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# Fracture toughness and Charpy impact properties of several RAFMS before and after irradiation in HFIR

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#### Abstract

As part of the development of candidate reduced-activation ferritic steels for fusion applications, several steels, namely F82H, 9Cr–2WVTa steels and F82H weld metal, are being investigated in the joint DOE–JAEA collaboration program. Within this program, three capsules containing a variety of specimen designs were irradiated at two design temperatures in the ORNL High Flux Isotope Reactor (HFIR). Two capsules, RB-11J and RB-12J, were irradiated in the HFIR removable beryllium positions with europium oxide (Eu<sub>2</sub>O<sub>3</sub>) thermal neutron shields in place. Specimens were irradiated up to 5 dpa. Capsule JP25 was irradiated in the HFIR target position to 20 dpa. The design temperatures were 300 °C and 500 °C. Precracked third-sized V-notch Charpy ( $3.3 \times 3.3 \times 25.4$  mm) and 0.18T DC(T) specimens were tested to determine transition and ductile shelf fracture toughness before and after irradiation. The master curve methodology was applied to evaluate the fracture toughness transition temperature,  $T_0$ . Irradiation induced shifts of  $T_0$  and reductions of  $J_Q$  were compared with Charpy V-notch impact properties. Fracture toughness and Charpy shifts were also compared to hardening results.

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## 1. Introduction

The reduced-activation ferritic-martensitic steels (RAFMS) are the primary- candidate materials for fusion applications, and they are being investigated in the joint US Department of Energy-Japan Atomic Energy Agency (US DOE–JAEA) collaboration program. Within this program, three capsules containing a variety of specimen designs were irradiated at

two design temperatures in the ORNL High Flux Isotope Reactor (HFIR). The potential for application of RAFMS as the structural material for fusion power plants depends on their ability to maintain adequate levels of fracture toughness at the operating temperatures and neutron doses. At present, only limited data on the effects of radiation on fracture toughness of RAFMS are available. In this study, the effects of irradiation to 20 dpa in the temperature range 250–500 °C on fracture toughness and Charpy impact properties of the RAFMS F82H and ORNL 9Cr–2WVTa steels and F82H weld metal were investigated.

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#### 2. Materials and experimental procedures

The materials used in this research were IEAmodified F82H steel (F82H-IEA) and its TIG weldments (weld metals and weld joint), and ORNL 9Cr-2WVTa steel. In order to study the effects of prior austenite grain size on radiation embrittlement of RAFMS, part of a F82H-IEA plate was renormalized at a lower austenization temperature (920 °C) to obtain a finer grain structure (F82H-HT2). Chemical compositions and heat treatments of RAFMS are summarized in Table 1.

Three capsules containing a variety of specimen designs were irradiated at two design temperatures in the ORNL High Flux Isotope Reactor (HFIR). Two capsules, RB-11J and RB-12J, were irradiated in the HFIR removable beryllium positions with europium oxide ( $Eu_2O_3$ ) thermal neutron shields in place for neutron spectrum tailoring. Specimens were irradiated up to 5 dpa at the design temperatures of 300 °C and 500 °C. Details of the irradiation conditions and the loading of the capsules can be found elsewhere [1].

The bottom and top parts of these capsules were loaded with disk-shaped compact tension [DC(T)]specimens that were used for fracture toughness characterization. The small (12.5 mm-diam and 4.6 mm-thick) DC(T) specimen was developed at ORNL for testing irradiated materials [2]. These specimens were irradiated in each 'low-' and 'high-' irradiation temperature capsule to  $\sim$ 3.8 dpa. Irradiation temperatures were measured by thermocouples. In the low-temperature capsule, specimens were irradiated at an average temperature of 250 °C; temperature variation during irradiation was within  $\pm 19$  °C for a given specimen. In the high-temperature capsule, all six specimens were irradiated at an average temperature of 377 °C in the bottom part of the capsule; temperature variation during irradiation was within  $\pm 30$  °C for a given specimen.

In addition to DC(T) specimens, miniature SS-3 type sheet tensile specimens and 1/3-size Charpy specimens both with regular V-notch (CVN) and

precracked (PCVN) to a/W ratio of ~0.5 were irradiated in both capsules. Dimensions of the SS-3 specimens were 7.62 mm gage length, 1.52 mm gage width and 0.76 mm gage thickness, and the CVN/ PCVN specimens were  $3.3 \times 3.3 \times 25.4 \text{ mm}^3$  with a 0.51 mm-deep 30 °C V-notch and a 0.05-0.08 mm root radius. Tensile and Charpy specimens were irradiated in the middle sections of their capsules. In the 'low-temperature' capsule, tensile specimens were irradiated at an average temperature of 307 °C, and 1/3-size Charpy specimens were irradiated at an average temperature of 288 °C to  $\sim$ 4.7 dpa. In the 'high-temperature' capsule, tensile specimens were irradiated at an average temperature of 497 °C, and 1/3 size Charpy specimens were irradiated at an average temperature of 509 °C to  $\sim$ 4.8 dpa.

