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Evaluation of fracture toughness master curve shifts for JMTR irradiated F82H using small specimens

T. Yamamoto [a,*], G.R. Odette [a], D. Gragg [a], H. Kurishita [b], H. Matsui [b], W.J. Yang [a], M. Narui [b], M. Yamazaki [b]

[a] *Department of Mechanical Engineering, University of California, Santa Barbara, Santa Barbara, CA 93106-5080, USA*
[b] *International Research Center for Nuclear Materials Science, IMR, Tohoku University, Oarai, Ibaraki 311-1313, Japan*

Abstract

Small to ultra-small 1/3 size pre-cracked Charpy and $1.65 \times 1.65 \times 9$ mm deformation and fracture minibeam (DFMB) specimens of the F82H IEA heat were irradiated to 0.02 and 0.12 dpa at 290 °C in the Japanese Materials Test Reactor. Nominal cleavage transition temperature shifts, based on the measured toughness, $K_{Jm}(T)$, data (ΔT_m) as well as reference temperature shifts (ΔT_0) found after size-adjusting the $K_{Jm}(T)$ data yielded $\Delta T_{m/0} \approx 27 \pm 10$ and 44 ± 10 at the two doses, respectively. Using measured yield stress changes ($\Delta\sigma_y$), the $C_0 = \Delta T_0/\Delta\sigma_y = 0.58 \pm 0.14$ at 0.12 dpa, is in good agreement with data in the literature. The dynamic transition temperature shift, ΔT_d, derived from DFMB tests, was $\approx 30 \pm 20$ °C at 0.1 dpa, also in good agreement with the estimated ΔT_0 shifts. The ΔT_d and ΔT_0 are also in excellent agreement with a $\Delta T_0 = C_0\Delta\sigma_y$ (dpa, T_i) hardening-shift model, where the $\Delta\sigma_y$ (dpa, T_i) was found by fitting a large database of tensile properties.

1. Introduction

A key issue in developing 8–9Cr–1–2W normalized and tempered martensitic steels (TMS) for fusion reactor applications is irradiation embrittlement, as characterized by upward shifts (ΔT_0) in the cleavage fracture toughness master curve (MC) [1–5]. At irradiation temperatures less than ≈ 400 °C, the ΔT_0 are primarily due to irradiation hardening, $\Delta\sigma_y$ [1,6,7]. However, the weakening of grain boundaries by very high levels of helium may also interact synergistically with large $\Delta\sigma_y$, resulting in very large ΔT_0 [1,6]. Assessment of ΔT_0 requires utilization of small specimens due to both limited space and high heating rates in available irradiation facilities. However, the fracture toughness measured using small specimens, K_{Jm}, is generally higher than values obtained from larger, conventional specimens, due to both statistical and constraint loss size effects [1,4,8]. It has been shown that physically based models can be used to adjust K_{Jm} data to full constraint conditions (plane strain, small scale yielding) K_{Jr} at a reference size [4,8]. For example, application of the adjustment procedure to a large F82H K_{Jm} database obtained from 13 types

* Corresponding author. Tel.: +1 805 893 3848; fax: +1 805 893 4731.
E-mail address: yamataku@engineering.ucsb.edu (T. Yamamoto).

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of specimens resulted in a self-consistent population of K_{Jr} data well described by a single MC of $T_0 \approx -100 \pm 3$ °C [4].

In the hardening dominated regime, $\Delta T_0 \approx C_0 \Delta\sigma_y$, where estimates of C_0 range from ≈ 0.7 °C/MPa for reactor pressure vessel (RPV) steels and low dose (dpa) irradiations at $T_i \approx 300$ °C [1,9] to less than ≈ 0.4 °C/MPa for higher dose TMS alloys, particularly for irradiations at lower temperatures [1,10,11]. Odette et al. reported a $C_0 \approx 0.58$ °C/MPa for F82H irradiated between ≈ 250 and 380 °C [1,10]. It has also been shown that the lower C_0 for TMS alloys and irradiation conditions, compared to the RPV case, can be attributed to much larger reduction of strain hardening after irradiation to high dose [1,10,12]. If C_0 is assumed to be approximately constant for a particular alloy and irradiation regime, then ΔT_0 can be related to $\Delta\sigma_y$, or other measures of irradiation-induced strength increases, such as measured by Vickers microhardness (ΔH_v) [13,14]. A large database on $\Delta\sigma_y$ in irradiated TMS compiled by Yamamoto et al. [6] was used to derive a semi-empirical model for $\Delta\sigma_y$ (dpa, T_i, T_t), where T_t is the test temperature. This work showed that the hardening model could be combined with hardening-shift coefficients for Charpy (C_c) tests and C_0 to predict $\Delta T_c = C_c \Delta\sigma_y$ (dpa, T_i) and $\Delta T_0 = C_0 \Delta\sigma_y$ (dpa, T_i), respectively.

