APPLICABILITY OF THE FRACTURE TOUGHNESS MASTER CURVE TO IRRADIATED HIGHLY EMBRITTLED STEEL AND INTERGRANULAR FRACTURE

R. K. NANSTAD, M.A. SOKOLOV, and D.E. McCABE
Oak Ridge National Laboratory
P.O. Box 2008
Oak Ridge, TN 37831-6151, USA
nanstadrk@ornl.gov

Abstract: *The Heavy-Section Steel Irradiation (HSSI) Program at Oak Ridge National Laboratory has evaluated a submerged-arc (SA) weld irradiated to a high level of embrittlement, and a temper embrittled base metal that exhibits significant intergranular fracture (IGF) relative to representation by the Master Curve. The temper embrittled steel revealed that the intergranular mechanism significantly extended the transition temperature range up to 150°C above T_o. For the irradiated highly embrittled SA weld study, a total of 21 1T compact specimens were tested at five different temperatures and showed the Master Curve to be nonconservative relative to the results, although that observation is uncertain due to evidence of intergranular fracture.*

Keywords: Master Curve, reactor pressure vessel, irradiation, intergranular fracture, temper embrittlement, fracture toughness, heat treatment.

1. Introduction

1.1 BACKGROUND

The issue of fracture toughness curve shape is a major aspect of the Heavy-Section Steel Irradiation (HSSI) Program at Oak Ridge National Laboratory (ORNL) sponsored by the U.S. Nuclear Regulatory Commission. In the early 1980s, the prospect of decreases in the slopes of the ASME Code K_{Ic} and K_{Ia} curves due to irradiation was discussed, and also included the relationship between the transition temperature shifts of Charpy impact toughness and fracture toughness. These discussions led to planning and execution of the HSSI Fifth (K_{Ic}) and Sixth (K_{Ia}) Irradiation Series [1,2], both conducted with two submerged-arc welds with copper contents of 0.23 and 0.31 wt% (HSSI Welds 72W and 73W), respectively. Fig. 1 shows the effect of an irradiation-
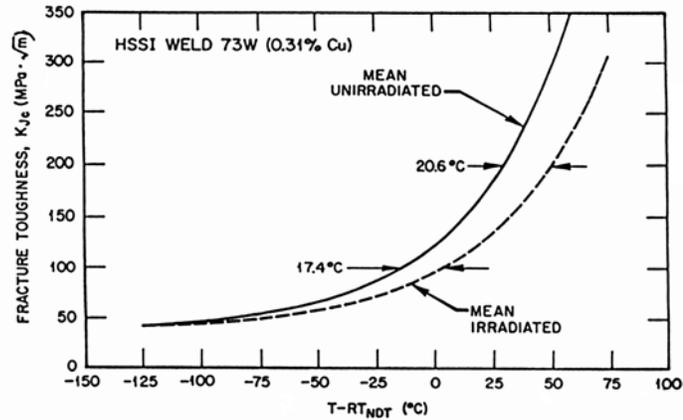
Figure 1. Mean curve fits to HSSI Weld 73W K_{Jc} data normalized to RT_{NDT} and showing effect of irradiation on curve shape.

induced shift of 100°C on the mean fracture toughness curve fits for HSSI weld 73W. The results indicated a change in shape due to irradiation, although statistical analysis concluded the slope change was significant at a 90% confidence level but not at a 95% confidence level. Fig. 2. shows results from Kaun and Koehring [3] in which they
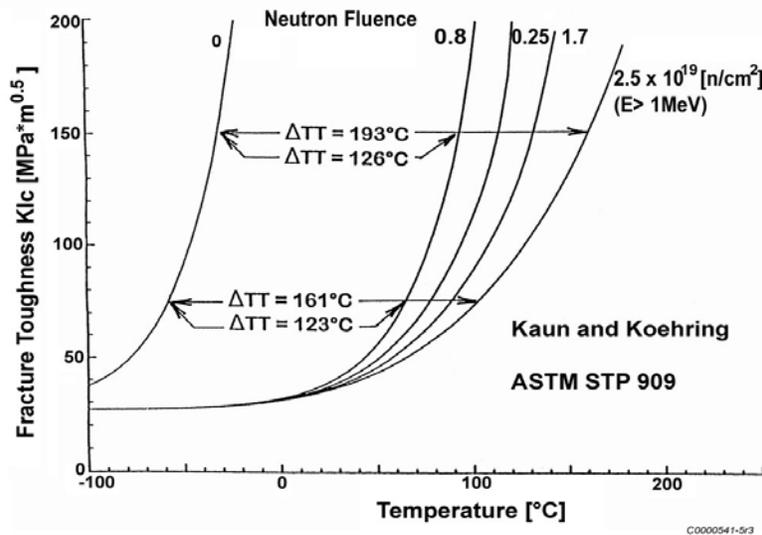

Figure 2. Mean curve fits to K_{Jc} data from Kaun and Koehring showing substantial decrease in curve slope with increasing irradiation fluence.

tested 4T specimens at different neutron fluences with fracture toughness transition temperature shifts up to about 170°C. As indicated on the figure, the slope of the curve fits decrease significantly at the higher fluences. The published paper does not provide the actual measured K_{Ic} data, so a statistical analysis is not possible. Regarding crack-arrest toughness, results from the HSSI Sixth Irradiation Series resulted in a conclusion that there was no apparent change in the shape of the mean curve fits to the K_{Ia} results. Those studies and others in the literature fit the data with, for example, three-parameter exponential functions in a manner that allowed for judgements regarding change in curve shape, but the methods are not consistent.

