APPLICATION OF THE MASTER CURVE TO INHOMOGENEOUS FERRITIC/MARTENSITIC STEEL—M. A. Sokolov (Oak Ridge National Laboratory) and H. Tanigawa (Japan Atomic Energy Agency)

OBJECTIVE

In this study, extensive fracture toughness characterization of the IAE heat of F82H steel was performed using 1T C(T), 0.4T C(T), and 0.18T C(T). The objective of this study was to examine the application of the master curve methodology to the transition fracture toughness of this ferritic/martensitic steel.

SUMMARY

A total of 53 specimens of F82H steel have been tested in the transition region, 27 1T C(T), 19 0.4T C(T) and 7 0.18T DC(T). The results of this study showed that the scatter of fracture toughness for a 25 mm plate of F82H-IEA steel was larger than anticipated by the conventional master curve analysis. Several other types of analysis that are the advanced extensions to the conventional master curve method were applied to theses data. It was shown that the random inhomogeneity analysis provides a better description of the data scatter for F82H steel, than does the conventional master curve analysis or any other analyses. At the same time, the T_o values derived using the conventional and random inhomogeneity analyses are quite similar.

PROGRESS AND STATUS

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 K. 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 1T C(T) compact specimens. Broken halves of 1T C(T) specimens were later used to machine and test smaller, 0.4T C(T) and 0.18T DC(T) size specimens which could be more suitable for irradiation experiments.

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). All fracture toughness specimens were taken from 25-mm plates 31W-29 and 31W-3 from heat #9753. These plates were EB- and TIG-welded, respectfully, followed by post weld heat treatment. All specimens were machined in the L-T orientation such that the crack would propagate in the transverse orientation.

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_o, 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_{Jc} by the following equation:

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

where E is Young's modulus and v=0.3 is Poisson's ratio.
The master curve methodology imposes two validity criteria to qualify K_{Jc} data. A K_{Jc} datum would be considered invalid if this value exceeded the $K_{Jc(limit)}$ requirement of E 1921:

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

where b_o 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 $K_{Jc\ (limit)}$ value for T_o calculation. A noticeable number of specimens (14 out of 53) in this study violated this requirement. The second validity requirement limits the amount of stable crack growth prior to cleavage instability. A K_{Jc} datum was considered invalid if the test terminated in cleavage after more than $0.05b_o$ or 1 mm, whichever is smaller, of slow-stable crack growth. Several specimens in this study violated this requirement. The same specimens violated the requirement by Eq. 2 and were censored by the procedure described above for T_o calculation.

All K_{Jc} data were converted to 1T equivalence, $K_{Jc(1T)}$, using the weakest-link size adjustment procedure of E1921:

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

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

$$P_f = 1 - \exp\left[-\left(\frac{K_{Jc} - 20}{K_o - 20}\right)^4\right] \tag{4}$$

where two parameters are fixed to 4 and 20 MPa√m, respectfully. Thus, only the scale parameter, K_o, needs to be determined. K_o is determined using the maximum likelihood function

L. The censored likelihood function, *L*, is the product of the probability density function and survival function [3]. The probability density function, *f*, for the master curve distribution function is given by:

$$f\left(K_{Jc}\right)=\frac{dP}{dK_{Jc}}=\frac{4\left(K_{Jc}-20\right)^{4-1}}{\left(K_o-20\right)^4}\exp\left[-\left(\frac{K_{Jc}-20}{K_o-20}\right)^4\right] \tag{5}$$

The master curve survival function is given by:

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

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

$$L=\prod_{i=1}^{N} f_i^{\delta_i}\times S_i^{1-\delta_i}=\prod_{i=1}^{N}\frac{4\left(K_{Jc(i)}-20\right)^{3\delta_i}}{\left(K_o-20\right)^{4\delta_i}}\cdot\exp\left(-\left\{\frac{K_{Jc(i)}-20}{K_o-20}\right\}^4\right) \tag{7}$$

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

$$K_{Jc(med)}=20+\left(K_o-20\right)\left(\ln 2\right)^{1/4}=30+70\exp\left[0.019\left(T-T_o\right)\right] \tag{8}$$

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

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

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

It is firmly stated in the E1921 standard that this methodology should be applied to the transition fracture toughness data as close as possible to K_{Jc} level of 100MPa√m. Moreover, the E1921 requires that only fracture toughness data within -50°C≤ T_o ≤50°C should be used for final T_o calculation using Eq. 9.