In this study, we characterize the effects of low dose irradiation on the IEA heat of F82H using small to ultra-small specimens. Our primary objectives were to:

- evaluate the use and limitations of using small bend bars with dimensions one-third and one-sixth of standard Charpy specimens to evaluate static ΔT_0 (1/3PCC) and dynamic ΔT_d (so-called deformation and fracture minibeams, DFMBs) transition toughness temperature shifts;
- compare the ΔT_0 based directly on the measured toughness (K_{Jm}) to that determined from estimates of the corresponding small scale yielding toughness at a reference size (K_{Jr}) derived using a physically based size adjustment procedure;
- compare the ΔT_0 and ΔT_d to hardening model predictions and general $\Delta T_0 - \Delta\sigma_y$ trends.

2. Experiment

Detailed information on the IEA heat of F82H characterized in this study is summarized elsewhere [15]. The 1/3PCC specimens with dimensions of $W = 3.33$, $B = 3.33$ and $L = 18.3$ mm were fabricated in L–T orientation, where W, B and L are the specimen width, thickness and length, respectively. The fatigue pre-cracks were grown to nominal a/W of ≈ 0.5, where a is the crack length, at a final maximum peak stress intensity factor of ≈ 18 MPa$\sqrt{m}$. DFMBs, with dimensions $W = 1.67$, $B = 1.67$ and $L = 9.2$ mm, were fabricated in L–S orientation from pre-cracked coupons using special procedures described elsewhere [16]. SS-J2-type sheet dogbone tensile specimens, with gauge section dimensions of $W = 1.2$, $L = 5$ and $t = 0.5$ mm, were also fabricated in L-axis orientation. Irradiations were carried out in the Japan materials test reactor (JMTR) at 290 °C. The 1/3PCC and the tensile specimens were irradiated to between ≈ 0.02 and 0.12 dpa, while the DFMB specimens were irradiated to ≈ 0.1 dpa.

The post irradiation mechanical tests were carried out in the hot cell facility at the IMR-Oarai Center, Tohoku University in Oarai, Japan. The 1/3PCCs were tested statically on a screw driven load frame. The bending fixture, with a span of 13.2 mm, and specimens were immersed in a isopentane cooling bath, and stabilized to within ± 1 °C of T_t for at least ten minutes prior to testing at a displacement rate of 2 μm/s. Tensile tests were performed in the same bath at a nominal strain rate of 6.67×10^{-4} (s^{-1}) on the same load frame. The DFMB specimens were tested in a so-called IZOD configuration [17] on an oil pressure driven drop tower impact tester. An impact displacement at 1 m/s was applied by an instrumented striker at a distance 3.3 mm from the crack line, near the end the cantilevered beam specimen. Slower impact at 0.2 m/s was also performed for comparison. Note, that the effect of the warmer hammer briefly contacting the cold specimen is negligible in all cases. Data for DFMB control specimens tested at UCSB, both in three point bending and in a similar IZOD configuration, were generally consistent with the Oarai results. In all cases the load–time data was converted to load–displacement curves that were analyzed for K_{Jm} and K_{Jd} using the procedures in ASTM E1921-05 [5].

