sample micro translator could move the specimen at 5 μm (min.) intervals. The x-ray source was Cu K_α line, the x-ray generation power was 40 kV - 40 mA, the measuring time / step was 40sec. / 0.04°(2θ), the filter used to eliminate the Cu K_β line was Ni filter, the detector was a scintillation counter, the detection slit width was 0.6 mm and the scanning range was $26^o < 2\theta < 43^o$.

2.2 Preparation of oxidized titanium specimen

Titanium metal (size : 10 mm × 9 mm × 3 mm) was heated at 1100 °C for 30 minutes in the atmospheric condition to examine inside oxidation of it. Oxidized titanium specimen was cut and molded with the epoxy resin. Figure 1 is the optical micrograph of the cross section of oxidized titanium specimen and shows the titanium oxide layer (about 140 μm in depth) at the edge. The chemical structure of oxidized titanium specimen was examined by the micro-XRD developed.

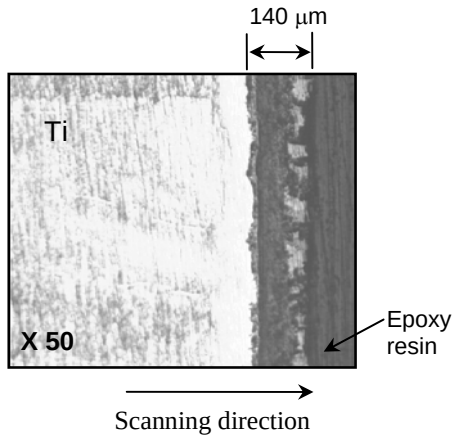

Figure 1. Cross section of oxidized titanium specimen

2.3 Determination of chemical structure for cross section of oxidized titanium specimen

The x-ray diffraction patterns were measured at 50 μm intervals from the center to the edge of oxidized titanium specimen(Figure 2). In the result, titanium was not oxidized at the center and oxidized to TiO and TiO-TiO_2 mixture inside the edge, and oxidized to TiO_2 at the edge. TiO_2 layer was also identified by the optical microscope but TiO layer, TiO-TiO_2 mixture layer were not identified by it due to the same color as Ti metal. But, the chemical structures and the thickness of the individual layers were identified by the micro-XRD system developed. TiO_2 layer was about 150 μm thick, TiO – TiO_2 mixture layer was about 80 μm thick, and TiO layer was 120 μm thick.

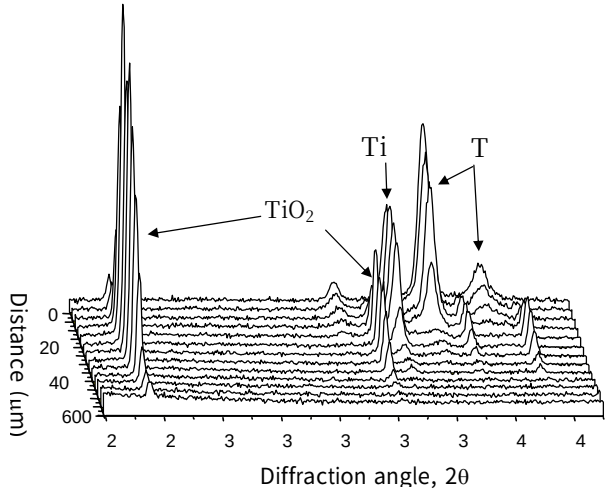

Figure 2. X-ray diffraction peak intensity vs. diffraction angle (2θ) at various distances for oxidized titanium specimen

3. Conclusion

The chemical structure change for the cross section of oxidized titanium specimen was measured at 50 ㎛ intervals in a narrow region by the micro-XRD system (beam size: 4,000 × 20 ㎛) developed. Therefore, in the future, the micro-XRD system will be installed with gamma shielding, and the structural change in the narrow region of the irradiated fuels (rim of spent fuel) and the radioactive samples will be measured with the shielded system.

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