Results and Discussion

A total of 53 specimens of F82H steel have been tested in the transition region, 27 1T C(T), 19 0.4T C(T) and 7 0.18T DC(T). One 0.4T 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 $K_{Jc\ (limit)}$ value; thus it was treated as an invalid specimen. All data are summarized in Table 1. The reference fracture toughness transition temperature, T_o, for this data set is determined to be -105°C and standard deviation σ=3.4°C. Figure 1 illustrates the 1T 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 MPa√m. At same time, the scatter of fracture toughness within data sets for larger or smaller specimens appears to be similar. It indicates that size adjustment procedure by Eq. 3 works well for these data.

Since there were replicate tests performed at several temperatures, it was decided to determine median fracture toughness values at each of these temperatures and than compare how well those median values follow the master curve, Fig. 2. On this plot, the solid line is the master curve and each data point represents the median fracture toughness at the given temperature. Number in parentheses is the ratio of total number of tested specimens to the number of valid K_{Jc}.

Table 1. Fracture toughness data of the IAE heat of F82H steel

I.D.	Size	Test T	σ_y	K_{Jc}	K_{Jc}^{1T}	$K_{Jc\ (limit)}$	δ_i
		°C	MPa	MPa√m	MPa√m	MPa√m	
M10	1T	-20	537	88.6	88.6	311	1
5_10	1T	-20	537	416.7	416.7	311	0
M7	1T	-20	537	395.6	395.6	311	0
5_12	1T	-20	537	524.2	524.2	311	0
M8	1T	-50	550	128.4	128.4	316	1
5_5	1T	-50	550	114.6	114.6	316	1
5_4	1T	-50	550	146.7	146.7	316	1
5_7	1T	-50	550	94.6	94.6	316	1
5_8	1T	-50	550	412.4	412.4	316	0
M2	1T	-70	560	136.9	136.9	320	1
M5	1T	-70	560	117.1	117.1	320	1
5_6	1T	-70	560	122.1	122.1	320	1
M6	1T	-70	560	137.4	137.4	320	1
M3	1T	-70	560	127.3	127.3	320	1
M4	1T	-70	560	129.0	129.0	320	1
M1	1T	-70	560	113.8	113.8	320	1
5_11	1T	-100	575	111.7	111.7	326	1
5_1	1T	-100	575	96.6	96.6	326	1
5_9	1T	-100	575	173.4	173.4	326	1
5_2	1T	-100	575	96.4	96.4	326	1
5_3	1T	-100	575	131.5	131.5	326	1
3-16	1T	-40	546	87.3	87.3	314	1
3-13	1T	-40	546	80.2	80.2	314	1
3-17	1T	-60	555	86.2	86.2	318	1
3-14	1T	-60	555	87.2	87.2	318	1
3-18	1T	-80	564	99.7	99.7	322	1
3-15	1T	-80	564	81.1	81.1	322	1
52-1	0.4T	-50	550	124.5	103.2	209	1
52-2	0.4T	-50	550	322.9	261.1	204	0
m71	0.4T	-20	537	347.7	280.8	204	0
m72	0.4T	-20	537	414.6	333.9	208	0
56-3	0.4T	-100	575	81.0	68.6	214	1
56-2	0.4T	-100	575	161.3	132.5	211	1
58-4	0.4T	-100	575	92.9	78.0	218	1
58-3	0.4T	-100	575	135.1	111.5	211	1
58-2	0.4T	-100	575	116.8	96.9	217	1
58-1	0.4T	-100	575	225.5	183.4	217	0
56-1	0.4T	-50	550	306.0	247.5	207	0

Table 1. Fracture toughness data of the IAE heat of F82H steel (continued)

I.D.	Size	Test T	σ_y	K_{Jc}	K_{Jc}^{1T}	$K_{Jc\ (limit)}$	δ_i
		^oC	MPa	MPa√m	MPa√m	MPa√m	
56-4	0.4T	-50	550	359.1	289.6	212	0
59-1	0.4T	-50	550	393.0	316.9	206	0
59-2	0.4T	-50	550	340.6	275.0	204	0
59-4	0.4T	-50	550	394.4	318.0	207	0
59-3	0.4T	-50	550	335.9	271.6	209	0
510-1	0.4T	-165	612	56.1	48.7	211	1
510-2	0.4T	-165	612	40.9	36.7	225	1
510-3	0.4T	-165	612	37.9	34.3	220	1
HM1	0.18T	-140	597	107.6	77.2	153	1
HM2	0.18T	-140	597	111.1	79.5	155	1
HM3	0.18T	-140	597	91.0	66.3	155	1
HM5	0.18T	-140	597	98.1	71.0	153	1
HM6	0.18T	-140	597	84.7	62.2	149	1
HM8	0.18T	-140	597	48.7	38.8	154	1
HM9	0.18T	-140	597	101.2	73.0	145	1

values at the given temperature. Remarkably, the median fracture toughness values are following the master curve trend very well within the wide temperature range studied.

