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Application of the master curve to inhomogeneous ferritic/martensitic steel

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Abstract

Three sizes of fracture toughness specimens of F82H steel were tested to verify the master curve concept. Specimens were tested at several temperatures in the transition region with at least four tests at each temperature to allow application of the Weibull statistic/master curve analysis procedure. The largest specimens were 1 T C(T) compact specimens. Broken halves of 1 T C(T) specimens were later used to machine and test smaller, 0.4 T C(T) and 0.18 T DC(T), specimens more suitable for irradiation experiments. The scatter of fracture toughness was rather high relative to scatter predicted by conventional master curve concept, but was similar for larger and smaller specimens. It was assumed that this material exhibited inhomogeneity of fracture toughness. Random inhomogeneity analysis provides a very good description of the scatter of fracture toughness data of F82H steel. At the same time, values of T_0 derived using conventional and random inhomogeneity analyses are similar. TEM and SEM analysis helped identify microstructural features that might be responsible for such behavior.

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1. Introduction

Recent advances in fracture toughness have led to employment of Weibull statistics to model scatter of fracture toughness in the transition region of low-alloyed reactor pressure vessel steels. This methodology, proposed by Wallin [1,2], uses a concept of the universal temperature dependence of fracture toughness in the transition region, the so-called ‘master curve’. The current physical background for this methodology suggests that it is applicable to a wide variety of ferritic bcc steels, including tempered ferritic–martensitic steels. These steels are structural material candidates for fusion reactors, yet the transition fracture toughness data for this class of steels are rather sparse.

In this study, three sizes of fracture toughness specimens of F82H steel were tested to verify the master curve concept. Specimens were tested at several temperatures in the transition region and at least four specimens were tested at each temperature allowing for application of the Weibull statistic/master curve analysis procedure. The largest specimens were 1 T C(T) compact specimens. Broken halves of 1 T C(T) specimens were later used to machine and test smaller, 0.4 T C(T) and 0.18 T DC(T) size specimens which could be more suitable for irradiation experiments.

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The reduced-activation ferritic–martensitic (RAFM) 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 Agency (DOE–JAEA) collaboration program. The F82H alloy (Fe–8Cr–2W–V–Ta) was developed by JAEA and NKK Corporation, Kawasaki, Japan and provided to participants in the International Energy Agency (IEA) round-robin tests. Material used for the IEA round-robin tests was melted in two 5 metric-ton heats (heat #9741 and #9753). The 7.5-mm and 15-mm thick plates were produced from heat #9741 and the 15 mm and 25 mm thick plates were produced from heat #9753. All 1 T C(T) fracture toughness specimens were taken from 25-mm plates from heat No.9753. This size plate was distributed in two conditions: one was standard heat treatment, and the other was TIG-welded or EB-welded followed by post weld heat treatment (PWHT). In this study, the fracture toughness of 25-mm-thick plate from heat #9753 that underwent PWHT after TIG welding was characterized in the transition region. All specimens were machined in the L–T orientation such that the crack would propagate in the transverse orientation.

2. Testing and analysis procedures

The fracture toughness tests were conducted in accordance with the ASTM E 1921-05 standard test method for determination of reference temperature, T_0, for ferritic steels in the transition range. The specimens were fatigue precracked to a ratio of the crack length to specimen width (a/W) of about 0.5. The unloading compliance method was used for measuring crack growth. Specimens were tested in the laboratory on a 98-kN (22-kip) capacity servohydraulic machine. An outboard clip gage was used to measure load-line displacement. The broken specimens were examined with a calibrated measuring optical microscope to determine the initial and final crack lengths.