2.1. Size effect adjustments of fracture toughness data

Cleavage fracture initiates in the high stress region near the tip of a blunting crack [1,18–21]. The crack tip fields can be described by isostress contours that reach peak values of 3–5 times σ_y, depending on the alloy strain-hardening rate [1,12,18–20]. Under plane strain, small-scale yielding (SSY) conditions for specimens with $a/W \approx 0.5$, the spatial dimensions of the stress field scale with the crack tip opening displacement, $\delta \approx K_J^2/2\sigma_y E$ [1,18,20]. However, if the deformation level, hence the δ, that is required to produce cleavage is not very small compared to the characteristic dimension of the specimen, typically taken as the uncracked ligament length, $b = W - a$, then the crack tip stress fields fall below small scale yielding values. The reduction of the stress fields at higher δ/b is known as constraint loss (CL) [1,8,12]. Constraint loss begins at $\delta/b \approx 0.01$ and becomes significant at values of 0.02 [1,8]. Assuming an irradiated $\sigma_y \approx 600$ MPa and $\delta/b = 0.02$ suggests that the maximum toughness that can be measured without significant constraint loss is about 65 MPa $\sqrt{m}$ for the 1/3PCC specimens. Beyond this limiting value, the K_{Jm}/K_{Jc} increase rapidly. Thus rapid CL with increasing toughness and T_t, is marked by a very steep slope of the $K_{Jm}(T_t)$ curves for small specimens in the cleavage transition.

A simple but powerful micromechanical model proposes that cleavage occurs when a critical stress ($\sigma*$) encompasses a critical volume V^* of material near a crack tip [1,8,12]. Three-dimensional finite element (FE) simulations were performed to obtain the average stressed areas ($\langle A \rangle$) along the crack front as a function of the alloys constitutive law, $\sigma(\varepsilon)$, applied loading K_J, and normalized stress σ_{22}/σ_y perpendicular to the crack plane, for both large scale yielding (LSY) conditions with the actual specimen geometry and the SSY condition [1,4,8]. The CL size adjustment is defined as the ratio, $[K_J/K_{ssy}]$, of the LSY-K_J at K_{Jm} to the corresponding SSY K_{ssy} at K_{Jc} for the same stressed $\langle A \rangle$ at a specified $\sigma*/\sigma_y$, where σ^* is the critical microcleavage fracture stress [1,8,19]. Thus, $K_{Jc}(B) = K_{Jm}/[K_J/K_{ssy}]$. A second statistical stressed volume (SSV) size adjustment relates to the variations in the probability of initiating weakest link cleavage as a function of the total volume (V) of material under high stress. Since for SSY conditions, $\langle A \rangle$ scales with K_J^4 and $V^* = B\langle A \rangle^*$, simple theory suggests that K_{Jc} scales as $\approx B^{-1/4}$ [1,8]. However, other mechanistic considerations and empirical observations show that this $B^{-1/4}$-scaling is modified by a minimum toughness, K_{min}, as $K_{Jr}(B_r) = [K_{Jc}(B) - K_{min}][B_r/B]^{1/4} + K_{min}$ [1,5,8]. The ASTM MC standard E1921-05, specifies a $K_{min} = 20$ MPa $\sqrt{m}$ and a reference thickness $B_r = 25.4$ mm [5]. More detailed description on the CL and SSV size effects adjustment procedures can be found in the literature [1,8].

3. Results and discussion

The $K_{Jm}(T)$ data for the 1/3PCC specimens irradiated in JMTR to 0.02 and 0.12 dpa at 290 °C are shown in Fig. 1(a). The measured shifts, ΔT_m, evaluated by the temperatures marking the sharp toughness transitions, as indicated by the lines, are about 24 ± 10 and 44 ± 10 °C for doses of 0.02 and 0.12 dpa, respectively. Fig. 1(b) shows K_{Jm} data adjusted to the toughness, K_{JB}, at a reference thickness $B_r = 25.4$ m, using the ASTM E1921 statistical adjustment procedure cited above [5]. Unirradiated K_{JB} toughness values for the same heat of F82H reported by Wallin [3] from tests on similar bend specimens with dimensions of $W = 4$, $B = 3$ and $L = 27$ mm, shown in Fig. 1(b) for comparison, and are generally consistent with our unirradiated control K_{JB} data. A multi-temperature MC analysis based on ASTM E1921 procedure [5] yields a $T_0 \approx -126$ °C. The deviation of ≈ -26 °C for the small 1/3PPC specimens relative to full constraint conditions is consistent with previous observations [2–4]. The corresponding adjusted T_0 values of the irradiated specimens are −99 and −76 °C, yielding $\Delta T_0 \approx 27 \pm 8$ and 50 ± 7 °C, for irradiations to 0.02 and 0.12 dpa, respectively. Note, the data in Fig. 1(a) and (b) do not account for CL effects, which are discussed below.

