X-RAY DIFFRACTION ANALYSIS ON PRECIPITATES OF 11J IRRADIATED RAFS—H. Tanigawa (Japan Atomic Energy Research Institute), H. Sakasegawa (Kyoto University), E. A. Payzant, S. J. Zinkle, and R. L. Klueh (Oak Ridge National Laboratory), and A. Kohyama (Kyoto University)

OBJECTIVE

The objective of this work is to analyze the precipitation behavior of irradiated reduced activation ferritic/martensitic steels (RAFs) by X-ray diffraction (XRD) analysis.

SUMMARY

XRD analyses were performed on the extraction residue of HFIR 11J-irradiated RAFs to investigate the overall precipitate character. Un-irradiated and aged specimens were examined as well. Results suggested that the distinctive peaks of $M_{23}C_6$ (M; Cr, Fe, W) were observed on all specimens. Peaks possibly related to MX (M;Ta,Ti,V : X ; C, N) were observed on the specimens extracted from un-irradiated JLF-1 and ORNL9Cr, but those peaks were not observed on irradiated specimens.

PROGRESS AND STATUS

Introduction

XRD analysis on an extraction residue specimen is the commonly used technique to analyze the precipitate characteristics on the R&D of steels in an irradiation-free condition. This technique has not been used for irradiated steels because of the practical limitation on making and handling the neutron - irradiated specimens. Transmission electron microscope (TEM) observation on thin film specimens or on extraction replica specimens is the most commonly used technique for precipitate analysis [1,2]. The conclusion obtained from TEM thin film and extraction replica specimens is that the results usually represent a very local microstructure, and it requires a lot of work to obtain a certain level of statistical reliability on the results. In order to cover the above problem, the authors decided to develop the extraction residue procedure to perform XRD analysis on irradiated steels.

In this study, XRD analyses were performed on the extraction residue of HFIR 11J-irradiated RAFs, which showed a variety of brittleness-hardness combination, to investigate the overall precipitate character. The un-irradiated and aged RAFs were also examined for comparison.

Experimental

The material used was IEA-modified F82H (F82H-IEA). Base metal with two heat treatment variations (standard IEA heat treatment and another heat treatment designated HT2) was irradiated. ORNL9Cr-2WVTa (ORNL9Cr), JLF-1 HFIR heat (JLF-1) and 2% natural Ni-doped F82H (F82H+2Ni) were also irradiated for comparison. Details of the chemical compositions and the heat treatments are shown in other reports [3,4]. Irradiation was performed in the Oak Ridge National Laboratory (ORNL) High Flux Isotope Reactor (HFIR) up to 5 dpa at 573K in the removable beryllium (RB) position. Specimens selected to be used were the 1/3-size Charpy specimens. Unirradiated (normalized-and-tempered) specimens of all materials and aged specimens of ORNL9Cr (873K for 10k h) were also used for comparison. The extraction residue from these specimens was collected on a filter with 200 nm pore size [5].

Samples were analyzed using PANalytical X'Pert Pro MPD x-ray diffractometer with a Ni-filtered Cu K-alpha radiation source at 45kV and 40mA. The diffractometer was configured with an incident beam multilayer mirror optic and diffracted beam parallel plate collimator, which yield parallel beam optics and thereby minimize sample surface displacement errors (mainly associated with sample mounting). Axial divergence limiting Soller slits were used on both the incident and diffracted beam, and the detector was a gas proportional counter. The fine powders were examined as-deposited on the filter materials and radiological safety requirements necessitated covering the powders with a thin film of Mylar (6.35μm thick)