1.2 THE MASTER CURVE

The technology associated with the Master Curve has the potential of more accurate defining the position and shape of the ductile-to-brittle transition range for ferritic steels [4]. An integral part of the technology has been the observation that most grades of ferritic steels share a common transition curve shape. This has been the underlying assumption in the ASME code for almost 30 years regarding the form of lower-bound K_{IC} and K_{Ia} curves [5]. However, a statistical rationale for construction of those curves was not developed. On the other hand, the Master Curve analysis procedure has been developed with statistical concepts, and has been justified using a physical rationale. Wallin [6,7] developed the Weibull-based statistical analysis and procedures for specimen size normalization that resulted in a universal curve shape for cleavage fracture of ferritic steels. Equation 1 represents the three-parameter Weibull distribution for failure probability in which two of the parameters are fixed, a minimum K_{Jc} of 20 MPa√m and a shape parameter of 4, where B is the thickness of tested specimens and B_o is the reference thickness. Equation 2 represents the Master Curve normalized to temperature T_o, the temperature at which the median fracture toughness of a 1T specimen (the reference thickness B_o in equation (1) is 100 MPa√m.

$$P_f = 1 - \exp\left\{ -\frac{B}{B_o}\left(\frac{K_I - 20}{K_o - 20} \right)^4 \right\}, \quad (1)$$

$$K_{Jc(median)} = 30 + 70 \cdot \exp[0.019(T - T_o)], \quad (2)$$

These developments then eventually evolved into a consensus test standard, ASTM E 1921-97 [8], for determination of the parameter T_o, the background for which was published in reference [9]. There are now numerous publications in the literature demonstrating the applicability of the Master Curve to the cleavage fracture toughness of ferritic steels, especially RPV steels (see, for example, refs. 10 and 11). Studies are necessarily continuing, however, to investigate the phenomenological basis for the Master Curve as well as the limitations of its applicability. For example, Rathbun and Odette [12] have recently completed a comprehensive experimental study with test specimens of varying thicknesses and widths aimed at separating statistical effects and mechanical constraint.

1.3 MASTER CURVE AND IRRADIATION-INDUCED EMBRITTLEMENT

Other questions have been raised that challenge the universal aspect of the Master Curve. A case in point is that steels of low upper-shelf toughness will prematurely truncate their Master Curve trend, as demonstrated with the irradiated Midland Reactor RPV weld [11]. Cleavage crack initiation is assumed to be the controlling transition-temperature mechanism, and microstructural imperfections have been demonstrated to be the cleavage crack-triggering sources.[13] Usually, commercially produced steels conform to this behavior and this characteristic, to a limited amount of transition temperature shift, has been shown to be unchanged by embrittlement mechanisms such as irradiation damage. Sokolov and Nanstad [14] assembled and analyzed a fracture toughness database relative to the Master Curve and showed that, although the overall database of irradiated data indicated no significant deviation from the Master Curve shape, the subset of highly embrittled steels (those with transition temperature shifts of 100°C or greater) within that database did reveal a shallower slope. These results are shown in Fig. 3, which shows the multiplier parameter inside the exponent of the equations and reveals the lower value for the metals with a T_{41J} greater than 100°C. Thus, the results of Nanstad, et. al. on HSSI Weld 73W [1], those of Kaun and Koehring [3], and those of Sokolov and Nanstad [14] led to the HSSI investigation of a highly embrittled steel described in this paper.
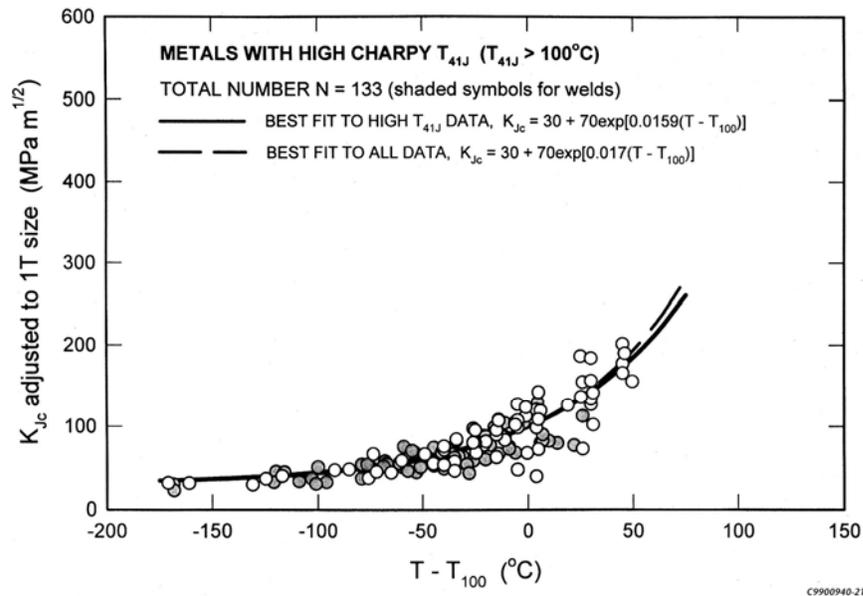

Figure 3. Mean curve fits comparing curve shape for all irradiated data with that for metals with a high T41J.

1.4 MASTER CURVE AND INTERGRANULAR FRACTURE

Another embrittlement mechanism that has not received sufficient attention relative to the Master Curve is intergranular fracture (IGF). As mentioned above and as explicitly stated in E 1921, the Master Curve development was based on fracture toughness results from transgranular cleavage fracture. Some metallurgical phenomena, such as temper embrittlement, can lead to diffusion of solutes to or out of grain boundaries such that the boundaries are weakened. This fracture mode is typically associated with temper embrittlement[15], a phenomenon that can lead to intergranular rather than transgranular fracture. A number of low alloy steels have been identified as being susceptible to this particular type of embrittlement mechanism. On the other hand, reactor pressure vessel (RPV) steels tend to be relatively insensitive to temper embrittlement.[16] However, if the prior austenite grain diameter is nominally 65 µm [American Society for Testing and Materials (ASTM) E112] grain size 5) or larger, sensitivity to temper embrittlement can be shown after long-term exposure within the critical temper embrittlement temperature range, which is about 400 to 600°C, a temperature well above the operating range for current light-water reactors, except in the case of post-irradiation thermal annealing. This condition has been the subject of study in a few recent experiments. For example, the heat treatment described in reference [17] was used on five heats of commercial RPV steels at ORNL. All were sensitized at high austenitization temperatures to obtain an ASTM grain size as large as 0 to 00 (360 to 510 µm grain diameter). After embrittlement-aging at 450°C (842°F) for 2000 h, Charpy impact transition temperature shifts, ΔT, ranged from 35 to 145°C (63 to 261°F). Another austenitization process used was a weld-cycle simulation, which produced a smaller average grain diameter, nominally 65 µm (ASTM E112, size 5). This embrittlement aging resulted in Charpy impact ΔTs of 56 to 133°C (101 to 239°F). Measurements of grain boundary composition demonstrated the significant increase of phosphorus in the aged steel. The same five commercially made steels in the as-received condition with ASTM grain sizes from 8 to 10, when embrittlement-aged, showed transition-temperature shifts from 0 to 64°C (0 to 115°F).

