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INTER-LABORATORY RESULTS AND ANALYSES OF MINI-C(T) SPECIMEN TESTING OF AN IRRADIATED LINDE 80 WELD METAL

William Server
ATI Consulting
Black Mountain, NC 28711 USA

Mikhail Sokolov
Oak Ridge National Laboratory
Oak Ridge, TN 37830 USA

Masato Yamamoto
CRIEPI
Yokosuka, Kanagawa 240-0196 JAPAN

Robert Carter
Electric Power Research Institute
Charlotte, NC 28221 USA

ABSTRACT

An irradiated low-upper-shelf Linde 80 weld metal has been tested by four laboratories as part of an inter-laboratory assessment of use of the miniature compact tension [mini-C(T)] test specimen for Master Curve fracture toughness evaluation following ASTM E1921. The preliminary results from each of the laboratories have been compiled and evaluated together to assess the validity and use of the mini-C(T) specimen for an irradiated reactor pressure vessel material which can exhibit ductile crack growth at low temperatures relative to cleavage initiation fracture toughness. The preliminary results from this mini-C(T) testing can also be compared to extensive specimen test results from larger C(T) specimens of the same irradiated material. Comparisons of the results from each of the laboratories and some inter-laboratory differences in the fracture testing are assessed. The evaluations indicate reasonable agreement between the mini-C(T) and larger specimen results, but the selection of test temperature and the number of test specimens needed to obtain reliable results are more difficult when testing a low-upper-shelf toughness material.

INTRODUCTION

The use of miniature fracture toughness specimens in the Master Curve approach as prescribed in ASTM Standard Test Method E1921 [1] to determine material-specific fracture toughness of ferritic steels is strongly desired especially for irradiated reactor pressure vessel (RPV) steels. The volume of irradiated material is generally small, and in the case of nuclear RPV surveillance programs, material the size of broken Charpy V-notch (CVN) specimens is often all that is available. The mini-C(T) specimen is a compact tension fracture toughness specimen that has a thickness of 0.16 in. (4 mm) and is termed a 0.16-T C(T) – see Figure 1. Two different arrangements are possible for the determination of load-line displacement during the test. As shown in Figure 1, front face measurements are possible using the machined knife-type edges. Determination of the actual load-line displacement requires a correction based upon the location of the measurement points and the actual load line. Alternatively, so-called outboard measurements can be made exactly on the load line by having the machined 90° notches placed on the outside surfaces in line with the centerline of the clevis pin holes as shown in Figure 1. The advantage of the outboard approach is that one is measuring the displacement directly at the load-line without the need to correct for position.

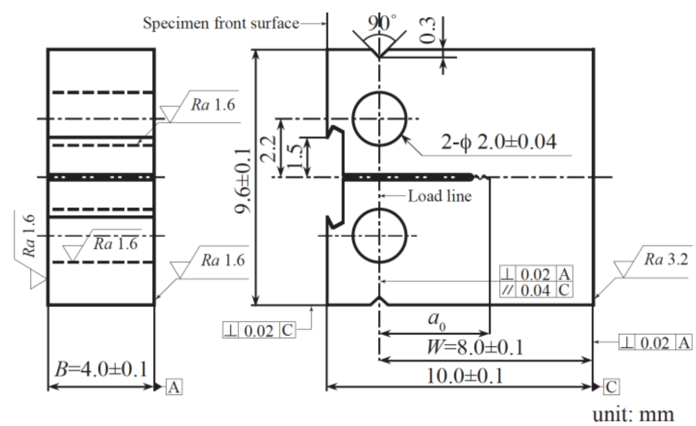

Figure 1. Geometry and Size of Mini-C(T) Specimen

From a base metal broken CVN specimen half, up to four mini-C(T) specimens can be prepared maintaining the same testing orientation as in the CVN test. It is also possible to change the orientation if desired and even more mini-C(T) specimens can be obtained. If the entire broken CVN half is weld metal, again up to four mini-C(T) specimens of the same orientation can be obtained; however, many weld metal broken CVN halves are not entirely weld metal, and only two mini-C(T) specimens can be machined maintaining the same orientation.

