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# **USE OF MINI-CT SPECIMENS FOR FRACTURE TOUGHNESS CHARACTERIZATION OF LOW UPPER-SHELF LINDE 80 WELD BEFORE AND AFTER IRRADIATION<sup>1</sup>**

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## **ABSTRACT**

Mini-CT specimens are becoming highly anticipated geometry for use in reactor pressure vessel (RPV) community for direct measurement of fracture toughness in transition region using the Master Curve methodology. In the present study, Mini-CT specimens were machined from previously tested Charpy specimens of Midland low upper-shelf Linde 80 weld in both, unirradiated and irradiated condition. The irradiated specimens have been characterized as part of joint ORNL-EPRI-CRIEPI collaborative program. The Linde 80 weld was selected based on the facts that it has been previously extensively characterized in the irradiated condition by conventional specimens and because of the awareness to validate application of Mini-Ct specimens for material with lower upper shelf which is more likely will be the case for irradiated material of older generation of RPVs. It is shown that the reference transition fracture toughness temperatures,  $T_o$ , derived from these Mini-CT specimens are in good agreement with  $T_o$  values previously recorded for this material in the unirradiated and irradiated conditions. However, this study indicates that in real practice it is highly advisable to use a much larger number of specimens than the minimum amount prescribed in ASTM E1921.

## **INTRODUCTION**

Any fracture toughness specimen that can be made out of the broken halves of standard Charpy specimens may have exceptional utility for evaluation of reactor pressure vessels since it would allow one to determine and monitor directly actual fracture toughness instead of requiring indirect

predictions using correlations established with impact data. The Charpy V-notch specimen is the most commonly used specimen geometry in surveillance programs and most likely to be used in advanced reactors as per ASME code. The advantage of the Mini-CT specimen technique is coming from the same cross-section (10x10 mm) as standard Charpy specimen such that it can be made from the simple slice of a broken half of Charpy specimen, used in a standard surveillance capsule of a reactor pressure vessel (RPV). On the other side, the thickness of this Mini-CT (slightly below 5 mm) is enough to fit in very narrow validity limit window allowed by ASTM E1921 standard. Up to now, most of the work on validation of this type of specimen has been performed on base metal and only recently limited work has been performed on weld metals in the unirradiated condition (1-9). In this study, Mini-CT specimens were used to perform fracture toughness characterization of low upper-shelf Linde 80 weld, designated WF-70. This weld was utilized in the Midland Reactor Unit 1 beltline weld and has been previously well characterized at the Oak Ridge National Laboratory (ORNL) with various conventional fracture toughness specimens (10-14). The unirradiated broken Charpy specimens were machined into Mini-CT specimens at ORNL and preliminary results has been previously reported (9). The irradiated broken Charpy specimen were retrieved from the storage and machined into Mini-CT specimens as part of joint ORNL-EPRI-CRIEPI collaborative project. The Mini-CT were machined and distributed for testing to different laboratories. The details of this joint program can be found in (15). This paper describes results of fracture toughness characterization of these specimens at the ORNL.

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## MATERIAL DESCRIPTION

In the 1990's, the Heavy Section Steel Irradiation Program at the ORNL performed a very wide-ranging characterization program of the beltline and nozzle course welds from Midland Nuclear Power Plant Unit 1 in the unirradiated and irradiated conditions. The Unit 1 had been canceled and large pieces of various parts of the beltline and nozzle course welds from the RPV were removed and used for that study. The current study deals with the unirradiated and irradiated beltline weld of this reactor which was a double-V submerged-arc (WF-70) weld made with Heat No. 72105 weld wire and lot 8669 Linde-80 flux.

