

**FRACTURE TOUGHNESS CHARACTERIZATION OF IRRADIATED F82H IN THE TRANSITION REGION**— M. A. Sokolov, R. L. Klueh (Oak Ridge National Laboratory), G. R. Odette (University of California, Santa Barbara), K. Shiba, and H. Tanigawa (Japan Atomic Energy Research Institute)

## **OBJECTIVE**

The objective of this work is to characterize the fracture behavior of ferritic/martensitic steels.

## **SUMMARY**

The ferritic-martensitic steel F82H is a primary candidate low-activation material for fusion applications, and it is being investigated in the joint U.S. Department of Energy-Japan Atomic Energy Research Institute collaboration program. As part of this program, two capsules containing a variety of specimen designs were irradiated at two different temperatures in the Oak Ridge National Laboratory (ORNL) High Flux Isotope Reactor. 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. This small (12.5 mm in diameter with thickness of 4.6 mm) DC(T) specimen was developed at ORNL for testing irradiated materials. Six specimens were irradiated in each “low-“ and “high-“ irradiation temperature capsule up to ~3.8 dpa. Irradiation temperatures were measured by thermocouples. In the low-temperature capsule, three specimens were irradiated at an average temperature of 261°C and another three at 240°C; temperature variation during irradiation was within  $\pm 19^\circ\text{C}$  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  $\pm 30^\circ\text{C}$  for a given specimen. All irradiated specimens failed by cleavage instability. From these data, fracture toughness transition temperatures were evaluated for irradiated F82H steel and compared to unirradiated values. Specimens irradiated at the higher temperature exhibited a relatively modest shift of the fracture toughness transition temperature of  $\sim 57^\circ\text{C}$ . However, the shift of fracture toughness transition temperature of specimens irradiated at 250°C was much larger,  $\sim 191^\circ\text{C}$ . These results are compared with available tensile and impact Charpy data for this material.

## **PROGRESS AND STATUS**

### **Introduction**

The ferritic-martensitic steel F82H is a primary candidate low-activation material for fusion applications, and it is being investigated in the joint USA-JAERI collaboration program. As part of this program, two capsules containing a variety of specimen designs were irradiated at two different temperatures in the ORNL High Flux Isotope Reactor (HFIR). Two capsules with europium oxide ( $\text{Eu}_2\text{O}_3$ ) thermal neutron shield were irradiated in the HFIR removable beryllium (RB) positions. Details of the irradiation conditions and the contents 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 in diameter with thickness of 4.6 mm) DC(T) specimen was developed at ORNL for testing irradiated materials [2]. Six specimens were irradiated in each “low-“ and “high-“ irradiation temperature capsule up to ~3.8 dpa. Irradiation temperatures were measured by thermocouples. In the low-temperature capsule, three specimens were irradiated at an average temperature of 261°C in the bottom part of the capsule and another three at 240°C in the upper part of the capsule; temperature variation during irradiation was within  $\pm 19^\circ\text{C}$  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  $\pm 30^\circ\text{C}$  for a given specimen.

In addition to DC(T) specimens, miniature SS-3 type sheet tensile specimens (7.62 mm in gage length, 1.52 mm in gage width and 0.76 mm in gage thickness), and 1/3-size Charpy specimens (3.3 x 3.3 x 25.4 mm<sup>3</sup> with 0.51 mm deep 30° V-notch and a 0.05-0.08 mm root radius) were irradiated in both capsules. However, 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 ~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 ~4.8 dpa.

### Testing Procedure

The fracture toughness tests were conducted in general accordance with the American Society for Testing and Materials (ASTM) E 1921-02 [3] Standard Test Method for Determination of Reference Temperature,  $T_o$ , for Ferritic Steels in the Transition Range, with a computer-controlled test and data acquisition system [4]. The specimens were fatigue precracked before irradiation to a ratio of the crack length to specimen width ( $a/W$ ) of about 0.5, and then side-grooved by 20% of their thickness (10% from each side). The unloading compliance method used for measuring the J-integral using these specimens is outlined in Ref. [2]. Unirradiated specimens were tested in the laboratory on a 98-kN (22-kip) capacity servohydraulic machine, and irradiated specimens were tested in a hot cell with a 490-kN (110-kip) capacity servohydraulic machine with a 22-kN (5-kip) load cell. All tests were conducted in strain control, with an outboard clip gage having a central flexural beam that was instrumented with four strain gages in a full-bridge configuration. The broken unirradiated specimens were examined with a calibrated measuring optical microscope to determine the initial and final crack lengths. The irradiated specimens were photographed, and enlarged prints of the fracture surfaces were fastened to a digitizing table to allow the crack length to be measured.