The primary question to be answered is: can the mini-C(T) specimen produce reliable ASTM E1921 reference temperature, T_0, values equivalent to the more commonly used ½ in. and 1 in. (12.7 mm and 25.4 mm) thickness fracture toughness specimens, 0.5T-C(T) and 1T-C(T), for all irradiated RPV steels? Work has been ongoing on the development and use of the mini-C(T) specimen to be applicable to RPV steels in both the non-irradiated and irradiated conditions as documented in previous PVP Conferences [2-6]. Validity of using the mini-C(T) Master Curve approach has been shown on non-irradiated base metals of typical Japanese RPV steels, including an international round robin [2-4] to confirm reproducibility of mini-C(T) Master Curve fracture toughness data. Larger specimen Master Curve fracture toughness data for these base metals was available to compare with the measured mini-C(T) test results. Testing of non-irradiated weld metals and irradiated base and weld metals also has shown good agreement between mini-C(T) and larger specimen measured T_0 values [5-7]. One type of RPV steel that can provide the biggest challenge for the mini-C(T) specimen is an irradiated low-upper-shelf (LUS) weld metal. This paper provides a comparison of results from a four-laboratory round robin testing program using an irradiated LUS weld metal for which a database of larger specimen fracture toughness results exists. Please note that final validation of all test data from each laboratory is still underway, so the results presented here are preliminary, although any changes should be minor.

MATERIAL AND MINI-C(T) SPECIMEN PREPARATION

The material evaluated in this study is a Linde 80 flux weld metal from the Midland RPV circumferential weld, which has the heat code 72105, made using Linde 80 weld flux lot 8669 and designated as WF-70. The Midland RPV was never put into actual service, but this weld metal heat is known to have a LUS, especially in the irradiated condition. The reason that a LUS material can be more of a challenge is the early initiation of ductile crack growth (DCG) due to the LUS prior to cleavage-initiated fracture. The mini-C(T) specimen has a limited temperature range in which valid test data can be generated, and the LUS material further limits this temperature range. The potential for DCG prior to cleavage fracture can create a unique testing issue in which many mini-C(T) specimens will exhibit excessive DCG (i.e., greater than 5% of the remaining ligament length, b_o, or 0.2 mm) before cleavage fracture; or in a few cases, no cleavage fracture at all.

The Midland WF-70 weld metal was tested at Oak Ridge National Laboratory (ORNL) in the non-irradiated condition using test specimens ranging from 0.4T-C(T) to 4T-C(T). Additionally, there were some 0.4T (0.4 in, 10 mm thickness) three-point bend (precracked Charpy) specimens tested. These results have been reported by ORNL and the combined Master Curve T_0 temperature was reported to be -60°C [8]. However, Sokolov [9] has reanalyzed the data using the latest procedures in ASTM E1921, and the unirradiated global T_0 value is calculated to be -60°C. Eleven mini-C(T) specimens were tested with a measured value of T_0 of -53°C [9], which is in excellent agreement with the larger specimen results.

The WF-70 weld metal also was irradiated in the Ford Nuclear Reactor (FNR), and 0.5T-(CT), 1T-C(T), and precracked Charpy specimens were tested to determine the irradiated value of T_0 [8] corresponding to an average neutron fluence of 1 x 10^{19} n/cm^2 (E > 1 MeV). The global reported T_0 value was 27.4°C, and the irradiated CVN upper shelf energy was 80 J (reduced from the non-irradiated value of 89 J). Sokolov [9] also reanalyzed the irradiated specimen data and determined that the global irradiated T_0 is -18°C. Broken CVN and precracked Charpy test specimen halves from the irradiation study were machined into sixty (60) mini-C(T) specimens for this evaluation. Three different blocks from the Midland weld were used in fabricating the original CVN and precracked Charpy specimen halves.