The following is a brief description of the master curve methodology. More details can be found in Ref. [3], for example. Values of J-integral at cleavage instability, J_c, were converted to their equivalent values in terms of stress intensity factor K_{J_c} by the following equation:

$$K_{J_c} = \sqrt{J_c \frac{E}{1-\nu^2}}, \tag{1}$$

where E is Young's modulus and $\nu = 0.3$ is Poisson's ratio. The K_{J_c} value was considered invalid if it exceeded the validity limit:

$$K_{J_c(\text{limit})} = \sqrt{\frac{Eb_0\sigma_{\text{YS}}}{30(1-\nu^2)}}, \tag{2}$$

where b_0 is the in-plane size of the remaining ligament of the specimen and σ_{ys} is yield strength of the material. If the measured value exceeds the validity limit, it is considered an invalid value and replaced (censored) with the K_{J_c} value for T_0 calculation. All K_{J_c} data were converted to 1 T equivalence, K_{J_c} (1 T), using the weakest-link size adjustment procedure of E1921:

$$K_{J_c(1\text{T})} = 20 + \left[K_{J_c(xT)} - 20\right] \cdot \left(\frac{B_{xT}}{B_{1T}}\right)^{1/4}, \tag{3}$$

The distribution of fracture toughness values is described by the three-parameter Weibull cumulative probability function:

$$P_{\text{f}} = 1 - \exp\left[-\left(\frac{K_{J_c} - 20}{K_0 - 20}\right)^4\right], \tag{4}$$

where two parameters are fixed to 4 and 20 MPa$\sqrt{\text{m}}$, respectfully. Thus, only the scale parameter, K_0, needs to be determined. K_0 is determined using the maximum likelihood function L. The censored likelihood function, L, is the product of the probability density function and survival function. The probability density function, f, for the master curve distribution function is given by:

$$f(K_{J_c}) = \frac{\text{d}P}{\text{d}K_{J_c}} = \frac{4(K_{J_c} - 20)^{4-1}}{(K_0 - 20)^4}\exp\left[-\left(\frac{K_{J_c} - 20}{K_0 - 20}\right)^4\right]. \tag{5}$$

The master curve survival function is given by:

$$S(K_{J_c}) = \exp\left[-\left(\frac{K_{J_c} - 20}{K_0 - 20}\right)^4\right]. \tag{6}$$

Thus, the censored likelihood function, L, can be expressed as:

$$\begin{aligned} L &= \prod\nolimits_{i=1}^{N} f_i^{\delta_i} \times S_i^{1-\delta_i} \\ &= \prod\nolimits_{i=1}^{N} \frac{4(K_{J_c(i)} - 20)^{3\delta_i}}{(K_0 - 20)^{4\delta_i}} \cdot \exp\left(-\left\{\frac{K_{J_c(i)} - 20}{K_0 - 20}\right\}^4\right), \end{aligned} \tag{7}$$

where $\delta_i = 1.0$ if the $K_{J_c(i)}$ datum is valid or zero if datum is invalid and censored. The master curve temperature dependence is described as:

$$K_{J_c(\mathrm{med})} = 20 + (K_0 - 20)(\ln 2)^{1/4} = 30 + 70\exp[0.019(T - T_0)], \tag{8}$$

where T_0 is the reference fracture toughness transition temperature that corresponds to the temperature at which $K_{J_c(\mathrm{med})} = 100\ \mathrm{MPa}\sqrt{\mathrm{m}}$. The T_0 is determined using the multi-temperature equation from E1921 by inserting the master curve dependence, Eq. (8), into Eq. (7) and solving it for $\partial\ln(\mathrm{L})/\partial T_0 = 0$:

$$\sum_{i=1}^{N}\delta_i\frac{\exp[0.019(T_i - T_0)]}{11 + 77\exp[0.019(T_i - T_0)]} - \sum_{i=1}^{N}\frac{(K_{J_c(i)} - 20)^4\exp[0.019(T_i - T_0)]}{\{11 + 77\exp[0.019(T_i - T_0)]\}^5} = 0, \tag{9}$$

where T_i = test temperature corresponding to $K_{J_c(i)}$ value.