   Given the demonstrated potential to develop significant temper embrittlement in specially heat treated commercial RPV steels by aging, the HSSI Program embarked on a project to investigate the potential for temper embrittlement in an irradiated and thermally annealed coarse grain weld heat-affected zone. Such potential was demonstrated in specially heat treated, irradiated, and thermally annealed model steels by McElroy, et. al. [17] This study dramatically exhibited the issue of temper embrittlement in an RPV steel in that post-irradiation thermal annealing recovered the hardening but the ductile-brittle transition temperature was further increased and exhibited significant intergranular fracture. McCabe [16] utilized the electrical resistance method offered by the Gleeble system to prepare weld cycle simulated heat-affected-zone specimens of one of the five as-received A 302 grade B (modified) steels used in the thermal aging study. Charpy impact specimens were irradiated at 288°C to a fluence of 1×10^{23} n/m^2 (>1 MeV). Nanstad et al. [18] showed that, although the steel in the as-received condition exhibited no intergranular fracture following irradiation, the simulated heat-affected-zone exhibited 10 to 20% intergranular fracture following irradiation. Moreover, following post-irradiation thermal annealing at 460°C for 168 h,

the material exhibited predominantly intergranular fracture (>75%). These results are shown in the scanning electron fractographs in Fig. 4. The results of this study lend further support for the need to investigate the relationship between intergranular fracture and the Master Curve.
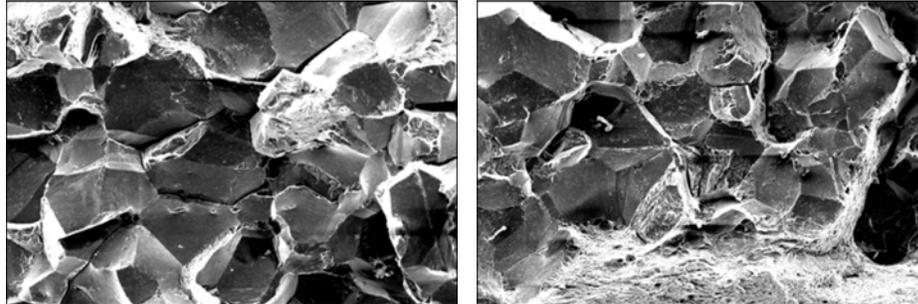

Figure 4. Gleeble simulated heat-affected-zone in A302B (mod) steel exhibited predominantly intergranular fracture after irradiation at 288°C to 1×10^{23} n/m² and annealing at 460°C/168 h.

2. Description of Materials

As stated earlier, the A 302 grade B (modified) steel was one of five steels tested in a previous thermal aging program [16]. The chemical composition and the tensile strengths of the steel in the final aged condition are provided in Table I; the heat treatment schedule is discussed in the next section. The phosphorus content in the steel is not particularly high, although it exhibited significant sensitivity to the temper embrittlement treatment. Additional properties of this material are available in references [16 and 19]. The submerged-arc weld, designated KS-01, was provided by MPA in Stuttgart, Germany, collaborators in the irradiation study. This weld was specially fabricated with the intent to produce a high-strength weld metal with high radiation-sensitivity. As shown in Table I, the high strength was achieved and high radiation sensitivity, discussed later, was achieved due to the high contents of copper, nickel, and phosphorus.

TABLE I. Chemical Composition and Tensile Strength for A302 grade B modified steel and SA Weld

Material	YS /UTS[a] (MPa)	C[b]	Mn	P	S	Si	Ni	Cr	Mo	Cu
A 302B (mod)[c]	593/730	0.26	1.42	0.010	0.014	0.17	0.53	0.09	0.50	0.17
SA Weld KS-01[d]	U: 600/700 I: 826/900	0.06	1.64	0.017	0.012	0.18	1.23	0.47	0.70	0.37

[a] YS and UTS are tensile yield strength and ultimate tensile strength, respectively.

[b] All chemical composition measurements are in wt%.

[c] Condition after austenitization, PWHT, and thermal aging at 460°C for 2000 h.

[d] U: Unirradiated condition; I: Irradiated condition

3. Temper Embrittlement Heat Treatment for Intergranular Fracture Study

The material preparation for the present experiment was as follows: austenitize at 1200°C (2192°F) for 30 min, oil quench and PWHT at 615°C for 24 h. The resulting grain size was ASTM 1.5, or a grain diameter of about 210 μm. For the aging treatment, specimens were heat treated at 460°C for 2000 h. The before-and-after embrittlement-aging Charpy transition curves are shown in Fig. 5. The transition temperature shift at the 41-J level was 122°C (252°F). A total of 10 0.5T compact specimens were tested in the unaged condition, while 50 compact specimens, 0.5T, 1.0T, and 2T, were machined from the temper embrittled steel and tested with the E 1921 procedure. Fracture toughness test specimens classified as 2T are more accurately denoted as 1.875T. These specimens had to be made slightly under size because they were machined from previously tested 4T specimens [16].

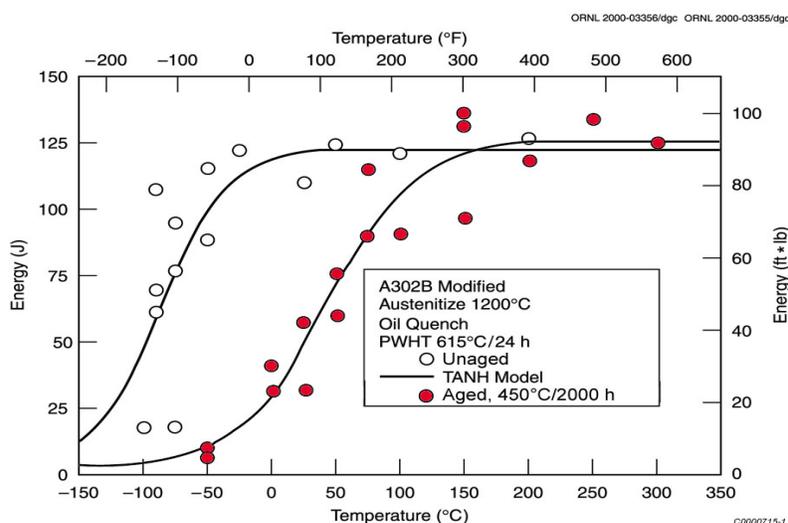

ORNL 2000-03356/dgc ORNL 2000-03355/dgc

C0000715-1

Figure 5. Charpy impact energy vs temperature data and curve fits for A302B (mod) steel after austenitization at 1200°C, oil quenching, and PWHT at 615°C/24 h: without aging and after aging at 460°C/2000 h.