All of the mini-C(T) specimen machining was performed by one laboratory (Laboratory A) and fifteen (15) specimens each provided to the three other laboratories. Fatigue precracking also was performed by Laboratory A for testing at Laboratories A, B, and D. Laboratory C performed both fatigue precracking and testing for their specimens. Note also that the specimens provided to Laboratories C and D had a slightly higher height dimension of 10.0 mm instead of 9.6 mm as shown in Figure 1. This minor difference in dimension should have no effect on the test results since all specimens are precisely measured prior to any testing and actual dimensions are used in the calculations. The reason for this small variation was to assess any possible difference when the specimen orientation is altered.

The two load-line measurement methods (front face machined knife edges and direct outboard load-line) were assessed. Laboratories A and B used the machined knife edges on the front face; Laboratory C used the outboard direct load-line method; and, Laboratory D used both methods simultaneously. There were only small differences in the analyzed values of K_{Jc} between the methods, and the effect on the determination of T_0 was insignificant based on the results from Laboratory D. The load-line results are presented in Table 1 for Laboratory D.

The Master Curve fracture toughness testing of these irradiated specimens is the focus of this investigation. Since the upper shelf ductile initiation toughness of the irradiated weld metal may have decreased to a level near the T_0 value corresponding to 100 MPa√m, the interaction of the LUS ductile toughness with the Master Curve cleavage initiation toughness is being assessed in this study. The measured K_{Jc} values from the mini-C(T) tests are converted to equivalent 1T-C(T) values, $K_{Jc(1T)}$, following the specimen size adjustment in ASTM E1921:

$$K_{Jc(1T)} = 20\ \mathrm{MPa\sqrt{m}} + [K_{Jc} - 20\ \mathrm{MPa\sqrt{m}}]\ (B_{mini}/B_{1T})^{1/4} \quad (1)$$

where B_{mini} is the specimen thickness of the mini-C(T) and B_{1T} = 25.4 mm.

EXPERIMENTAL RESULTS

The four laboratories and the valid test results following ASTM E1921 are listed in Table 1. Of the sixty (60) test specimens, one specimen was lost due to a malfunction during the test, seven other specimens did not meet the fatigue crack front straightness criteria, and two specimens did not meet the fatigue crack length requirement. The tests listed as having excessive DCG (greater than 0.2 mm) have censored values corresponding to the highest value of $K_{Jc(1T)}$ from all the tests where no excessive DCG was observed; this value is termed $K_{Jc(1T)(\Delta a)}$. Specimen MW9A14-A1 from Laboratory C had the highest non-DCG value of $K_{Jc(1T)}$ = 80 MPa√m.

Another censoring limit is based on the specimen size and is termed $K_{Jc(limit)}$ as prescribed in ASTM E1921 [1]:

$$K_{Jc(\text{limit})} = \sqrt{\frac{E b_0 \sigma_y}{30\left(1-\nu^2\right)}} \tag{2}$$

where E is the elastic modulus (in GPa) as a function of temperature (T, °C):

$$E = 204 - T/16 \tag{3}$$

σ_y is the material yield strength in MPa as a function of T based upon the average room temperature value of 646 MPa:

$$\sigma_y = 646 + 10^5 / (491 + 1.8\ T) - 189 \tag{4}$$

b_0 is the initial ligament size ($W - a_o$) as shown in Figure 1, and ν is Poisson's ratio. The value of $K_{Jc(limit)}$ is then adjusted to reflect the 1T size, $K_{Jc(1T)(limit)}$, by using Eq. 1. None of the cleavage fracture test specimens had $K_{Jc(1T)}$ values equal to or greater than $K_{Jc(1T)(limit)}$.

Specimen MW15AK3-A1 from Laboratory C exhibited a cleavage pop-in from which the value of $K_{Jc(1T)}$ was determined at the pop-in as indicated in Table 1.