Values of J-integral at cleavage instability,  $J_c$ , were converted to their equivalent values in terms of stress intensity  $K_{Jc}$  by the following equation [3]:

$$K_{Jc} = \sqrt{J_c \frac{E}{1-\nu^2}} \quad (1)$$

where  $E$  is Young's modulus and  $\nu = 0.3$  is Poisson's ratio.

It was assumed that the transition fracture toughness of F82H steel complied with the master curve concept. Therefore, a  $K_{Jc}$  datum was considered invalid if this value exceeded the  $K_{Jc(\text{limit})}$  requirement of the ASTM Standard E 1921 [3]:

$$K_{Jc(\text{limit})} = \sqrt{\frac{b_o \sigma_{YS}}{30} \cdot \frac{E}{1-\nu^2}} \quad (2)$$

where  $b_o$  was the remaining ligament and  $\sigma_{YS}$  was the yield strength of the material at the test temperature. All invalid data were censored and substituted by the  $K_{Jc(\text{limit})}$  values for calculation of the transition fracture toughness temperature,  $T_o$ . After that, all  $K_{Jc}$  data (valid and substituted) were converted to 1T equivalence,  $K_{Jc(1T)}$ , using size adjustment procedure of the ASTM Standard E1921 [3]:

$$K_{Jc(1T)} = 20 + \left[ K_{Jc(x)} - 20 \right] \cdot \left( \frac{B_x}{B_{1T}} \right)^{1/4} \quad (3)$$

where  $K_{Jc(x)}$  = measured  $K_{Jc}$  value,  
 $B_x$  = gross thickness of test specimen,  
 $B_{1T}$  = gross thickness of 1T C(T) specimen.

The reference fracture toughness transition temperature,  $T_o$ , was determined using the multi-temperature equation from E1921 [3]:

$$\sum_{i=1}^N \delta_i \frac{\exp[0.019(T_i - T_o)]}{11 + 77 \exp[0.019(T_i - T_o)]} - \sum_{i=1}^N \frac{(K_{Jc(i)} - 20)^4 \exp[0.019(T_i - T_o)]}{\{11 + 77 \exp[0.019(T_i - T_o)]\}^5} = 0 \quad (4)$$

where  $\delta_i$  = 1.0 if the datum is valid or zero if datum is a dummy substitute value,  
 $T_i$  = test temperature corresponding to  $K_{Jc(i)}$ .

## Results

The unirradiated DC(T) specimens were examined in two orientations, L-T and T-L, while only T-L-oriented DC(T) specimens were irradiated. Twenty-three T-L specimens were tested in the unirradiated condition in the temperature range from -100°C to -20°C and 16 L-T specimens were tested in the unirradiated condition in the temperature range from -140°C to -50°C. Only two T-L and one L-T specimens exhibited stable crack growth and did not demonstrate any unstable fracture. From their full J-R curves, values of the critical integral at the onset of the stable crack growth,  $J_{Ic}$ , were determined using ASTM Standard E 1820 [5]. However, these values could not satisfy the validity requirements of E1820 because of the relatively small dimensions of the DC(T) specimens, and they were designated as  $J_q$  on the Figures. These three tests were censored for  $T_o$  determination and substituted with the  $K_{Jc(\text{limit})}$  values from Eq. (2). All other specimens failed by cleavage instability. From these data, using Eqs1-4, fracture toughness transition temperatures were evaluated for the F82H steel.

The size-adjusted to 1T fracture toughness data of F82H steel in the T-L orientation are presented in Figure 1. This figure illustrates the temperature dependence of the transition fracture toughness data relative to its master curve with 5 and 95% tolerance bounds. While 5 and 95% tolerance bounds provide a reasonable description of the transition fracture toughness data, there are a noticeable number of data points outside the bounds. Two  $J_q$  values were converted to their stress-intensity factor equivalent using Eq. (1) and plotted as filled symbols on Figure 1 to indicate the upper end of the transition region. The temperature dependence of  $K_{Jc(\text{limit})}$  is also plotted on Figure 1. It can be seen that for a given strength of F82H, this specimen design ( $b_o$ ) leaves a relatively narrow test temperature window to obtain valid  $K_{Jc}$  data. For example, out of 21 specimens that failed by cleavage, nine specimens exceeded the validity limit,  $K_{Jc(\text{limit})}$ , and were censored for  $T_o$  determination.