The goal of that original program was to perform a very comprehensive characterization of chemical composition, Charpy impact toughness, drop weight nil-ductility, tensile, and fracture toughness properties of the beltline and nozzle course welds before and after irradiation in test reactors. For example, a total of 230 Charpy specimens were tested for impact toughness characterization of the beltline weld in the unirradiated condition. Fourteen 0.5T, thirty-five 1T, fourteen 2T, and two 4T compact tension and 19 precracked Charpy specimens were used to perform transition region fracture toughness characterization of the beltline weld in the unirradiated condition, while twenty four 1T, seventeen 0.5T and 25 precracked Charpy specimens were tested in the irradiated condition. Based on the results of such a large number of conventional fracture toughness specimens, the reference transition fracture toughness temperature,  $T_o$ , was determined to be -54°C for the unirradiated and 24°C for irradiated beltline weld. The irradiation has been performed at the University of Michigan Ford reactor at 288°C to  $1.0 \times 10^{19}$  n/cm<sup>2</sup> ( $E > 1$  MeV). The detailed results of this program can be found in these publications (10-14). It needs to be pointed out that the main goal of that original program was to investigate potential variability of chemical composition and  $RT_{NDT}$  in a low upper-shelf weld from actual reactor vessel. The Master Curve methodology was still in development stage at that time and was considered as an alternative method for fracture toughness characterization of RPV materials.

The availability of such an ample data bank of the irradiated properties by means of conventional fracture toughness specimens was one of the reasons for selecting this beltline material to perform validation of Mini-CT specimens for fracture toughness characterization of a lower upper-shelf weld material for a joint ORNL-EPRI-CRIEPI program.

## TEST RESULTS

A slightly modified version of Mini-CT specimen is being used at ORNL (9). The main modification is related to use of grooves to allow an "outboard" clip gage with sharp razor blades to be placed such that load-line displacement can be directly measured for J-integral calculation rather than front face gage placement to avoid subsequent recalculation to load-line displacement. Moreover, from previous experience at the fracture mechanics laboratory at ORNL, it was determined that

razor blades improved reliability and sensitivity as compared to integrated front-face cut-off notches like the one suggested in CRIEPI-run round robin specimen design (4-7). Another advantage of this set-up is simplicity of handling such a small specimen and clip gage in the hot cell or other remote conditions. All specimens were fatigue precracked to the target  $a/W$  value of 0.5

The testing was performed under carefully controlled conditions in accordance with ASTM E1921 such that the values can be compared to the fracture toughness performance of previously tested large specimens. The analysis of unirradiated Mini-CT data (9) yield the  $T_o$  value of -53°C which is in very good agreement with previously reported  $T_o$  of -54°C, derived from substantial number of larger fracture toughness specimens. The irradiated Mini-CT were precracked and tested on the same equipment, following the same procedure as unirradiated specimens. Total of 15 Mini-CT specimens have been tested in the irradiated condition. All tests were performed at -10°C. Out of 15 specimens, only one specimen did not cleave. This test was stopped after specimen developed substantial stable crack growth and final J value well in excess of  $K_{Jc \text{ (limit)}}$ . All other specimens cleaved and all measured  $K_{Jc}$  values were within the validity limit of E1921 as per equation:

$$K_{Jc \text{ (limit)}} = \sqrt{(E(W-a_o)\sigma_{ys}/30(1-\nu^2))} \quad (1)$$

In equation 1,  $W$  is the width of the specimen,  $a_o$  is the initial crack length, and  $\nu$  is Poisson's ratio. Also,  $E$  is the Young's modulus determined using the following equation from ASTM E1921-17:

$$E = 204 - T/16, \text{ GPa} \quad (2)$$

while  $\sigma_{ys}$  is the yield strength determined using the following equation from ASTM E1921-17:

$$\sigma_{ys} = \sigma_{ysRT} + 10^5/(491 + 1.8T) - 189, \text{ MPa} \quad (3)$$

where  $T$  is the test temperature, °C, and  $\sigma_{ysRT}$  is the yield strength at room temperature in the irradiated condition as reported in (12). Test results are summarized in Table 1.