The $[K_J/K_{ssy}]$ CL adjustment factors were determined from FE simulations for $\sigma^* = 2100$ MPa using the $\sigma(\varepsilon)$ from the −100 °C tensile test data on the unirradiated control and the specimens irradiated to 0.12 dpa. The σ^* was calibrated by fitting a MC shape to the SSY $K_{Jc}(T_t)$ curve for F82H [4]. The magnitude of the adjustment, $K_{Jm} - K_{Jc}$, plotted versus K_{Jm} in Fig. 2(a), is similar in both cases. Thus the fitted relation in Fig. 2(a) was used for CL adjustments of the K_{Jm} data obtained from the 1/3PCC specimens irradiated to 0.02 dpa. Fig. 1(c) shows the corresponding CL and SSV adjusted, K_{Jr} data. Multi-temperature MC analyses resulted in T_0 values of −77, −48 and −34 °C, yielding ΔT_0 of 29 ± 8 and 43 ± 7 °C for 0.02 and 0.12 dpa irradiation conditions, respectively. These ΔT_0 are very similar to the other estimates, with overall averages are 27 ± 2 °C and 44 ± 6 °C for the 0.02 and 0.12 dpa irradiations, respectively. However,

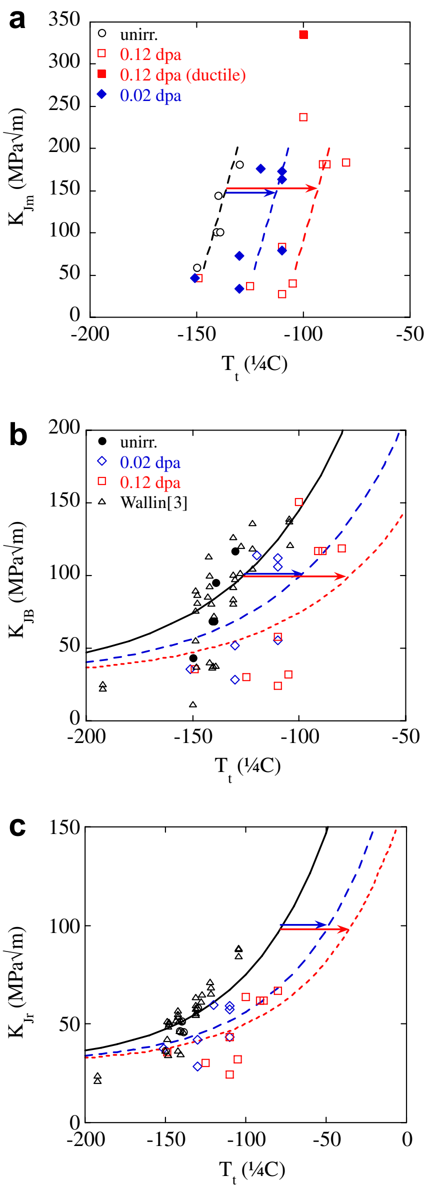

Fig. 1. (a) Measured fracture toughness data, K_{Jm}, on the F82H IEA 1/3PCC specimens before and after the neutron irradiation at 290 °C in JMTR; (b) MC curves derived based on ASTM E1921-05 SSV adjustment procedure; (c) MC curves derived based on the combined CL and SSV adjustment procedure.