Figure 2 compares the fracture toughness data of F82H in the T-L and L-T orientations. It is apparent from the Fig. 2 that both the transition region and the upper shelf fracture toughness have a distinct orientation dependence. The reference transition temperature is lower in the L-T orientation; -109°C compared to -68°C in the T-L orientation. The upper-shelf toughness is higher in the L-T orientation compared to T-L. Thus overall, the F82H steel is tougher in the L-T orientation than in T-L. Such orientation dependence is typical for many ferritic steels, and the L-T orientation is usually the toughest orientation. It needs to be pointed out that the optical microscopy did not reveal an orientation dependence in the microstructure of this steel.

As mentioned above, tensile properties were determined by testing miniature SS-3 sheet-tensile specimens with a 7.62-mm gage length, a 1.52-mm gage width, and a 0.76-mm in gage thickness. Figure 3 provides the temperature dependence of the yield strength of F82H steel before and after irradiation. These results were used in the present study to evaluate the fracture toughness data. Irradiation at ~300°C to 4.5 dpa resulted in significant hardening of F82H. Room temperature yield strength increased by 368 MPa (from 528 MPa in the unirradiated condition to 896 MPa after irradiation). Specimens tested after irradiation at ~500°C to 5.0 dpa did not show any noticeable hardening as result of irradiation.

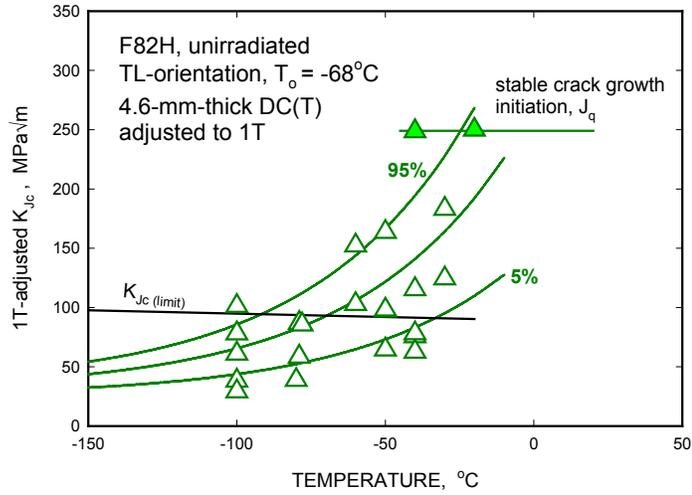


Fig. 1. Temperature dependence of fracture toughness data of F82H in T-L orientation relative to its master curve.

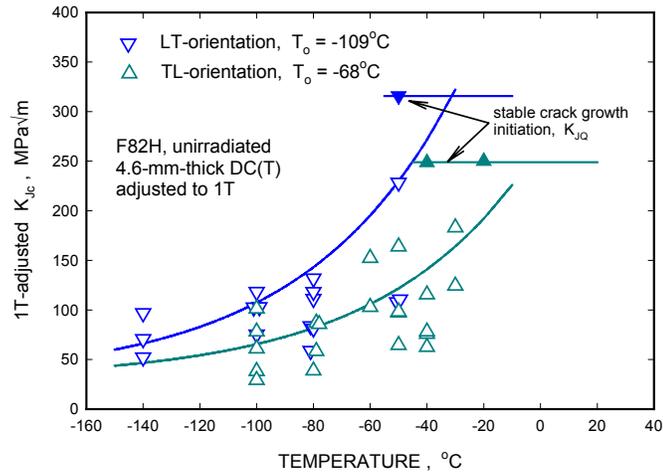


Fig. 2. Size-adjusted to 1T fracture toughness data of F82H steel in two orientations, T-L and L-T, relative to their master curves.

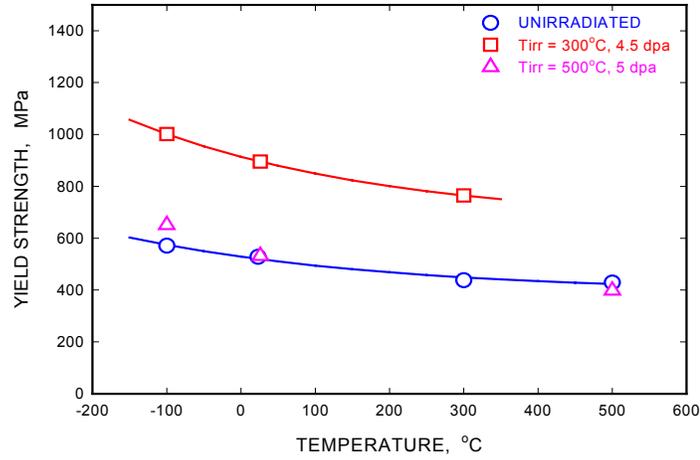


Fig. 3. Temperature dependence of yield strength of F82H Steel in the unirradiated condition and after irradiation at 300 and 500°C.