4. Irradiation Conditions for the Highly Embrittled Weld Study

The SA Weld KS-01 was irradiated in the HSSI Irradiation/Anneal/Reirradiation (IAR) facility at the University of Michigan Ford Nuclear Reactor (FNR). Charpy, tensile, precracked Charpy, and 1T compact specimens were irradiated at $288°C \pm 2°C$ and a neutron flux of about 0.6×10^{16} n/m^2 to a neutron fluence of about 0.8×10^{23} n/m^2 (>1 MeV). A total of 21 1T compact specimens were irradiated and tested with the E 1921 procedure. Irradiated data, mostly Charpy and tensile, were available from MPA to provide guidance for selecting a desirable fluence. The target fluence was then selected

7

to result in a T_o after irradiation to be equal or slightly higher than 132°C (270°F). This temperature is the screening criterion reference temperature for pressurized thermal shock analysis of axial flaws in RPVs in the United States. The Charpy, tensile, and 1T compact specimen data are discussed in this paper.

5. Test Results

5.1 INTERGRANULAR FRACTURE OF TEMPER EMBRITTLED STEEL

The principal objective of the experiment was to determine whether fracture toughness data for ferritic steels that fail in an intergranular fracture mode follow the Master Curve shape. The first step was to establish the temperature, T_o, at which the median K_{JC} is 100 MPa√m for the unaged and embrittlement-aged materials. The tests for this purpose were made at −125 and −100°C (−193 and −148°F), respectively. The remaining specimens were used to establish the transition curve shape. Fig. 6 shows the results for the aged material, involving intergranular fracture, in the form of as-
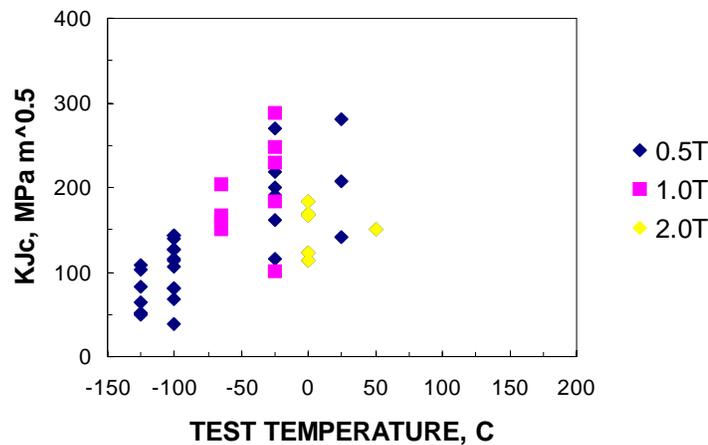

Figure 6. As-measured K_{Jc} data for temper embrittled A302B (mod) steel. All results shown represent specimens that failed by intergranular fracture.

measured K_{JC}, at each test temperature.

Figure 7 shows the median K_{Jc} adjusted results at each test temperature. The Master Curve is keyed to the first as-embrittled T_o temperature determined by testing at −125°C. The one test at 50°C (122°F) represents an attempt to find the upper-shelf temperature. Upper-shelf performance was expected from this specimen because full K_R-curve data had been produced in two out of the five tests made at 24°C (75°F). The 2T specimen tested at 50°C instead showed crack pop-in at 173 MPa√m, which was followed by stable crack growth up to 283 MPa√m and then crack instability. Hence, the transition range for the temper-embrittled material extends from below −100°C to above 50°C, significantly exceeding that for materials with conventional transition-
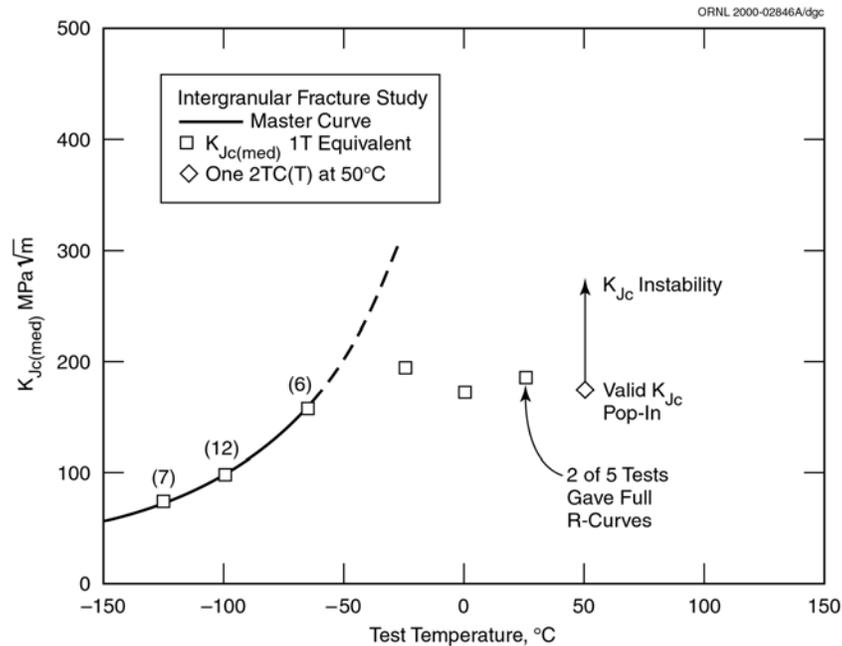
Figure 7. Median K_{Jc} values for temper embrittled A302B (mod) steel after normalization to 1T equivalence compared to Master Curve based on data at the three lowest test temperatures.

range mechanisms. A similar observation has been made by Kantidis, Marini, and Pineau for temper embrittled A533 grade B class steel [20]. When the transition range is controlled by a cleavage-to-ductile rupture mechanism, the transition range between T_o temperature and the upper shelf is of the order of 75 to 80°C. Four 0.5T compact specimens were subsequently tested at 100°C. One specimen exhibited a pop-in well out on the J-R curve, while the other three specimens showed fully ductile behavior but with evidence of ductile instabilities. Table II provides a summary of the various test results for the A302B (modified) steel in the as-received, heat treated, and aged conditions.