Larger than expected scatter 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.

D. Gelles performed metallographic and SEM examination of broken specimens from this study [4,5]. Among other observations, 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 [5], it became apparent from the spatial distribution that the particles tended to clump, but clumping was generally restricted to the center of the plate.

H. Tanigawa et al. [6] 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. [6] 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 [4-6] 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.

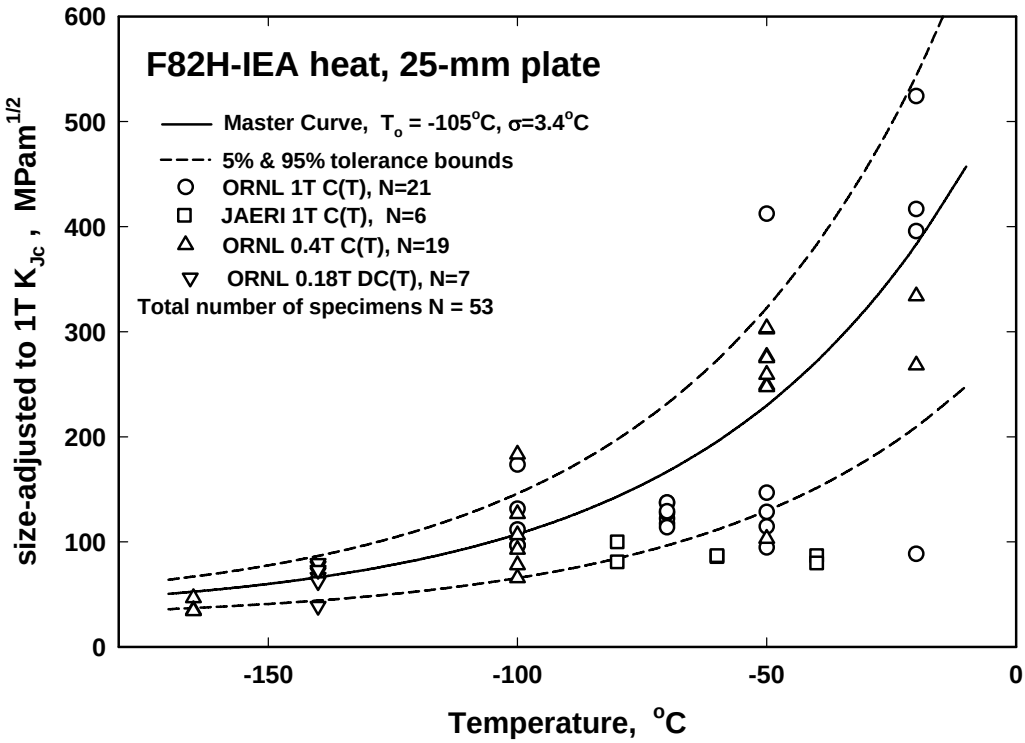


Fig. 1. Scatter of F82H-IAE fracture toughness data relative to the conventional master curve and 5% & 95% tolerance bounds.

Bimodal Master Curve Analyses 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. One way of doing it is to assume that our data consist of two combined master curve distributions. In this case, the total cumulative probability distribution can be expressed as a bimodal distribution of the form [7]:

$$P_f = 1 - p_1 \cdot \exp\left[-\left(\frac{K_{Jc} - 20}{K_{o1} - 20}\right)^4\right] - (1 - p_1) \cdot \exp\left[-\left(\frac{K_{Jc} - 20}{K_{o2} - 20}\right)^4\right] \quad (10)$$

where K_{o1} and K_{o2} are the scale parameters for the two distributions and p_1 is the probability of the toughness belonging to distribution 1. In the case of multi-temperature data set like in the present work, K_{o1} and K_{o2} should be expressed in terms of reference fracture toughness temperatures, T_{o1} and T_{o2}. While the Eq. 10 appears to be a simple modification of Eq. 4, there is a key difference in them. There are three parameters need to be determined in the bimodal distribution function, Eq. 10, compared to only one in the conventional master curve analysis, Eq. 4. As such, a larger number of specimens needs to be tested to perform the bimodal analysis