Six of the tests did not exhibit any cleavage fracture as listed in Table 1. The tests were terminated at $K_{Jc(1T)}$ values in excess of $K_{Jc(1T)(limit)}$. Since none of the cleavage test results ever reached $K_{Jc(1T)(limit)}$, but several exceeded the level of $K_{Jc(1T)(\Delta a)}$ = 80 MPa√m, it is likely that all of the non-cleavage tests exceeded the $K_{Jc(1T)(\Delta a)}$ level and showed extensive DCG before exceeding the $K_{Jc(1T)(limit)}$. The interpretation of which censoring to use is vague in ASTM E1921. In section 8.9.2 it is stated that

> "A K_{Jc} datum requires censoring if the specimen exceeds the $K_{Jclimit}$ … or if a test has been discontinued at a value of K_J without cleavage fracture after surpassing $K_{Jclimit}$. Another limit, $K_{Jc\Delta a}$, is violated in tests that terminate in cleavage after slow stable crack growth … A K_{Jc} datum exceeding $K_{Jc\Delta a}$ also requires censoring… the K_{Jc} datum shall be replaced with $K_{Jc\Delta a}$….If both $K_{Jclimit}$ and $K_{Jc\Delta a}$ are violated, the lower value of the two shall be used to replace the K_{Jc} datum for data censoring purposes in the analysis."

One interpretation is that the DCG limit should be used since it is the lower of the two values, but it is also stated that the DCG limit is only applicable to cleavage fracture. So it can be interpreted instead that the $K_{JC(1T)(limit)}$ should be used since there was no cleavage, even though it is probably true that the $K_{Jc(1T)(\Delta a}$ level was exceeded prior to the $K_{JC(1T)(limit)}$. ASTM E1921 needs better clarity as to how the censoring should be performed for these non-cleavage tests.

Determination of T_0 can be calculated from the data listed in Table 1 using the two different censoring approaches for the non-cleavage tests. T_0 = 13.2°C for the case when the censoring uses the $K_{JC(1T)(limit)}$, and T_0 = 17.5°C for the case when the censoring uses the $K_{Jc(1T)(\Delta a)}$ limit. Of the fifty tests meeting the ASTM E1921 testing requirements, thirty-three tests were considered as valid and seventeen had to be censored. The two calculated values of T_0 are within 4.3°C of each other and are less than 5°C lower than the ORNL recalculated value from all of the larger specimens. The 2σ uncertainty for the larger specimen tests is around 10°C and is slightly greater for the mini-C(T) tests. The mini-C(T) specimen testing produces T_0 results within the range of expected results based on all the larger specimen tests. If a much smaller number of test results are available, the difference between the two methods of censoring could be larger than 4.3°C due to fewer valid tests and less confidence in the somewhat arbitrary value of $K_{Jc(1T)(\Delta a)}$.

Figures 2 and 3 are plots of the mini-C(T) $K_{Jc(1T)}$ irradiated values and the two censoring methods as compared to the median Master Curve as well as the 2%, 5%, and 95% confidence/tolerance bounds. Four of the $K_{Jc(1T)}$ results at -10°C fall just below the 2% bound which is slightly more than expected. Due to these four values falling outside the 2% bound, the mini-C(T) data in Table 1 were also analyzed using bimodal and multi-modal [10,11] analyses, which are being considered for guidance in ASTM E1921. The bimodal analysis indicated that the material could not be determined to be heterogeneous while the multi-modal analysis indicated possible heterogeneous behavior. The resultant values of T_m (the mean reference temperature for all populations within the data) from the multi-modal analyses were 19.0°C and 21.6°C for non-cleavage censoring using $K_{Jc(1T)(limit)}$ and $K_{Jc(1T)(\Delta a)}$, respectively. These values are only 4-5°C higher than the valid T_0 results of 13.2°C and 17.5°C, again respectively for non-cleavage censoring using $K_{Jc(1T)(limit)}$ and $K_{Jc(1T)(\Delta a)}$. The large number of censored data could be the reason for the multi-modal indication of possible heterogeneous behavior.