5.1.1. Fracture Surface Features

It was of interest to know whether the ratio of ductile versus intergranular fracture areas changes as upper shelf temperatures are approached. Figure 8 is an SEM fractograph for a test at 24°C (75°F). At 0° and 24°C (32 and 75°F), the fracture surfaces are identical, with almost 100% being intergranular. At –100°C (–148°F), a small percentage, on the order of 5%, is cleavage-like with the balance being intergranular. However, at 24°C (75°F) there was some slow-stable crack growth that preceded the specimen failure by intergranular fracture. Figure 8 shows the slow stable growth area

9

TABLE II. Transition temperature summary for intergranular fracture study.

Property/Condition	As Received	Aus + PWHT[a]	Aus + PWHT + Age[b]
Tensile Strength[c] (MPa)			
Yield Strength	472	-	593
Ultimate Strength	632	-	730
CVN Properties[b] (ºC)			
T_o	-9	-87	42
T_{41J}	-29	-107	15
T_{28J}	-43	-123	-4
CVN Upper Shelf Energy (J)	130	122	125
NDT (ºC)	-26	-	-
Fracture Toughness Properties			
Master Curve T_o (ºC)	-88	-140	-100
J_{Ic} (kJ/m^2)	212	266	199
T_{mod}	96	71	86
ASTM Grain Size	9	1.5	1.5

[a]Aus means austenitize at 1200°C for 30 min. and oil quench. PWHT is post-weld heat treat at 615°C/24.
[b]Same condition as in footnote a, plus aging at 2000 h at 450°C.
[c]Room-temperature tests.
[d]This T_o stands for temperature at mid-Charpy impact transition energy.

near the left border of the figure, with the remaining area being intergranular fracture, while figure 9 shows a magnification of the area of ductile fracture.
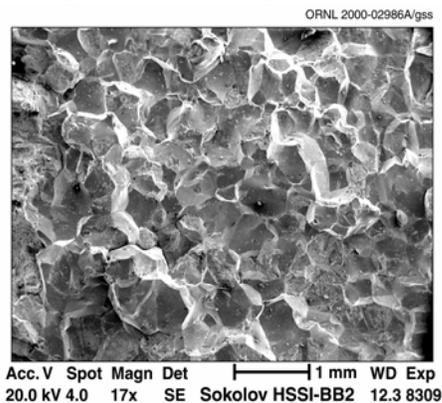

Figure 8. Scanning electron fractograph of a 0.5T compact specimen of the temper embrittled A302B (mod) steel tested at 24C, with nearly complete intergranular fracture.
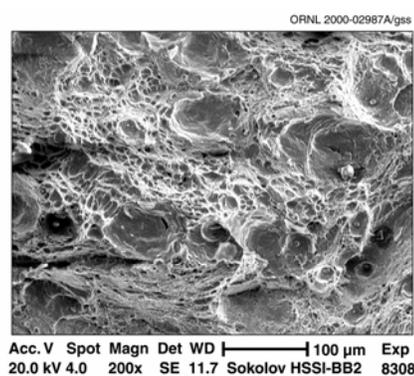

Figure 9. Scanning electron fractograph of the stable crack growth area preceding brittle intergranular fracture of the specimen

As mentioned before, the intergranular fracture portion of Fig. 8 does not appear to be much different from those at significantly lower temperatures. These fractographs illustrate why intergranular fracture causes such a delayed development of upper-shelf behavior, viewed in terms of fracture appearance and fracture toughness.

5.1.2 *Fracture Toughness Characteristics*

The present data have demonstrated that the transition-range behavior of steels that are embrittled by intergranular mechanisms is different from that for steels whose transition-range mechanisms are cleavage-to-ductile tearing. The correlations with Charpy data developed in the latter case do not apply in the former case. For example, embrittlement by neutron irradiation still results in the cleavage-to-ductile transition, and the transition-temperature shift occurs with little or no change in Master Curve shape. Also, there is some reduction of upper-shelf ductile-tearing resistance. At upper-shelf temperatures, both Charpy impact energy and K_R-curve fracture toughness mechanisms are diminished by irradiation. The present intergranular damage mechanism affects only transition temperature shift. Examination of the data show that there is no difference in K_R-curves caused by transforming the commercially produced material, by the heat treatment including aging, into an enlarged grain intergranular sensitized steel. The as-embrittled Charpy impact transition temperature, however, is considerably affected, comparing $-9°C$ to $42°C$ ($16°F$ to $108°F$), as indicated by Table II. Comparison of K_R-curves before and after embrittlement aging (large prior austenite grains) show only a slight decrease in the resistance curve for the aged material. In this case, the initial Charpy impact transition temperature was $-87°C$ ($-125°F$) and, as previously mentioned, that for the aged condition was $42°C$ ($108°F$). The one feature that intergranular and cleavage fracture modes have in common is that side-grooving of specimens reduces the fracture toughness described in terms of K_R-curve development [16, 18]. The effect on the intergranular material seems to be quite severe.

5.2 IRRADIATED HIGHLY EMBRITTLED SUBMERGED-ARC WELD

Following irradiation, Charpy impact, tensile, and 1T compact specimens of weld KS-01 were tested. Fig. 10 shows the Charpy impact data in both the unirradiated and irradiated conditions. The 41J temperature shift was $169°C$, while the upper-shelf decreased from 124 to 78 J, a decrease of 37%. Fig. 11 shows the yield and ultimate tensile strengths in both conditions. At room temperature, the yield strength increased from 600 to 826 MPa, an increase of 38%.
The ratio of Charpy shift in degrees centigrade and the increase in yield strength in MPa is 0.75, a value in close agreement with the 0.65 result reported by Odette, Lombrozo, and Wullaert [21], and with the value of 0.70 for low upper-shelf welds reported by Nanstad and Berggren for low upper-shelf welds. [22] Fig. 12 (a) shows the median K_{Jc} results at the lowest three test temperatures and the Master Curve fit to those three values. Fig. 12 (b) shows all the K_{Jc} data at all test temperatures and a similar effect as exhibited by the temper embrittled steel; that is, brittle fracture results at temperatures further above T_o than expected ($T_o + 63°C$), and a leveling off of the K_{Jc} data at about 110 MPa√m. Taking the T_o determination from the curve fit in

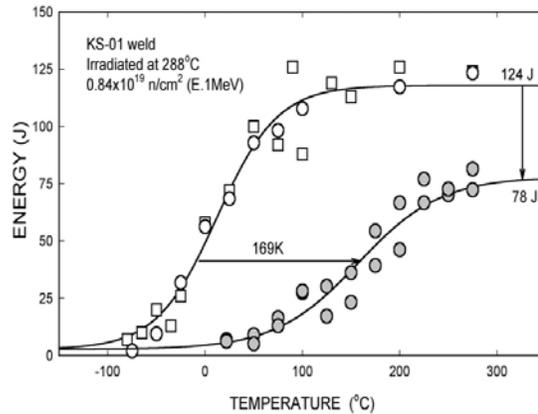Fig 12 (a), the ΔT_o was about 160°C. Scanning electron fractography revealed evidence of intergranular fracture mixed with cleavage fracture in the irradiated specimens. The cleavage fracture mode was predominant, but the intergranular fracture was estimated to be 10 to 20%. Interestingly, scanning electron fractography of the unirradiated fracture toughness specimens also showed a similar amount of intergranular fracture, although the unirradiated data did not exhibit similar brittle fracture at high temperatures beyond T_o. This result rather confounds the issue of the irradiation-induced change in curve shape and is still under evaluation. A more detailed presentation of this study is published in ref. [23].