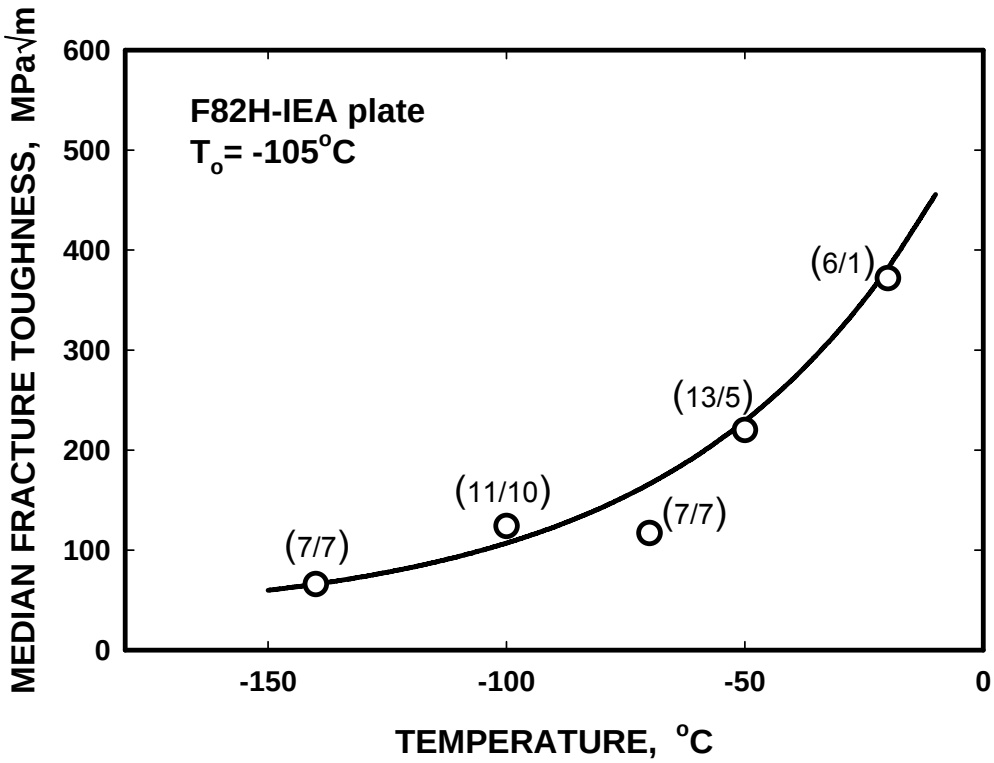


Fig. 2. Median fracture toughness values of F82H plate at different temperatures. The solid line is the master curve. Number in parentheses is the ratio of total number of tested specimens to the number of valid K_{Jc} values at the given temperature.

compared to the conventional master curve analysis. The general form of the likelihood function will remain the same as in Eq. 7, however, the probability density and the survival functions have the following forms in the bimodal analysis [7]:

$$f_c = 4 \cdot p_1 \cdot \frac{(K_{Jc}-20)^3}{(K_{o1}-20)^4} \exp\left[-\left(\frac{K_{Jc}-20}{K_{o1}-20}\right)^4\right] - 4 \cdot (1-p_1) \cdot \frac{(K_{Jc}-20)^3}{(K_{o2}-20)^4} \cdot \exp\left[-\left(\frac{K_{Jc}-20}{K_{o2}-20}\right)^4\right]$$

$$S_c = p_1 \cdot \exp\left[-\left(\frac{K_{Jc}-20}{K_{o1}-20}\right)^4\right] + (1-p_1) \cdot \exp\left[-\left(\frac{K_{Jc}-20}{K_{o2}-20}\right)^4\right] \qquad (11)$$

Application of the bimodal analysis gives the following results for the present data of F82H plate: p_1=0.63, T_{o1} = -80°C, and T_{o2} = -120°C, Fig. 3. The bimodal analysis provides slightly better description of scatter in toughness data that the conventional master curve analysis. This bimodal analysis was originally suggested by Wallin to characterize fracture toughness data of heat-affected zone (HAZ) of welds [7]. In the case of HAZ, any given result may be affected by location of the crack tip in the specimen. There might be a potential for some specimens to have crack tip near base metal rather than HAZ and thus justifying the bimodal distribution of data. Yet, the cause for bimodal distribution of fracture toughness in the F82H plate remains unclear. Figures 4a and 4b are the Weibull plots of F82H plate data at -100°C and -50°C, respectfully. The solid lines correspond to the conventional master curve analysis and the dashed lined correspond to the bimodal analysis. These plots do not provide any additional evidence that the bimodal analysis lines follow the data more closely than the conventional master curve analysis.

