ANALYSIS OF EXTRACTION RESIDUE OF HFIR 11J-IRRADIATED RAFS—H. Tanigawa (Japan Atomic Energy Research Institute), H. Sakasegawa (Kyoto University), S. J. Zinkle, 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 measuring the weight change of extraction residue.

SUMMARY

Extraction residue was made from several HFIR 11J-irradiated RAFs, and the mass change was measured to investigate the irradiation-enhanced change in precipitation. Two different types of filter with coarse and fine pores were used in order to separate the difference of irradiation effects between larger and smaller precipitates. Unirradiated specimens were examined as well. Results suggest that during irradiation the mass of larger precipitates increased in F82H-IEA, Ni-doped F82H, JLF-1 and ORNL9Cr, fine precipitates disappeared in JLF-1, and fine precipitates increased in Ni-doped F82H.

PROGRESS AND STATUS

Introduction

Measuring the mass change of extraction residue is the common technique to investigate the state of precipitates in R&D of steels. Until now this technique has not been used for the analysis of precipitation in irradiated steels, because of the expected practical difficulty in performing this technique. As for this, the irradiation effect on precipitation has been examined by observing TEM thin film or extraction replica samples [1,2]. The information obtained by this TEM observation is limited to the very local 2D information, and it is very difficult to obtain the overall tendency on precipitation changes with high statistical accuracy. Therefore, it is desirable to obtain a more accurate assessment on precipitation behavior, and such information could be obtained if the mass changes of extraction residue after irradiation could be determined.

It was reported that RAFs irradiated in HFIR 11J (5dpa/573K) show a variety of hardening and DBTT shifts [3]. TEM microstructure observation on these specimens revealed that the dislocation microstructure of these irradiated RAFs did not show a big enough difference to explain the difference in mechanical properties [4]. To look for the possibility that the precipitation was affected by the irradiation and caused these mechanical property differences, precipitates were extracted from 11J-irradiated RAFs to investigate the changes in precipitation after irradiation. Unirradiated (normalized-and-tempered) specimens were also measured for comparison.

Experimental

The material used for irradiation was IEA-modified F82H (F82H-IEA) and another heat treatment of F82H designated HT2 (F82H HT2), ORNL9Cr-2WVTa (ORNL9Cr), JLF-1 HFIR heat (JLF-1) and a 2% natural Ni-doped F82H (F82H+2Ni). Details of the chemical compositions and the heat treatments are shown in another report [3]. 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 1/3-size Charpy specimens. Unirradiated specimens of all materials were also used for comparison.

The precipitates were extracted electrochemically in a solution containing 10% hydrochloric acid in methanol at a potential of about 1.5V with respect to the platinum electrode. The specimen was slightly polished before extraction to remove surface microstructure. The filters used for vacuum filtering are a coarse filter (pore size 1μm) and a fine filter (pore size 200nm). The mass of the specimen and filter were measured with an accuracy of ±0.1 mg before and after extraction.

Results and Discussion

The mass of extracted residue before and after irradiation is shown in Fig. 1. Here the mass of residue obtained with the coarse filter (column denoted “Large”) is interpreted as the value corresponding to the mass of large precipitates, and the difference of mass obtained with fine filter and coarse filter (column denoted “Small”) is interpreted as that of the fine precipitates. These results indicate (1) the mass of larger precipitates increased in F82H-IEA, F82H+2Ni, JLF-1 and ORNL9Cr, (2) no mass change in F82H HT2, (3) small precipitates disappeared in JLF-1, and (4) small precipitates increased in F82H+2Ni. These results suggest that there is an apparent effect of irradiation on precipitation except for F82H HT2, even if irradiated only at 573K to 5dpa.

It is also worth noting that these increases of precipitate mass have good correlation with hardening, as shown in Fig. 2, i.e., the increases of yield stress of all RAFs except F82H-IEA are on the linear trend against the increase of precipitate mass. The reason that F82H-IEA is off this trend could be explained as the effect of the difference of prior austenite grain (PAG) size, since F82H-IEA has a large PAG size (ASTM grain size 3) than the other RAFs (ASTM grain size 7~8).This suggest the possibility that this irradiation-enhanced precipitation might cause additional irradiation hardening. Further analysis of this relationship will be performed in near future.

SUMMARY AND CONCLUSIONS

Extraction residue was obtained from HFIR-11J irradiated RAFs, and the mass change was measured to investigate the irradiation-enhanced change in precipitation. Two different types of filter with coarse and fine pores were used in order to separate the difference of irradiation effects between larger precipitate and the smaller precipitate. Unirradiated specimens were examined as well. The following is a summary of the important conclusions:

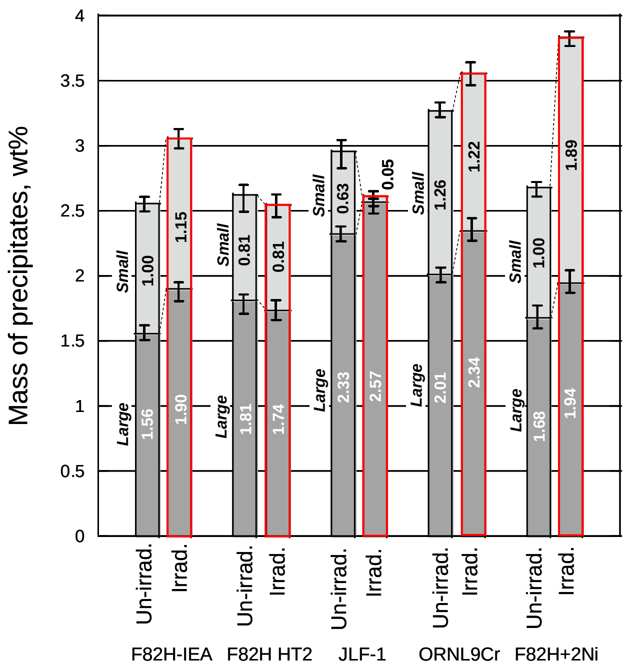

Fig. 1. Mass of extracted residue of unirradiated and irradiated RAFs extracted with coarse filter (Large) and fine filter (Large+Small). Number in the columns shows the actual wt% values of corresponding columns.

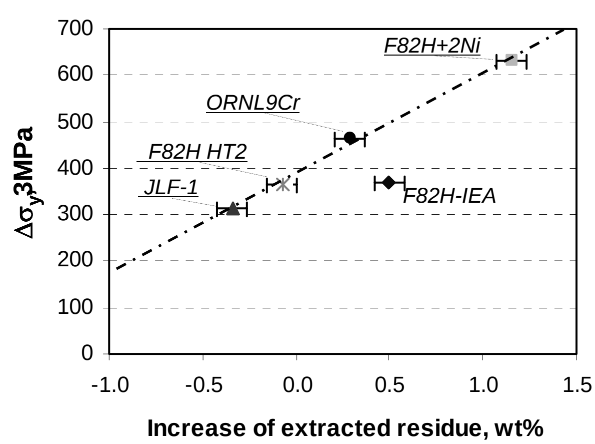

Fig. 2. Irradiation hardening plotted against the mass changes of precipitates of each RAFs.

(1) The mass of larger precipitates increased in F82H-IEA, F82H+2Ni, JLF-1 and ORNL9Cr after irradiation.
(2) The fine precipitates disappeared in JLF-1 after irradiation.
(3) The fine precipitates increased in F82H+2Ni after irradiation.
(4) Correlation between irradiation hardening and the mass change of precipitates was suggested.

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