As indicated earlier, three different blocks of the Midland WF-70 beltline weld metal were used to produce the CVN and precracked Charpy specimens that were later machined into the mini-C(T) specimens. In Table 1, the number just after MW in the specimen code is the block number; i.e., 9, 11, and 15. A T_0 evaluation of the mini-C(T) data from block 9 produced values of 20.4°C and 28.6°C for non-cleavage censoring using $K_{Jc(1T)(limit)}$ and $K_{Jc(1T)(\Delta a)}$, respectively; from block 15, T_0 values are 13.1°C and 18.0°C for non-cleavage censoring using $K_{Jc(1T)(limit)}$ and $K_{Jc(1T)(\Delta a)}$, respectively. Block 11 did not have enough non-censored values to determine a valid measure of T_0. These T_0 results between blocks 9 and 15 are within the 10°C uncertainty range of mini-C(T) data and in good agreement with the re-analyzed ORNL larger specimen data (18°C) regardless of how the non-cleavage censoring is performed.

Table 1. Mini-C(T) Test Results from Four Laboratories

Laboratory	Specimen Code	Temperature (°C)	Valid $K_{Jc(1T)}$ (MPa√m)	Possible Censored $K_{Jc(1T)}$ (MPa√m)		Comments
				$K_{Jc(1T)(\Delta a)}$	$KJc_{(1T)limit}$	
	MW9 AI2 A2	-5	67	--	--	
	MW9 AI2 B1	-5	66	--	--	
	MW9 AI2 B2	-5	58	--	--	
	MW11 AA2 A1	5	--	80	94	No cleavage
	MW11 AA2 A2	5	--	80	95	No cleavage
	MW11 BB1 A1	5	--	80	--	Violated DCG
A	MW11 BB1 A2	-5	65	--	--	
	MW11 BB1 B2	-5	--	80	--	Violated DCG
	MW15 AE3 A1	-5	62	--	--	
	MW15 AE4 A1	-5	--	80	--	Violated DCG
	MW15 DE1 A2	-5	45	--	--	
	MW15 DE1 B1	-5	48	--	--	
	MW15 DE1 B2	-5	54	--	--	
	MW9AI2 A1	-5	47	--	--	
	MW9EE1 A1	-5	49	--	--	
	MW15DE2 A2	-5	53	--	--	
	MW9EE1 A2	5	58	--	--	
	MW9EE1 B1	-5	57	--	--	
	MW11AB2 A2	-15	--	80	--	Violated DCG
B	MW11AB2 B1	-15	--	80	--	Violated DCG
	MW11AB2 B2	-25	--	80	--	Violated DCG
	MW15AE3 A2	-25	--	80	--	Violated DCG
	MW11AA2 B2	-5	--	80	--	Violated DCG
	MW15AE4 A2	-5	--	80	--	Violated DCG
	MW11AA2 B1	0	--	80	--	Violated DCG
	MW9EE1 B2	5	--	80	--	Violated DCG
	MW15AE4-B1	-10	46	--	--	
	MW15AK3-B1	-10	--	80	--	Violated DCG
	MW15AK3-A2	-10	45	--	--	
	MW9A14-B1	-10	65	--	--	
	MW9A14-A1	-10	80	--	--	Max. Valid $K_{Jc(1T)}$
C	MW15AK3-A1	-10	40	--	--	Pop-in
	MW15DE2-B2	-10	--	80	--	Violated DCG
	MW9A14-A2	-10	56	--	--	
	MW15AE3-B1	-10	--	80	--	Violated DCG
	MW9A1-B1	-10	60	--	--	
	MW15AK3-B2	-10	--	80	99	No cleavage
	MW 11CE2 A1	-10	41	--	--	
	MW 15AE4 B2	-10	54	--	--	
	MW 9EE3 B1	-10	40	--	--	
	MW 11CE2 A2	-10	43	--	--	
	MW 9AI1 A2	-10	68	--	--	
	MW 11CE2 B1	-10	36	--	--	
D	MW 9EE3 A2	-10	38	--	--	
	MW 9AI1 B1	-10	--	80	98	No cleavage
	MW 9AI1 B2	-5	--	80	97	No cleavage
	MW 9AI5 B2	-5	74	--	--	
	MW 9AI1 A1	-5	--	80	98	No cleavage
	MW 9EE3 B2	-5	54	--	--	
	MW 9EE3 A1	-5	54	--	--	