3. Results and discussion

A total of 53 specimens of F82H steel have been tested in the transition region, 27 1 T C(T), 19 0.4 T C(T) and 7 0.18 T DC(T). One 0.4 T compact specimen tested at −20 °C did not cleave as that test was stopped when the clip gage ran out of measuring range. Final stress intensity factor converted from the J-integral value at the end of the test was higher than the $K_{J_c(\mathrm{limit})}$ value; thus it was treated as an invalid specimen. The tabulated data are available in Ref. [4]. The reference fracture toughness transition temperature, T_0, for this data set is determined to be −105 °C and standard deviation $\sigma = 3.4$. Fig. 1 illustrates the 1 T size-adjusted fracture toughness data vs test temperature, with the master curve and the 5% and 95% tolerance bounds from this analysis added. The first observation of these data is that this material exhibited a very high scatter of fracture toughness data in the transition region. Sixteen out of 53 fracture toughness values or 30% are outside 5% and 95% tolerance bounds. For example, at −20 °C measured fracture toughness values of F82H varied from 84 to 497 $\mathrm{MPa}\sqrt{\mathrm{m}}$. At same time, the scatter of fracture toughness within data sets for larger or smaller specimens appears to be similar. This indicates a potential for inhomogeneity of fracture toughness properties of this F82H plate. Scanning electron-microscopy (SEM) has been performed to examine the fractured surfaces of the broken specimens. All specimens failed by cleavage; no evidence of intergranular fracture was observed on the fractured surfaces.

Gelles and Sokolov performed metallographic and SEM examination of broken specimens from this study [5,6]. Among other observations,

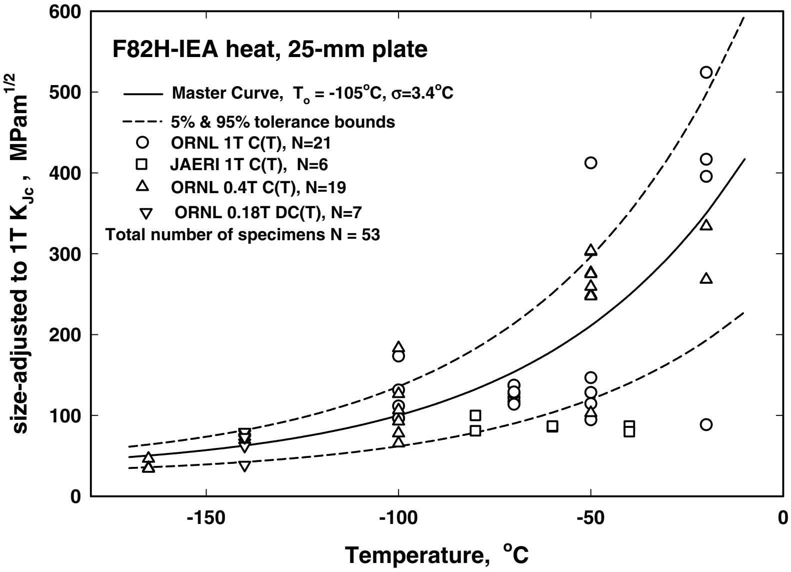

Fig. 1. Scatter of F82H-IAE fracture toughness data relative to the conventional master curve and 5% & 95% tolerance bounds.

metallographic carbide etchant revealed larger particles dispersed through the thickness of the plate. The particles were found to be rich in Ta and O. Nevertheless, size distribution measurements did not indicate any inhomogeneity in distribution through the thickness of the plate. However, in the course of examination [6], it became apparent from the spatial distribution that the particles tended to clump, but clumping was generally restricted to the center of the plate.

Tanigawa et al. [7] investigated inclusions formed in the plates of F82H steel by SEM and transmission electron-microscopy (TEM) equipped with EDS. Analyses by SEM and TEM for the plates revealed that Ta does not form MX precipitates, but instead, it forms composite Al_2O_3–Ta(V,Ti)O oxide, or single phase Ta(V)O oxide. The composite inclusions are rather dominant in the plate obtained from the bottom of the ingot, but not in the plate from the middle of the ingot. SEM observations by Tanigawa et al. [7] of broken specimens from this study also revealed that composite oxide tended to be observed at the crack-initiation site of broken specimens.