Capsule JP25 containing tensile and Charpy specimens was irradiated in target position to 20 dpa. The design temperatures were 300 °C and 500 °C. However, the post-irradiation study revealed that actual temperatures were 380 °C and 500 °C instead of designed 300 °C and 500 °C.

## 3. Results and discussion

The fracture toughness tests in the transition range were conducted in general accordance with the master curve methodology, see details in [3]. This methodology, proposed by Wallin [4,5], uses a concept of the universal temperature dependence of fracture toughness in the transition region. The master curve methodology received a wide application for low-alloyed reactor pressure vessel steels. The current physical background for this methodology suggests that it is applicable to a wide variety of ferritic bcc steels, including tempered ferriticmartensitic steels like RAFMS, yet the transition fracture toughness data for this class of steels are rather sparse. The biggest advantage of this methodology is the ability to describe temperature dependence of fracture toughness in the transition region by means of testing of a relatively low number of small size specimens. This is a great

Table 1 Chemical composition and heat treatments of RAFM steels studied

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Steel	C (wt%)	Cr (wt%)	W (wt%)	V (wt%)	Ta (wt%)	N (wt%)	Heat treatment
F82H-IEA	0.11	7.7	2.0	0.16	0.02	0.008	1040C/40 min/AC + 750C/1 h (IEA)
F82H-HT2	0.11	7.7	2.0	0.16	0.02	0.008	(IEA) + 920 C/1 h/AC + 750 C/1 h
9Cr–2WVTa	0.10	8.8	1.97	0.18	0.065	0.004	1050 C/1 h/AC + 750 C/1 h

recipe for post-irradiation characterization of RAFM steels for fusion application, including this study. However, the ASTM standard E1921 prescribes several validity requirements. For example, the specimen remaining ligament,  $b_0$ , must have sufficient size to maintain a condition of high crack-front constraint at fracture. A  $K_{Jc}$  datum is considered invalid if this value exceeded the  $K_{Jc(limit)}$  requirement of the ASTM Standard E 1921:

$$K_{\rm Jc(limit)} = \sqrt{\frac{b_0 \sigma_{\rm YS}}{30} \cdot \frac{E}{1 - \nu^2}},\tag{1}$$

where  $\sigma_{\rm YS}$  is the yield strength of the material at the test temperature. Some small specimen data may need constraint correction prior to application of E1921 analysis like the one suggested by Odette et al. [6,7]. Most of the irradiated  $K_{\rm Jc}$  data in this study satisfied the  $K_{\rm Jc(limit)}$  requirement of the ASTM Standard E 1921, Eq. (1). Thus, no constraint correction was made to the data.

The ASTM standard E1921 also requires of a certain number of specimens to be tested within  $T_0 \pm 50$  °C range, where  $T_0$  is the transition fracture toughness temperature. Unfortunately, this requirement of the E1921 standard was not satisfied in this study because too few specimens were irradiated in the capsules and some of them were tested at temperatures below  $T_0 - 50$  °C to satisfy requirement in Eq. (1). Thus, the current  $T_0$  values should be considered as estimate values only.

The DC(T) specimens of F82H-IEA steel were machined in T–L orientation and PCVN specimens were machined in L–T orientation and irradiated in the temperature range 250–500 °C. Shifts of  $T_0$  of F82H-IEA varied from 33 °C when irradiated at 500 °C to 191 °C for irradiation at 250 °C. Shifts of Charpy ductile-to-brittle transition temperature (DBTT) were similar to  $T_0$  shifts. These results are summarized in Fig. 1, which illustrates the effect of irradiation temperature and dose on radiation embrittlement of F82H-IEA steel.

As mentioned above, tensile properties were determined by testing miniature SS-3 sheet-tensile specimens. Fig. 2 shows the temperature dependence of the yield strength of F82H steel before and after irradiation. Specimens tested after irradiation at  $\sim$ 500 °C to 5.0 dpa and 20 dpa did not show any noticeable hardening as a result of the irradiation. It is interesting to note that both fracture toughness and Charpy data for this irradiation temperature indicated small but noticeable embrittle-



Fig. 1. The effect of irradiation temperature and dose on the transition temperature shift of F82H-IEA steel.



Fig. 2. Yield strength of F82H-IEA steel in the unirradiated condition and after irradiation in HFIR at different temperatures and doses.

ment, Fig. 1. Irradiation at  $\sim$ 300 °C to 4.7 dpa resulted in significant hardening of F82H-IEA steel with room temperature yield strength increased by 368 MPa (from 528 MPa in the unirradiated condition to 896 MPa after irradiation). Data after irradiation at 380 °C show that the 80 °C difference in the irradiation temperature (between 300 °C and 380 °C) is more important for radiation hardening than the increase in dose from 5 to 20 dpa.