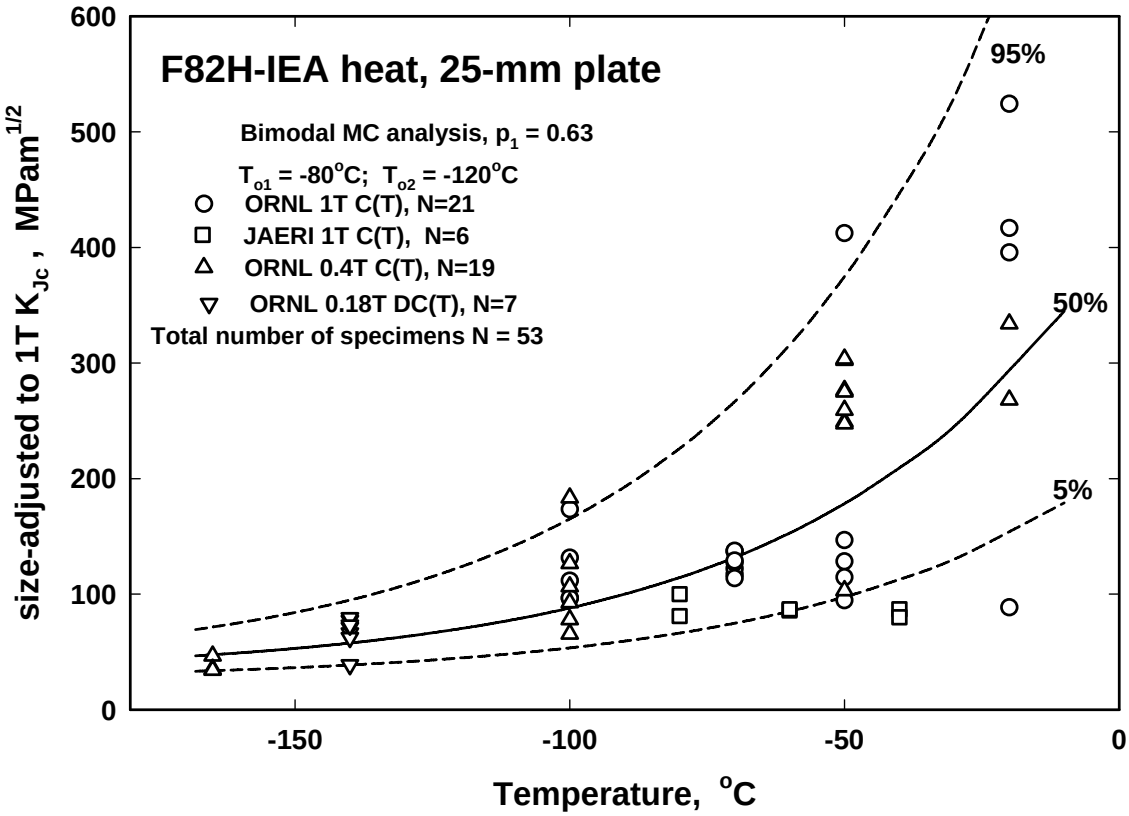
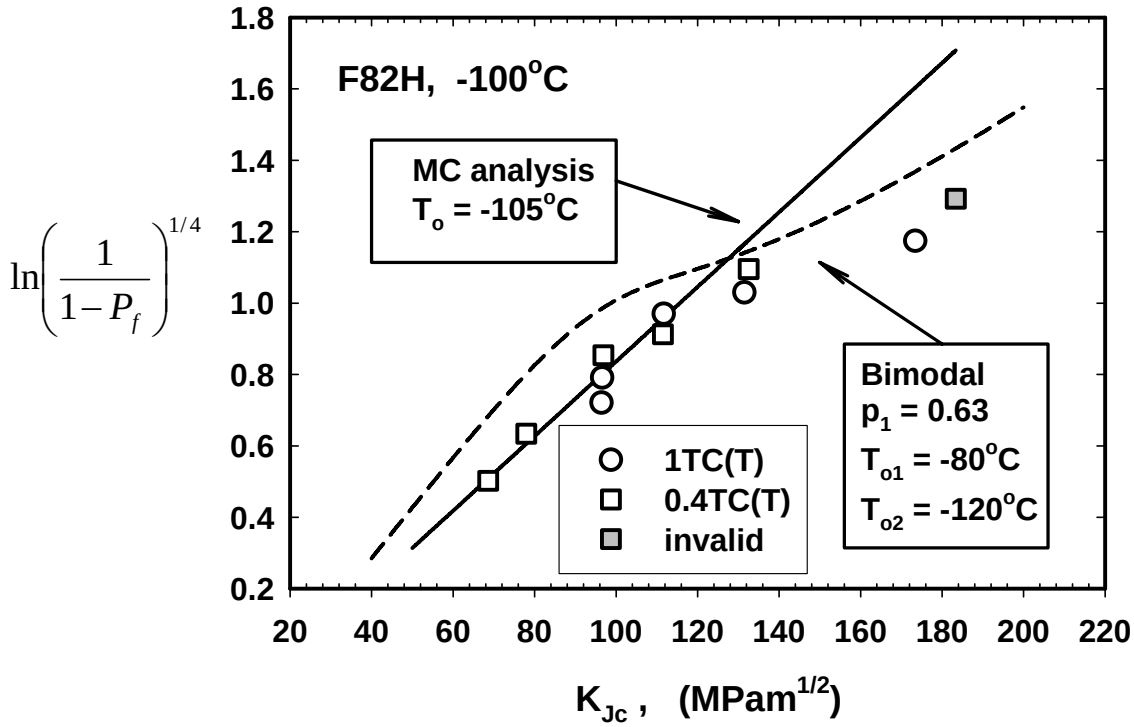