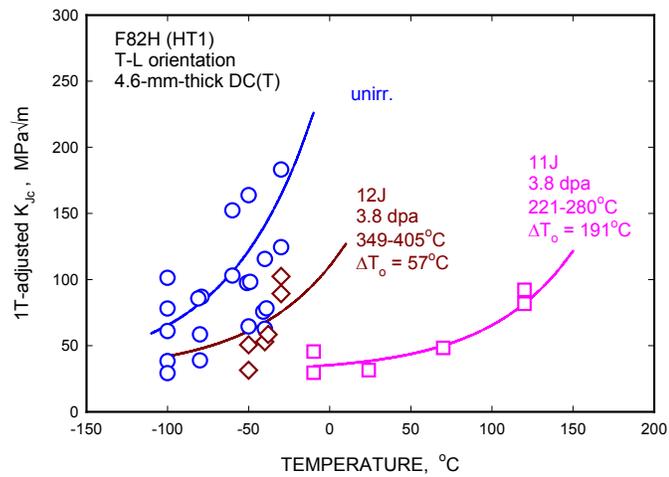


Fig. 4. Fracture toughness data of F82H steel (size-adjusted to 1T) in T-L orientation before and after irradiation.

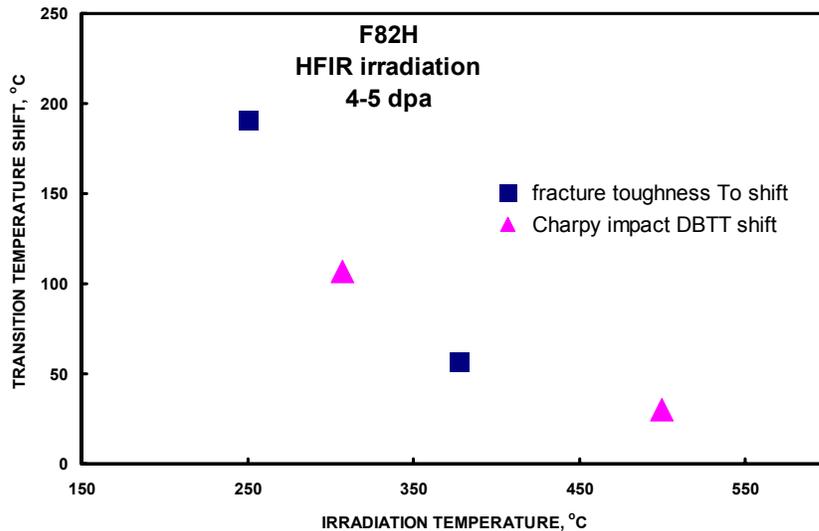


Fig. 5. Comparison of fracture toughness reference temperature  $T_o$  shifts derived from DC (T) specimens and Charpy impact DBTT shifts derived from third-sized specimens of F82H steel after irradiation in HFIR at different temperatures.

Using the following correlation between hardening and fracture toughness transition temperature shift from Ref. [6]:

$$\Delta T_o = 0.7 \cdot \Delta \sigma_{YS}, \quad (5)$$

it was estimated that the shift of fracture toughness transition temperature,  $\Delta T_o$ , after irradiation at 300°C might be about 260°C. This estimate was used to select test temperatures for irradiated fracture toughness specimens taking into account differences in irradiation temperatures of the DC(T) and tensile specimens.

Every irradiated specimen failed by cleavage instability. It turned out that all generated  $K_{Jc}$  values satisfied the validity requirement in Eq. 2. From these data, fracture toughness transition temperatures were evaluated for irradiated F82H steel and compared to the unirradiated  $T_o$ , see Fig. 4. Specimens irradiated at the higher temperature exhibited a relatively modest shift of the fracture toughness transition temperature of 57°C. However, the shift of fracture toughness transition temperature of specimens irradiated at ~250°C was much larger at 191°C.