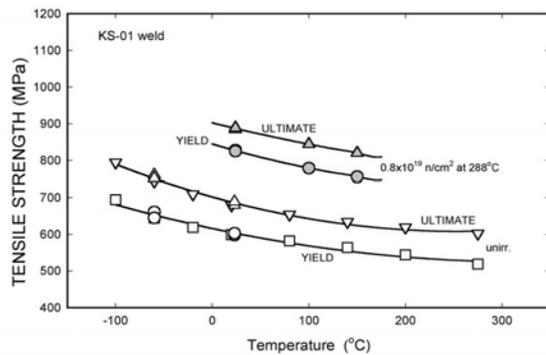*Figure 10. Charpy impact data for submerged-arc weld KS-01 in the unirradiated and irradiated conditions.*

Figure 11. Yield and ultimate tensile strengths vs temperature for submerged-arc weld KS-01 in the unirradiated and irradiated conditions.

6. Discussion

The original hypothesis that the fracture toughness of materials that fail due to intergranular fracture may not be appropriately represented by the Master Curve is supported by the results discussed in this paper. The Master Curve has been assumed to be universal for all ferritic steels, as limited to a certain yield-strength range. [8] The supporting data were for steels that displayed cleavage-to-ductile mechanisms in both unirradiated and irradiated conditions. Intergranular fracture is principally associated with temper-embrittlement (TE) damage, a mechanism that is not usually attributed to Western-type RPV steels. Commercially manufactured

RPV steels with prior-austenite grain sizes that range from 8 to 11 are not subject to significant TE damage.

The present experimental results illustrated in Fig. 7 have demonstrated that intergranular fracture is in fact unique from the standpoint of transition-range behavior. Although there was a moderate fracture toughness increase over a 150°C (270°F) transition range, there was almost no change in fractographic appearance over the same temperature range. This suggests that there is no operative weakest-link triggering mechanism governing intergranular fracture. Of additional interest is the fact that the Master Curve T_o temperature shift was only 40°C (72°F) in a material that had a 122°C (207°F) Charpy impact transition-temperature shift. Fig. 13 shows a comparison
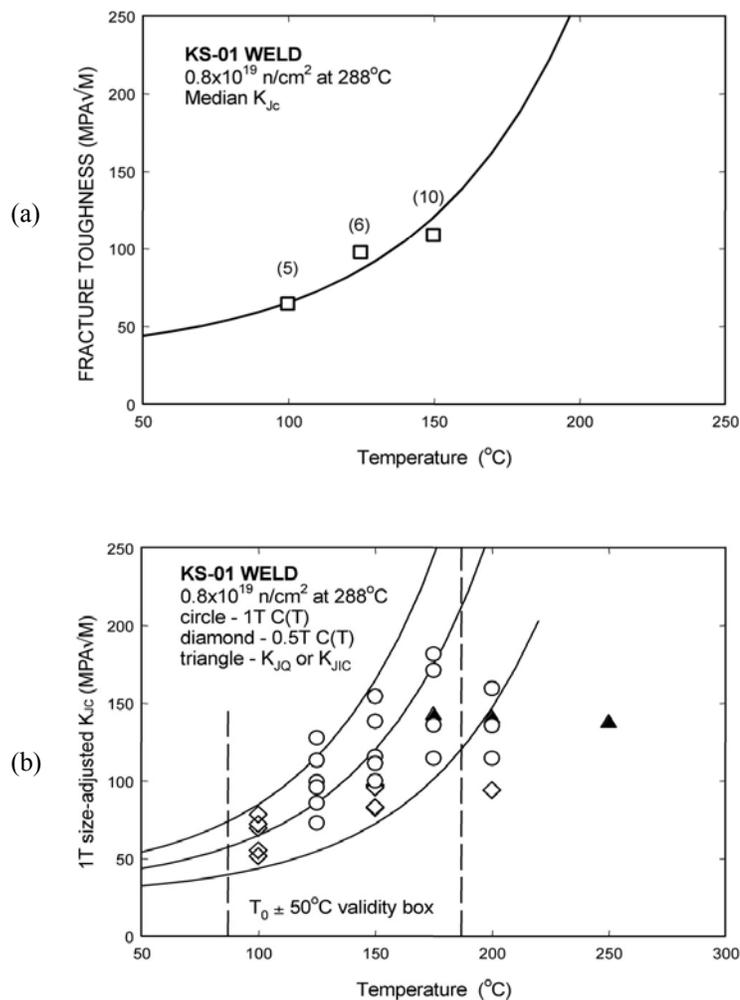

Figure 12. Fracture toughness, K_{Jc}, data for submerged-arc weld KS-01 in the irradiated condition (a shows median values at the lowest three temperatures with Master Curve fit to those values; (b) shows all as-measured data compared with Master Curve 5 and 95% tolerance bounds.

of T_o vs T_{41J} for a database of RPV steels [14] and the A302B (mod.) steel in three conditions. This relationship for the steel falls within the database scatter band for the as-received and austenized/PWHT conditions. However, that for the aged condition (i.e., temper embrittled) falls well outside the scatter band. Hence, there was a breakdown in the T_o vs Charpy impact curve correlations that have been developed for steels with cleavage operative mechanisms.