Despite some extensive microstructural investigations [5–7] of the F82H steel, including broken specimens from this study, it remains difficult to draw a strong link between distribution of these composite oxides and the inhomogeneity of fracture toughness.

4. Random inhomogeneity analysis of fracture toughness data

The large scatter in fracture toughness data required a reconsideration of the application of the conventional master curve approach as in Eqs. (4)–(9) and consideration of the idea of treating these data as an inhomogeneous dataset [8]. This means that the value of T_0 becomes a random variable in the inhomogeneous dataset. Then, the probability density function for T_0 is:

$$f_{\mathrm{T}} = \frac{1}{2\sqrt{\pi}\cdot\sigma_{T_{0\mathrm{RI}}}}\exp\left[-\frac{(T_0 - T_{0\mathrm{RI}})^2}{2\sigma^2_{T_{0\mathrm{RI}}}}\right], \tag{10}$$

where $T_{0\mathrm{RI}}$ and σ_{ToRI} are an estimate of the fracture toughness transition temperature from the random inhomogeneity analysis and its standard deviation, respectively. The local conditional density and survival probabilities at T_0, f_{T0} and S_{T0}, are the same as in conventional master curve methodology, Eqs. (5) and (6). Then, the total density and survival probabilities are:

$$f = \int_{-\infty}^{\infty} f_{\mathrm{T}}\cdot f_{T_0}\cdot \mathrm{d}T_0 \quad \text{and} \quad S = \int f_T\cdot S_{T_0}\cdot \mathrm{d}T_0. \tag{11}$$

The parameters $T_{0\mathrm{RI}}$ and σ_{ToRI} are then solved by maximizing:

$$\ln L = \sum_{i=1}^{N}[\delta_i\cdot\ln(f_i) + (1-\delta_i)\cdot\ln(S_i)]. \tag{12}$$

Application of this random inhomogeneity analysis yielded $T_{0\mathrm{RI}} = -93$ °C and $\sigma_{\mathrm{ToRI}} = 26.3$ °C. Note that the absolute value from this analysis is similar to the T_0 estimate of −105 °C from the conventional master curve analysis. The main difference comes in the standard deviation values. The standard deviation from the random inhomogeneity analysis provides a more realistic scatter band for these data. Five out of 53 fracture toughness values or 9.5% are outside 5% and 95% tolerance bounds compared to 16 data points (30%) in the case of the conventional master curve analysis. Fig. 2 illustrates the master curves (50%) and 5% and 95% tolerance bounds from the random inhomogeneity and the conventional analyses.

In addition to the present data, Odette et al. assembled a large database of fracture toughness of F82H from different sources [9] including most of data from this study. All data were constraint adjusted by a procedure developed at UCSB. There were a total of 219 data points in the UCSB database. The conventional master curve T_0 value for this large constraint-adjusted dataset of F82H was determined to be −103 °C [9]. This corresponds well with T_0 value (−105 °C) from this study. As in the case of the present study, there was a large number of data points (55 out of 219, 24%) outside 5% and 95% tolerance bounds, see Fig. 3. This provides another argument in favor of the inhomogeneity of fracture toughness of F82H steel. On the other hand, there were only 16 data points (or 7.3%) outside 5% and 95% tolerance bounds derived in this study by the random inhomogeneity analysis.

Clearly, the scatter in the transition fracture toughness of F82H-IEA steel is somewhat higher than expected by the conventional master curve analysis. The random inhomogeneity analysis provides a better description of the same scatter. Notably, the T_0 values derived by both analyses are similar, the main difference comes in values of