While the shift of  $T_o$  after irradiation at an average temperature of ~250°C is relatively large, 191°C, it is still smaller than expected from correlation Eq. 5 and tensile data after irradiation at ~300°C. The same correlation also suggests a larger than 51°C shift of  $T_o$  after irradiation at an average temperature of 377°C. The correlation in Eq. 5 was based entirely on low-alloy reactor pressure vessel (RPV) steels data [6], and the present results do not support direct application of this correlation to ferritic-martensitic steels like F82H, suggesting a different relationship between embrittlement and hardening than in RPV steels. It appears that 8%Cr-based steels, like F82H, have a smaller shift of  $T_o$  per the same amount of hardening than 0.3%Cr-based RPV steels. On the other hand, shifts of the fracture toughness transition temperature,  $T_o$ , and the Charpy ductile-to-brittle transition temperature, DBTT, appear to be comparable if the irradiation temperature is taken into account (see Fig. 5).

## Summary and Conclusions

Disk compact tension specimens of F82H steel of T-L orientation were irradiated in the HFIR removable beryllium (RB) positions in capsules with a europium oxide ( $\text{Eu}_2\text{O}_3$ ) thermal neutron shield to 3.8 dpa at average temperatures of  $\sim 250^\circ\text{C}$  and  $\sim 77^\circ\text{C}$ . The master curve methodology was applied to characterize the transition fracture toughness of this steel before and after irradiation. Application of the master curve methodology in accordance with ASTM standard E1921 showed that even for such a relatively large number (for fusion irradiation experiments) of specimens, there is a very narrow test temperature range where  $K_{Jc}$  values could be qualified as valid values per E1921. In this study, all irradiated specimens exhibited cleavage fracture with E1921 valid fracture toughness values.

In the unirradiated condition, both the transition range and the upper-shelf fracture toughness exhibited strong orientation dependence. The F82H steel was tougher in the L-T orientation than in T-L orientation.

Specimens irradiated at  $\sim 377^\circ\text{C}$  exhibited a modest shift of reference fracture toughness temperature,  $T_o$  ( $57^\circ\text{C}$ ). However, the  $T_o$  shift of specimens irradiated at  $\sim 250^\circ\text{C}$  was much larger ( $191^\circ\text{C}$ ).

The present results show that relatively high-Cr ferritic-martensitic steels, like F82H, exhibit smaller embrittlement in terms of  $T_o$  shift for the same amount of hardening (yield strength increase) than low-Cr ferritic RPV steels. Shifts of  $T_o$  and Charpy DBTT temperatures appear to be the same (taking into account irradiation temperature).

## References

- [1]. K.E. Lenox and M.L. Grossbeck, "Operating History of the HFIR MFE-RB-11J and -12J (P3-2 and P3-3) Experiments," *DOE/ER-0313/25 Fusion Materials*, 30 December, 1998, pp.307-23.
- [2]. D.J. Alexander, "Fracture Toughness Measurements with Subsize Disk Compact Specimens," *Small Specimen Test Techniques Applied to Nuclear Reactor Vessel Thermal Annealing and Plant Life Extension*, ASTM STP 1204, W.R. Corwin, F.M. Haggag, and W.L. Server, Eds., ASTM, Philadelphia, 1993, pp.130-142.
- [3]. Standard Test Method for Determination of Reference Temperature,  $T_o$ , for Ferritic Steels in the Transition Range, Designation E 1921-02, Annual Book of ASTM Standards, Vol. 03.01.
- [4]. R.K. Nanstad, D.J. Alexander, R.L. Swain, J.T. Hutton, and D.L. Thomas, "A Computer Controlled Automated Test System for Fatigue and Fracture Testing," *Applications of Automation Technology for Fatigue and Fracture Testing*, ASTM STP 1092, A.A. Braun, N.E. Ashbaugh, and F.M. Smith, Eds., ASTM Philadelphia, 1990, pp. 7-20.
- [5]. Standard Test Method for Measurement of Fracture Toughness, ASTM E1820-99a, ASTM, Philadelphia, Annual Book of Standards, Vol. 3.01.
- [6]. M.A. Sokolov and R.K. Nanstad, "Comparison of Irradiation-Induced shifts of  $K_{Jc}$  and Charpy Impact Toughness for Reactor Pressure Vessel Steels," *Effects of Radiation on Materials: 18<sup>th</sup> International Symposium*, ASTM STP 1325, R.K. Nanstad, M.L. Hamilton, F.A. Garner, and A.S. Kumar, Eds., American Society for Testing and Materials, Philadelphia, 1999, pp. 167-190.