Evidence of this correlation breakdown was also noted in Ref. [20]. Figure 8 shows that the relation between fracture toughness and temperature appears to follow the Master Curve until fracture toughness is about 150 MPa√m , but then levels off just above that value. Despite the leveling off of fracture toughness, the fracture appearance continues to be predominantly intergranular rather than completely ductile. For this material, even at 100°C, one of four specimens exhibited an audible and relatively large pop-in instability.
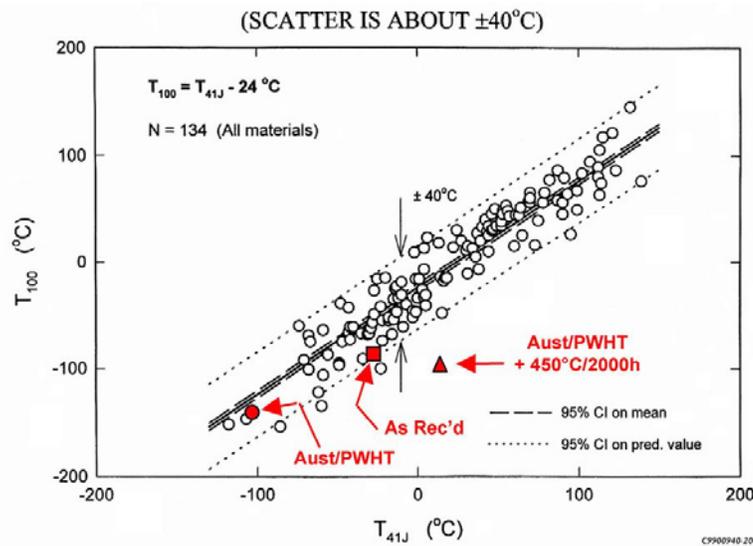

Figure 13. Comparison of fracture toughness transition temperature T_o (labeled T_{100} here) and Charpy T_{41J} for the A302B (mod) steel in three conditions with the RPV steels database from Ref. [14]., showing that the aged steel falls well outside of the 95% scatter band of ± 40ºC.

The extreme intergranular condition created in the present work does not represent more realistic cases involving considerably lower percentages of intergranular separation mixed with cleavage. However, the present work does suggest that the ΔT embrittlement-transition-temperature shift as measured by Charpy impact curves may

14

not be a reliable indicator for evaluations of changes in fracture-mechanics properties for temper-embrittled steels if intergranular fracture occurs.

This latter case of mixed intergranular and cleavage fracture applies to the irradiated KS-01 submerged-arc weld. The evidence of some curve shape change reported for HSSI weld 73W by Nanstad, et. al. [1] was represented by all cleavage fractures with no intergranular fracture. Although Kaun and Koehring [3] reported significant curve shape changes, the fracture modes were not reported. The KS-01 irradiated results indicate that the Master Curve applies to a limited extent such that brittle fracture may occur at temperatures well above T_o. In this case, however, it is difficult to separate the effects of irradiation from the 10 to 20% intergranular fracture observed on the fracture surfaces. On the other hand, the transition temperature shifts of the Charpy impact toughness and the fracture toughness were in good agreement (160 vs 169°C), unlike the temper embrittled steel where the difference was very large (40 vs 122°C). This may be an indication that intergranular fracture at the 10 to 20% level did not play a significant role in the fracture toughness tests. The very high transition temperature shifts lead to a significant reduction in the Charpy upper-shelf toughness such that the KS-01 steel behaves similar to other low-upper shelf steels by exhibiting low ductile initiation fracture toughness. The difference is that the onset of brittle fracture beyond the J_{Ic} toughness seems to result in lower than expected values of K_{Jc}, and it is this observation that results in fracture toughness values outside of the Master Curve tolerance bounds. This behavior may be due to the influence of intergranular fracture.

7. Summary and Conclusions

Two different RPV steels, one in the temper embrittled condition and the other in the irradiated condition, have been tested to obtain fracture toughness data and the results were evaluated relative to the fracture toughness Master Curve.

1. An A302 grade B (modified) steel plate was given an austenitization treatment that was needed to create 100% intergranular fracture. Temper-embrittlement aging at 450°C (482°F) for 2000 h resulted in a transition-temperature shift from –73°C to 42°C (–99°F to 108°F) as measured by Charpy impact curves, and from –140°C to –100°C (– 220°F to –148°F) as measured by Master-Curve-based T_o temperatures.

2. An original hypothesis that the transition-range fracture toughness versus temperature curve for materials fracturing by intergranular fracture might differ from that for the cleavage mode proved to be correct.

3. The occurrence of predominantly intergranular fracture extended the transition range of temperature between lower bound and upper-shelf behavior by a factor of at least 2 with respect to that for a typical cleavage-transition range.

4. The difference found between the Charpy-curve ΔT and the fracture-mechanics-based ΔT_o implies that correlations between the two, based on cleavage data, are not applicable when intergranular fracture is the predominant mode of fracture. A

remaining issue, however, is the propensity for some RPV steels (e.g., coarse grain heat-affected-zones) to be temper-embrittled following irradiation and thermal annealing at 450°C/168 h. Therefore, there is a need for more evaluations of temper-embrittlement effects on commercially produced steels, using fracture mechanics data as opposed to just Charpy impact evaluation.

5. For the irradiated KS-01 submerged-arc weld, brittle fracture results at temperatures further above T_o than expected ($T_o + 63$°C), and a leveling off of the K_{Jc} data at about 110 MPa√m. This behavior was not observed in the unirradiated condition, although the conclusion regarding curve shape change due to irradiation is confounded by the potential influence of 10 to 20% intergranular fracture observed on the fracture surfaces.

8. Acknowledgements

References

1. Nanstad, R. K. McCabe, D. E., Haggag, F. M., Bowman, K. O., and Downing, D. J., **"**Statistical Analyses of Fracture Toughness Results for Two Irradiated High-Copper Welds," *Effects of Radiation on Materials: 15th International Symposium, ASTM STP 1125*, R. E. Stoller, A. S. Kumar, and D. S. Gelles, Eds., American Society for Testing and Materials, Phildelphia, 1992, pp. 270-291.

2 Iskander, S. K., Corwin, W. R., and Nanstad, R. K., **"**Effects of Irradiation on Crack-Arrest Toughness of Two High-Copper Welds**,"** *Effects of Radiation on Materials: 15th International Symposium, ASTM STP 1125*, R. E. Stoller, A. S. Kumar, and D. S. Gelles, Eds., American Society for Testing and Materials, Phildelphia, 1992., pp. 251-269.

3. Kaun, U. E. and Koehring, F. K. A., **"**Investigation and Measures to Guarantee the Safety of a Pressurized Water Reactor's Pressure Vessel Against Brittle Fracture**,"** *Radiation Embrittlement of Nuclear Reactor Pressure Vessel Steels: An International Review (Second Volume), ASTM STP 909*, L. E. Steele, Ed., American Society for Testing and Materials, Philadelphia, 1986, pp. 127-148.