This evaluation of the data from the four laboratories provides insight into testing of mini-C(T) specimens of a LUS material. When the upper shelf has lower toughness and the mini-C(T) specimen ductile initiation toughness begins to interact with the cleavage initiation toughness near and under the 100 MPa√m level associated with the value of T_0, the determination of T_0 gets to be more complicated than for higher toughness steels. More test specimens are needed due to the higher number of tests exhibiting DCG. Selection of the test temperature(s) is very important since there is a small range of temperatures where valid data can be generated using mini-C(T) specimens. If the ORNL larger specimen results had not been available so that the expected value of T_0 was known, even more specimens may have had to be tested to determine T_0 in this limited temperature range. ASTM E1921 should consider more guidance in dealing with LUS materials in terms of the number of specimens needed and the proper way to perform data censoring for test specimens that do not exhibit cleavage.

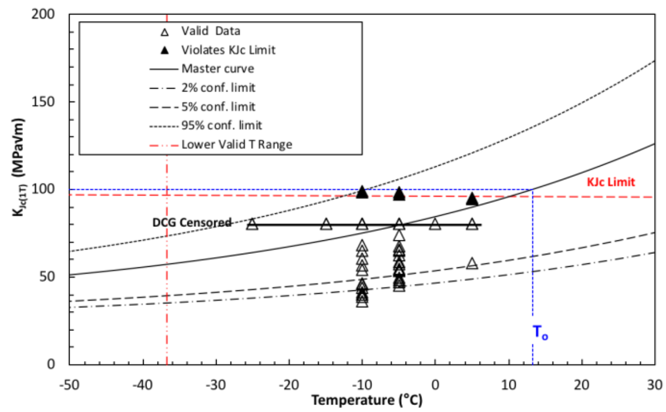

Figure 2. Mini-C(T) Data Compared with the Master Curve and Confidence Bounds Using Censoring of Non-cleavage Results Using $K_{Jc(1T)(limit)}$; T_0 = 13.2°C

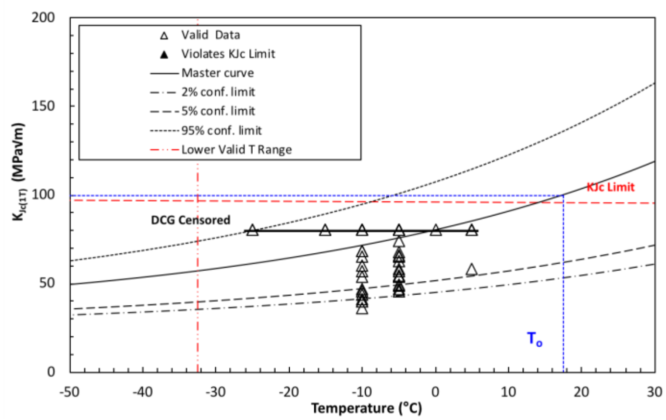

Figure 3. Mini-C(T) Data Compared with the Master Curve and Confidence Bounds Using Censoring of Non-cleavage Results Using $K_{Jc(1T)(\Delta a)}$; T_0 = 17.5°C

CONCLUSIONS

An irradiated LUS WF-70 Linde 80 weld metal has been tested by four laboratories as part of an assessment of use of the mini-C(T) test specimen for Master Curve fracture toughness evaluation following ASTM E1921. The results from each of the laboratories were combined to assess the validity and use of the mini-C(T) specimen for an irradiated reactor pressure vessel material that can exhibit ductile crack growth at relatively low toughness levels interacting with cleavage initiation fracture toughness. The results from this mini-C(T) testing also were compared to extensive larger specimen test results from fracture toughness specimens of the same irradiated material.

Comparisons of the results from the laboratories and some inter-laboratory differences in the fracture testing have been assessed. The evaluations have indicated good agreement between the mini-C(T) and larger specimen results, but the selection of test temperature and the number of test specimens needed to obtain reliable results is more restrictive when testing a low-upper-shelf toughness material.

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