Fig. 3. The bimodal master curve analysis of the F82H plate fracture toughness data.

a)



b)

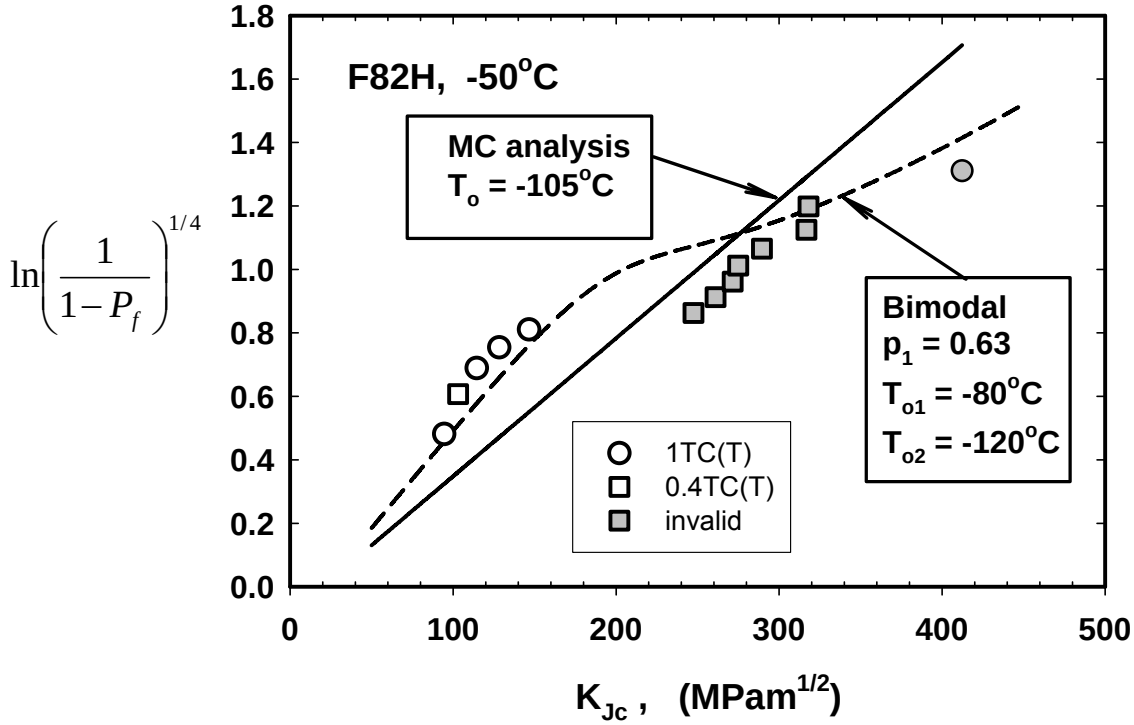


Fig. 4. Comparison of the bimodal and conventional master curve analyses of F82H fracture toughness data at -100°C (a) and -50°C (b).