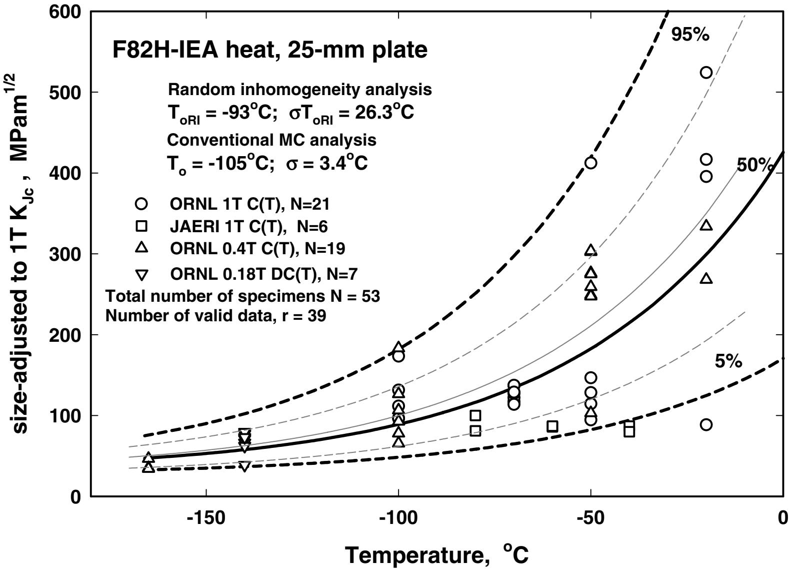

Fig. 2. Comparison of random inhomogeneity analysis (heavy lines) and conventional master curve analysis (light lines) of F82H data.

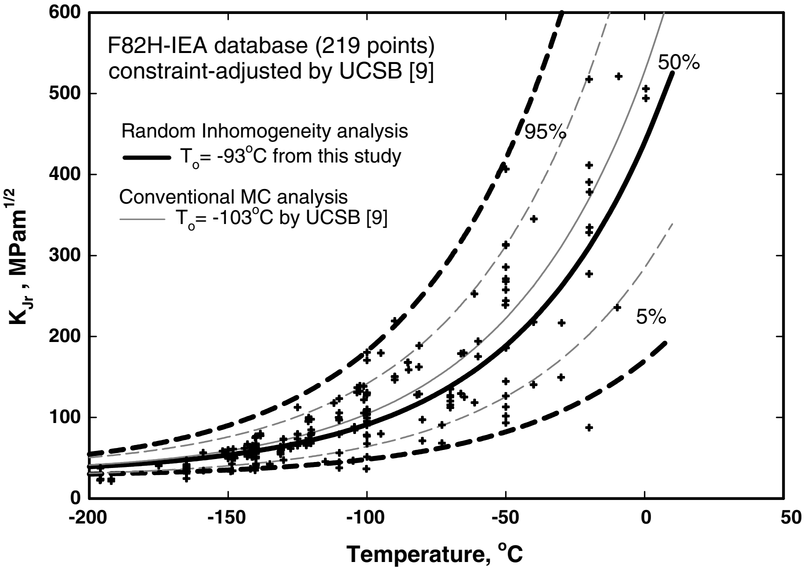

Fig. 3. Application of random inhomogeneity analysis from the present study to a large constraint-adjusted UCSB database [9] of fracture toughness data on F82H-IEA. Solid lines are the master curves (50%), dashed lines are 5% and 95% tolerance bounds. Heavy lines are the random inhomogeneity analysis; the light lines are the conventional master curve analysis.

the standard deviation. This has an important practical application for use of the small specimens for post-irradiation characterization of this steel. The random inhomogeneity analysis requires a relatively large number of data points. At the same time, only a small number of small size specimens can be irradiated in test reactors. The result is that researchers are forced to determine the irradiated T_0 values for candidate fusion materials using only few specimens. It appears from the analysis of data in this study that this practice provides a reasonable estimate of irradiated T_0 values, but it is not able to address the issue of proper description of the scatter in the transition fracture toughness.

5. Summary

The results of this study showed that the scatter of fracture toughness for a 25 mm plate of F82H-IEA was larger than anticipated by the conventional master curve analysis. The random inhomogeneity analysis provides a better description of the data scatter for F82H steel, than does the conventional master curve analysis. At the same time, the T_0 values derived using the conventional and random inhomogeneity analyses are quite similar.

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