Since several materials were tested, this study provided a rare chance to examine the relationship between hardening and fracture toughness shifts of RAFMS. Fig. 3 shows the relationship between hardening and  $\Delta T_0$  for several RAFMS from this study and Eurofer97 steel from published data [8,9]. This relationship is compared to a large database generated from low-alloy reactor pressure vessel (RPV) steels [10]. The current data show that the



Fig. 3. Relationship between fracture toughness shift and hardening for RAFM steels.

8–9%Cr RAFMS exhibit less embrittlement per unit of hardening than do low-alloy RPV steels. These results are in agreement with the recent work by Odette et al. [11] that predicted less embrittlement per unit of hardening for RAFMS than for RPV steels. However, the coefficient of relationship from the present study:

$$\Delta T_0 = 0.3 \cdot \Delta \sigma_{\rm y} \tag{2}$$

is somewhat less than that predicted from [11] and observed in [9]. Given a relatively small number of miniaturized fracture toughness specimens used in this study to determine  $T_0$  shifts, the absolute value of this coefficient should be considered as a preliminary. Future work will provide additional information on this discrepancy.

Both CVN and PCVN specimens were irradiated at the same temperature to the same dose. This



Fig. 4. Relationship between fracture toughness and Charpy impact transition temperature shifts for different steels.



Fig. 5. Charpy impact (a, c) and fracture toughness (b, d) data of F82H steel with 125 µm (a, b) and 55 µm (c, d) prior austenite grain size.

provided an opportunity to compare the shifts of fracture toughness,  $T_0$ , and Charpy DBTT shifts, see Fig. 4. Once again, Eurofer97 data [8,9] were used. The RAFMS data are in the general scatter band observed for RPV steels [10], although Eurofer97 data tend to be on the upper part of the scatter band.

In order to study the effects of prior austenite grain size on radiation embrittlement of RAFMS, part of a F82H-IEA plate was renormalized at a lower austenization temperature (920 °C) to obtain a finer prior austenite grain structure (F82H-HT2). The additional heat treatment resulted in reduction of the prior austenite grain size from 125 to 55  $\mu$ m. Fracture toughness and Charpy data showed that the finer grain structure and higher upper-shelf energy before irradiation, but no effect was observed on transition temperature shift after irradiation, Fig. 5.

Charpy CVN specimens of F82H TIG weld metal and weld joints were irradiated with the CVN specimens of F82H-IEA steel. The present



Fig. 6. J-R curves of F82H-HT2 (a) and 9Cr–2WVTa (b) steels before and after irradiation in HFIR.

study showed that TIG weld metal and weld joints exhibited embrittlement similar to F82H base metal, see Ref. [12].

Some of the DC(T) specimens of RAFMS irradiated in RB-11J and RB-12J were tested at temperatures above the transition range to study the effects of radiation on the resistance to stable crack growth of RAFMS. It was found that for all RAFMS, irradiation caused a reduction of tearing resistance and a slight reduction in  $J_{Ic}$ , see Fig. 6 for examples. This is in general accordance with behavior of lowalloy ferritic RPV steels. Overall, RAFMS exhibited high ductile fracture toughness both before and after irradiation to ~5 dpa.

## 4. Summary

The RAFM steels are being investigated in the joint DOE–JAEA collaborative irradiation program. As part of this program, three capsules, containing a variety of specimen designs were irradiated in the ORNL High Flux Isotope Reactor in the temperature range 250–500 °C and doses from 3.8 to 20 dpa. The current observations can be summarized as follows:

- F82H-IEA steel exhibited fracture toughness transition temperature shifts from 33 °C after irradiation at 500 °C to 191 °C after irradiation at 250 °C, for doses of 3.8–5.0 dpa. Shifts measured with DC(T) and PCVN specimens appear to be in good agreement. An increase in the dose from 5 dpa to 20 dpa at 500 °C did not result in any noticeable differences in transition temperature shifts. The same increase in dose at 380 °C resulted in increase of transition temperature shift from 57 °C to 109 °C.
- 2. The current 8–9% Cr RAFM steels exhibited less embrittlement per unit of hardening than lowalloyed RPV steels. Comparison of fracture toughness and Charpy impact shifts show they are in agreement with published data.
- 3. The RAFM steel exhibited high-ductile fracture toughness. Irradiation reduces tearing resistance and, to a lesser degree, reduces  $J_{Ic}$  values.
- 4. Reduction of the prior austenite grain size from 125 to  $55 \,\mu\text{m}$  resulted in a slightly lower transition temperature and higher upper-shelf energy in the unirradiated condition, but no effect was observed on radiation-induced shifts.
- 5. F82H TIG weld metal and weld joints exhibited similar embrittlement to F82H-IEA base metal.

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