Random Inhomogeneity Analysis of Fracture Toughness Data

In the previous section, the assumption was made that the present fracture toughness data might consist of two populations. However, the bimodal analysis did not noticeably improve description of the scatter of fracture toughness data. Thus, the next logical step is to assume that we are dealing with an inhomogeneous dataset.

Wallin suggested [7] a simple engineering method, the single point estimation, to examine this assumption. In this method, individual T_o estimates are determined from all non-censored size-adjusted K_{Jc} values:

$$T_{oi} = T_i - \frac{\ln\left(\frac{K_{Jc\,i} - 30}{70}\right)}{0.019} \tag{12}$$

The single point T_o value (T_{osp}) is then estimated as follows [8]:

$$T_{oSP} = \frac{\sum_{i=1}^{r} T_{oi}}{r} - 4\ ^oC \tag{13}$$

where r is the number of non-censored (valid) results as defined above and the standard deviation becomes:

$$\sigma_{T_{o\,SP}} = \sqrt{\frac{\sum_{i=1}^{r} T_{oi}^2 - \frac{\left(\sum_{i=1}^{r} T_{oi}\right)^2}{r}}{r} - \left(21^o C\right)^2} \tag{14}$$

Application of the single point estimate method to the current data set resulted in T_{oSP} = -84°C and σ_{ToSP}=24°C. The T_{oSP} estimate is 21°C higher than the T_o value from the conventional master curve analysis. More importantly, there is a significant difference in the standard deviation estimates. The standard deviation from the single point method is several times more that from the conventional master curve analysis. This is a strong indication that this data set is inhomogeneous. As such, the maximum likelihood method for random inhomogeneity case as suggested by M. Scibetta [8] can be applied to the data analysis. In this method, the value of T_o becomes a random variable in the inhomogeneous dataset. Then, the probability density function for T_o is:

$$f_T = \frac{1}{2\sqrt{\pi} \cdot \sigma_{T_{oRI}}} \exp\left[-\frac{\left(T_o - T_{oRI}\right)^2}{2\sigma_{T_{oRI}}^2}\right] \tag{15}$$

where T_{oRI} 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_o, f_{To} and S_{To}, are the same as in conventional master curve methodology, Eqs. 5 and 6. Then, the total density and survival probabilities are:

$$f = \int_{-\infty}^{\infty} f_T \cdot f_{T_o} \cdot dT_o \quad and \quad S = \int f_T \cdot S_{T_o} \cdot dT_o \tag{16}$$

The parameters T_{oRI} and σ_{ToRI} are then solved by maximizing:

$$\ln L = \sum_{i=1}^{N} \left[\delta_i \cdot \ln(f_i) + (1-\delta_i) \cdot \ln(S_i) \right] \tag{17}$$

Application of this random inhomogeneity analysis yielded T_{oRI}=-93°C and σ_{ToRI}=26.3°C. Note that the absolute value from this analysis is similar to the T_o 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. Figure 5 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, G. 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_o value for this large constraint adjusted dataset of F82H was determined to be -103°C [9]. This corresponds well with T_o 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. 6. 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.