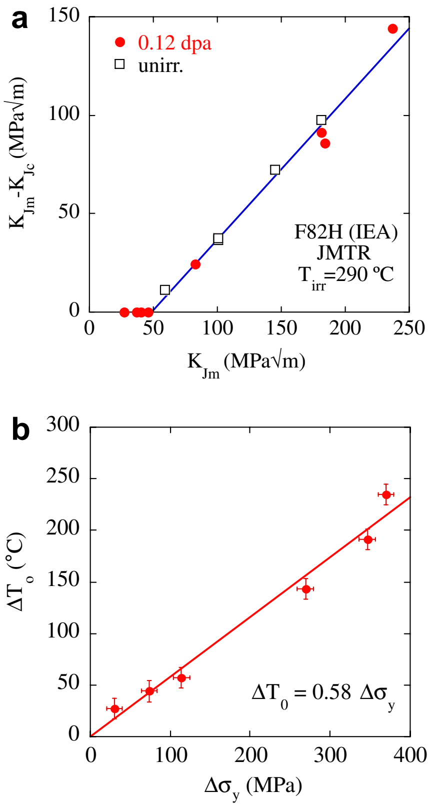

Fig. 2. (a) The magnitude of the CL adjustment as function of K_{Jm} for both the unirradiated control and the 0.12 dpa irradiation condition; (b) ΔT_0 versus $\Delta\sigma_y$ for data available on F82H irradiated between 250 and 380 °C.

the estimated T_0 for the unirradiated F82H is ≈ 23 °C higher than the nominal full constraint K_{Jr} value of ≈ -100 °C. This difference suggests that the combined CL and SSV procedures consistently over adjust the K_{Jm} data for these very small specimens.

Tensile tests performed at 15 °C showed $\Delta\sigma_y = 74 \pm 10$ MPa at 0.12 dpa. Using this and the average $\Delta T_0 = 44 \pm 10$ °C yields a $C_0 = 0.58 \pm 0.14$ °C/MPa at 0.12 dpa, in good agreement with previous estimates for F82H [1,10]. Assuming a $\Delta\sigma_y = K\,(\text{dpa})^{1/2}$ dose scaling, where $K = 213$ MPa for the 0.12 dpa case, the estimated average $\Delta\sigma_y \approx 30 \pm 4$ MPa at 0.02 dpa. Taking the average $\Delta T_0 = 27 \pm 10$ °C, the corresponding $C_0 = 0.9$ °C/MPa. The higher value in this case, may be partly due to a smaller loss of strain hardening for the low dose irradiation condition [12]. Note the uncertainties in the C_0 estimates are large, and are estimated to be $\approx \pm 0.3$ °C/MPa. Indeed, a better demonstration of the consistency of these new results with previous observations is shown in Fig. 2(b) plotting ΔT_0 versus $\Delta\sigma_y$ for available data on F82H [11,21,22].

Fig. 3(a) shows K_{Jd} data from dynamic fracture tests on the DFMB specimens irradiated to 0.1 dpa at 290 °C. The estimated T_{du} for the unirradiated control specimen at an impact velocity 1 m/s impact rate is about -60 °C, while that for 0.2 m/s

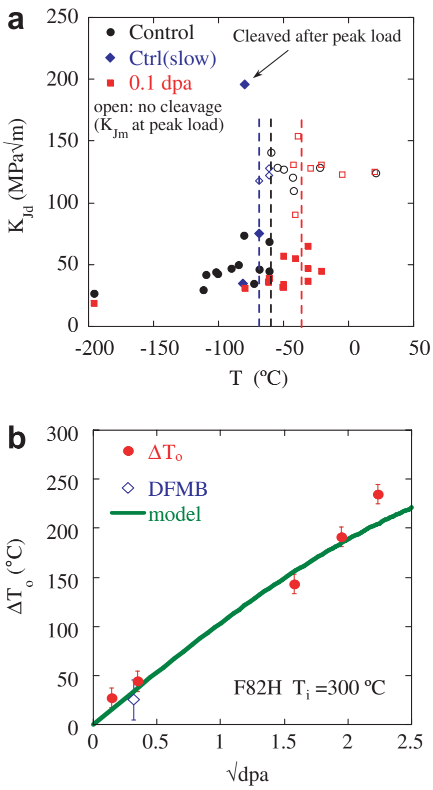

Fig. 3. (a) Dynamic toughness measured by IZOD-type impact tests on F82H IEA DFMB specimens in the unirradiated condition and irradiated to 0.1 dpa at 290 °C in JMTR; (b) the composite model prediction of the ΔT_0 vs dose ($\sqrt{\text{dpa}}$) for $T_i = 300$ °C.