4. M.A. Sokolov, K. Wallin, and D.E. McCabe, "Application of Small Specimens to Fracture Mechanics Characterization of Irradiated Pressure Vessel Steels," *Fatigue and Fracture Mechanics: 28th Volume*, ASTM STP 1321, ed. J. H. Underwood, B. D. MacDonald, and M. R. Mitchell, American Society for Testing and Materials, Philadelphia, 1997, pp. 263–279.

5. ASME Boiler and Pressure Vessel Code, Section XI, *Rules for In service Inspection of Nuclear Power Plant Components*, Appendix A, American Society of Mechanical Engineers, New York, 1992.

6. Wallin, K., "The Scatter in K_{IC} Results," *Engineering Fracture Mechanics,* **19(6)**, pp. 1085-1093, 1984.

7. Wallin, K., "The Size Effect in K_{Ic} Results," *Engineering Fracture Mechanics,* **22**, pp. 149-163, 1985.

8. "Determination of Reference Temperature, T_o, for Ferritic Steels in the Transition Range, E1921-97," *Annual Book of ASTM Standards*, Vol. 03.01, American Society for Testing and Materials, Philadelphia, 1998.

9. J.G. Merkle, K. Wallin, and D.E. McCabe, *Technical Basis for an ASTM Standard on Determining the Reference Temperature, T_o, for Ferritic Steels in the Transition Range*, USNRC Report NUREG/CR-5504 (ORNL/TM-13631), Oak Ridge National Laboratory, Oak Ridge, Tenn., 1998.

10. Kirk, M., Lott, R., Kim, C., and Server, W., "Empirical Validation Of The Master Curve For Irradiated and Unirradiated Reactor Pressure Vessel Steels," *Proceedings of the 1998 ASME/JSME Pressure Vessel and Piping Symposium*, July 26-30, 1998, San Diego, California, USA.

11. D.E. McCabe, R.K. Nanstad, S.K. Iskander, D.W. Heatherly, and R.L. Swain, *Evaluation of WF-70 Weld Metal from the Midland Unit 1 Reactor Vessel, Final Report*, USNRC Report NUREG/CR 5736 (ORNL/TM-13748), Oak Ridge National Laboratory, Oak Ridge, Tenn., February 1998.

12. Rathbun, H. , Odette, G. R., *Hribernik, Michael L. and He, Ming Y.,* "Progress on Establishing a Fundamental Basis for the Master Curve Method,**"** Symposium on Resque and Referee, Mol, Belgium, September, 2001.

13. J. Heerens, D.T. Read, A. Cornec, and K.H. Schwalbe, "Interpretation of Fracture Toughness in the Ductile-to-Brittle Transition Region by Fractographical Observations," *Defect Assessment in Components— Fundamentals and Applications*, ESIS/EFG9, J.G. Blauel and K. H. Schwalbe, Eds., 1991, pp. 659–678.

14. Sokolov, M.A., and Nanstad, R.K., "Comparison of Irradiation Induced Shifts of K_{JC} and Charpy Impact Toughness for Reactor Pressure Vessel Steels," *Effects of Radiation on Materials: 18th 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, West Conshohocken, PA, 1999, pp. 167-190.

15. C.J. McMahon, Jr., V. Vitek, and J. Kameda, "Mechanics and Mechanisms of Intergranular Fracture," Chapter 4 in *Developments in Fracture Mechanics-2*, G.G. Chell, Ed., Applied Science Publishers, Englewood, N.J., 1981.

16. D.E. McCabe, "*Initial Evaluation of the Heat-Affected Zone, Local Embrittlement Phenomenon as it Applies to Nuclear Reactors"*, ORNL Letter Report ORNL/NRC/LTR-99/10, Oak Ridge National Laboratory, Oak Ridge, Tenn., September 1999.

17. McElroy, R. J. A. J. E. foreman, Gage. G, Phythian, W. J., Ray, P. H. N. and Vatter, I. A., "Optimization of Reactor Pressure Vessel Surveillance Programmes and their Analysis," contribution to IAEA CRP 3 Research Program, AEA-RS-2426, December, 1993.

18. Nanstad, R. K., McCabe, D. E., Sokolov, M. A., English, C. A., and Ortner, S. R., "Investigation of Temper Embrittlement in Reactor Pressure Vessel Steels Following Thermal Aging, Irradiation, and Thermal Annealing," *Effects of Radiation on Materials: 20th International Symposium, ASTM STP 1405*, S. T. Rosinski, M. L. Grossbeck, T. R. Allen, and A. S. Kumar, Eds., American Society for Testing and Materials, West Conshohocken, PA, 2001.

19. Nanstad, R. K., McCabe, D. E., and Merkle, J. G., "Relationship of Fracture Toughness from Intergranular Fracture to the Master Curve," *Proceedings of International Atomic Energy Agency Specialists' Meeting on Master Curve Testing and Applications*, 17-19 September, 2001, Prague, Czech Republic.

20. E. Kantidis, B. Marini and A. Pineau, *A Criterion for Intergranular Brittle Fracture of a Low Alloy Steel,* Fatigue Fract. Engng Mater. Struct. Vol. 17, No. 6, pp 619-633, 1994.

21. Odette, G. R. Lombrozo, P. M., and Wullaert, R. A., "Relationship Between Irradiation hardening and Embrittlement of Pressure Vessel Steels," *Effects of Radiation on Materials: Twelfth International Symposijm, ASTM STP 870*, F. A. Garner and J. S. Perrin, Eds., American Society for Testing and Materials, Philadelphia, 1985, pp. 840-860.

22. Nanstad, R. K. and Berggren, R. G., "Effects of Irradiation Temperature on Charpy and Tensile Properties of High-Copper, Low Upper-Shelf, Submerged-Arc Welds," *Effects of Radiation on Materials: 16th International Symposium, ASTM STP 1175*, A. S. Kumar, D. S. Gelles, R. K. Nanstad, and E. A. Little, Eds., American Society for Testing and Materials, Philadelphia, 1993.

23. Sokolov, M.A., Nanstad, R.K., and Miller, M.K., "Fracture Toughness Characterization of a Highly Embrittled RPV Weld," *Effects of Radiation on Material:21st International Symposium, ASTM STP 1447*, M.L. Grossbeck, Ed., ASTM International, West Conshohocken, PA, 2004.