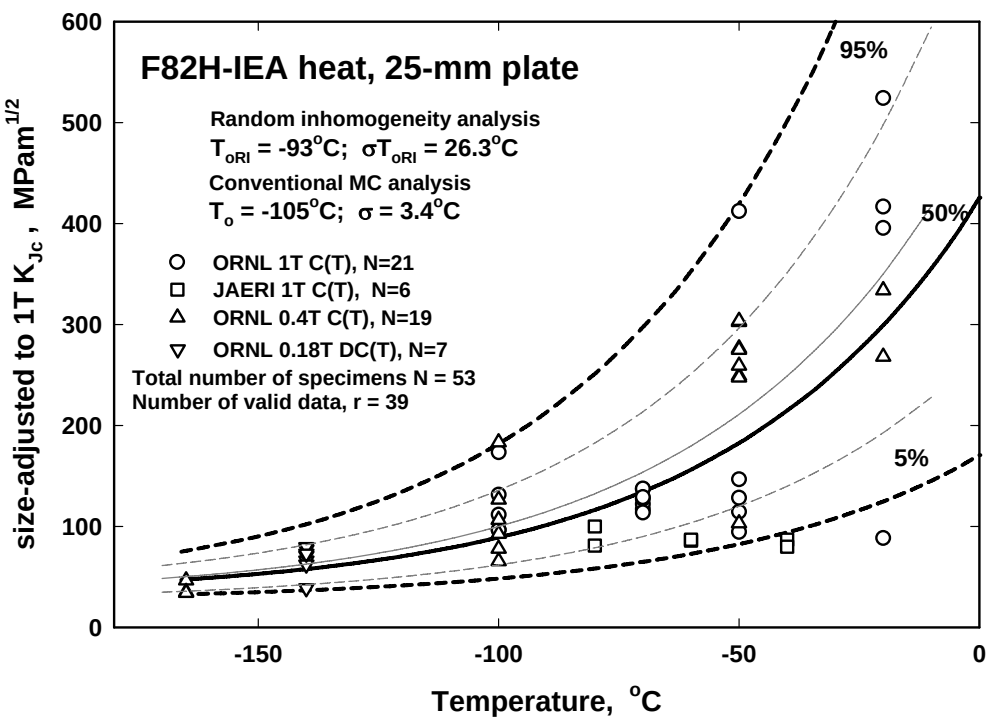


Fig. 5. Comparison of random inhomogeneity analysis (heavy lines) and conventional master curve analysis (light lines) of F82H data.

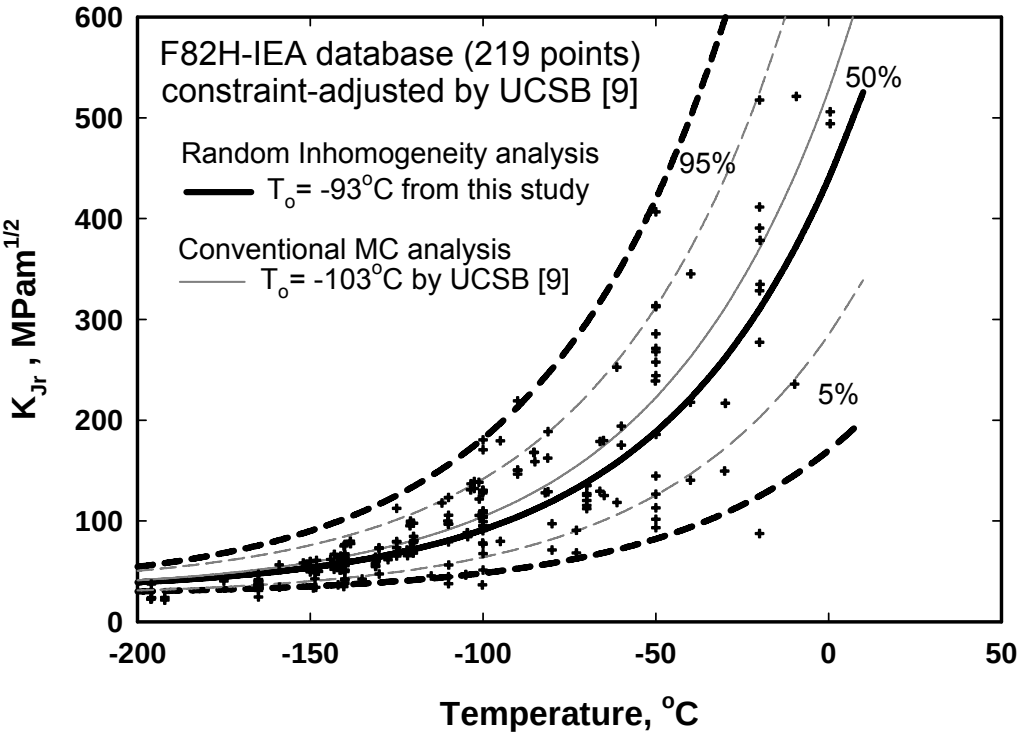


Fig. 6. Application of random inhomogeneity analysis from the present study to a large constraint-adjusted UCSB database [9] of fracture toughness data on F82H. Solid lines are the master curves (50%), dashed lines are 5% and 95% tolerance bounds. Heavy lines are the random inhomogeneity analysis; the light lines are the conventional master curve analysis.

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_o values derived by both analyses are similar, the main difference comes in values of 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_o 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_o values, but it is not able to address the issue of proper description of the scatter in the transition fracture toughness.

Conclusion

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_o values derived using the conventional and random inhomogeneity analyses are quite similar. It appears from the analysis of data in this study that small number of sub-sized specimens used for irradiation studies could provide a reasonable estimate of irradiated T_o values, but they may not able to address the issue

of proper description of the scatter in the transition fracture toughness of irradiated F82H-IAE steel.

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