tests is ≈ 10 °C lower. The temperature shift in σ_y due to higher (h) to lower (l) strain-rates given by $\Delta T_{\varepsilon'} = -CT_{ti}\ln(\varepsilon'_h/\varepsilon'_l) \approx -20$ °C, for a nominal value of $C = 0.07$ for the reference strain rate of 50 (s^{-1}), where T_{tl} is the absolute temperature at the lower loading rate [23]. The corresponding T_{di} for the 0.1 dpa irradiation condition is ≈ −35 °C, yielding a $\Delta T_d \approx 25$ °C. Thus ΔT_d is less than the corresponding static ΔT_0. Estimating $\Delta\sigma_{yd} = \Delta\sigma_y = 68$ MPa at 0.1 dpa, the $C_d = \Delta T_d/\Delta\sigma_{yd} = 0.36$ °C/MPa. This low value of C_d is similar to the C_c typically measured in subsized Charpy V-notch impact tests [1].

Assuming a factor of $\approx 5 \times 10^5$ higher loading rate in the dynamic versus static tests the $\Delta T_{\varepsilon'} = -CT_{th}\ln(\varepsilon'_h/\varepsilon'_l)$ relation, with $C = 0.035$ for the reference strain rate of 10^{-4} (s^{-1}), can be used to estimate a T_m for static DFMB tests as ≈ −158 °C. The corresponding T_0 for the DFMB tests evaluated by the ASTM E1921 procedure would be expected to be about 42 °C less than for the 1/3PCC, as expected due to the additional loss of constraint in this case.

We can also estimate ΔT_0 based on the hardening-shift relation $\Delta T_0 = C_0\Delta\sigma_y(\text{dpa}, T_i)$, where the $\Delta\sigma_y(\text{dpa}, T_i)$ was derived from our analysis of the larger TMS database study. For $T_i = 300$ °C and using the $\sigma_y(\text{dpa}, T_i)$ correlation for $T_t = 23$ °C and the $C_0 = 0.58$ °C/MPa from the fit shown in Fig. 2(b)

$$\Delta T_0 = 296[1 - \exp(-\text{dpa}/7.7)]^{1/2}. \quad (1)$$

As shown in Fig. 3(b), the hardening-shift model ΔT_0 (dpa) predictions are in good agreement with experimental data for $T_i = 290$–300 °C (note, $\Delta T_0(290) - \Delta T_0(300) \approx 3$ °C at around 0.1 dpa, which is within the experimental error). Fig. 3(b) also shows that the ΔT_0 prediction is within the estimated errors for the ΔT_d from the DFMB tests.

4. Closing remarks

Fracture toughness tests for 1/3PCC specimens of F82H IEA irradiated to 0.02 and 0.12 dpa at 290 °C in JMTR were carried out to estimate the MC reference temperature shift, ΔT_0, using a variety of procedures. These tests yielded average values of 27 ± 10 and 44 ± 10 °C, respectively. These results indicate that ΔT_m directly evaluated from the K_{Jm} data can be used to estimate $\Delta T_0 \approx \Delta T_m$, in spite of the large CL suffered by these small specimens. As expected, both the unirradiated T_d and that for irradiations at 290 °C to 0.1 dpa from the dynamic DFMB tests were higher than their static counterparts. The nominal $\Delta T_d = 25 \pm 20$ °C determined from dynamic tests is somewhat smaller than the estimated ΔT_0 for the same irradiation conditions of ≈ 40 °C, but the difference is within the estimated data uncertainties. Likewise, the individual values of C_0 and C_d for these small specimen tests varied, but averaged about 0.58 °C/MPa, consistent with previous observations. Indeed ΔT_0 and ΔT_d predicted by a simple hardening shift model based on this C_0 and $\Delta\sigma_y$ (dpa, T_i) derived from an analysis of a large database is also consistent with experimental estimates within expected data errors.

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