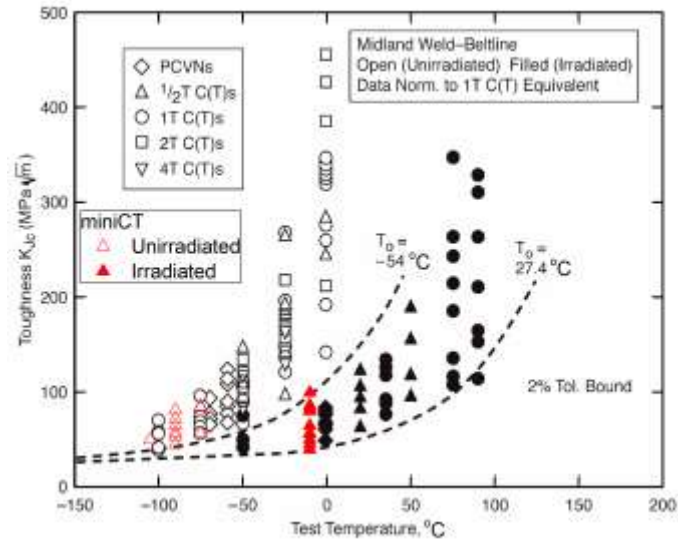
**Table 1. Fracture toughness of irradiated Midland beltline weld as measured by Mini-CT specimens**

ID	$K_{Jc}$	$K_{Jc \text{ (limit)}}$	$a_o$	$\Delta a$	Note
	MPa√m	MPa√m	mm	mm	
9AI4-B2	123.65	155.56	3.44	0.33	$a_o < 0.45W$
15AE4-B1	67.62	138.74	4.41	none	
9AI5-A1	68.19	143.48	4.17	none	8.9.1
15AK3-B1	124.56	142.82	4.18	0.24	$K_{Jc \Delta a}$
15DE2-B1	119.27	143.99	4.14	0.20	
15AK3-A2	59.77	148.73	3.83	none	
9AI4-B1	90.56	147.73	3.89	none	
9AI5-A2	119.48	155.56	3.41	none	$a_o < 0.45W$
9AI4-A1	114.39	141.91	4.21	0.14	
15AK3-A1	50.72	146.55	3.96	none	Pop-in
15DE2-B2	145.48	146.21	4.01	0.27	$K_{Jc \Delta a}$
9AI4-A2	77.11	148.39	3.88	none	
15AE3-B1	115.73	145.87	4.00	0.22	
9AI5-B1	78.31	146.55	4.17	none	
15AK3-B2	N/A	144.70	4.07	N/A	No cleave

Post-test examination of the fracture surfaces revealed that two specimens, 9AI4-B2 and 9AI5-A2, were precracked to a crack length  $a_0 < 0.45W$ , which falls below limit prescribed in section 7.1 of ASTM E1921 Standard. These two specimens data can not be used in calculation in order to obtain valid  $T_0$  value as per section 10.5 of ASTM E1921-17. For specimen 9AI5-A1, one crack measurement on a side of the specimen was differ from the average crack length measurement by more than 0.5 mm, thus violating the qualification of data requirement 8.9.1 in ASTM E1921-17 standard.

Few specimens cleaved after some amount of stable crack growth and column “ $\Delta a$ ” lists the longest crack extension among nine measurements for a given specimen. Out of these few, two specimens, 15AK3-B1 and 15DE2-B2, exhibited stable crack growth extension prior to cleavage in excess of  $0.05(W-a_0)$ , which violates  $K_{Jc \Delta a}$  limit as per section 8.9.2 in ASTM E1921-17 standard. Two more specimens barely met the  $K_{Jc \Delta a}$  requirement. One specimen exhibited pop-in just past linear part of load-displacement trace.

Figure 1 illustrates 1-T adjusted fracture toughness data of Midland beltline weld WF-70 from the present study in the irradiated condition and data in the unirradiated condition from (9) as derived using mini-CT specimens. These results (red color) are superimposed on large database of fracture toughness of Midland WF-70 weld that been previously produced at ORNL by testing large number of conventional specimens in both, unirradiated and irradiated, conditions (12).



**Figure 1. Fracture toughness data of unirradiated and irradiated Midland beltline weld from the present study using Mini-CT specimens and conventional specimens in (12)**

### CENSORING AND $T_0$ DETERMINATION

To summarize the test results in the previous chapter, out 15 specimens tested, three specimens produced invalid data, three specimens violated some requirements and needed to be censored for  $T_0$  determination, and nine specimens cleaved with

valid, uncensored  $K_{Jc}$  values – thus satisfying requirement for size of the data set as per 10.3 in ASTM E1921-17. Thus, next step was to perform censoring.

For specimen 15AK3-B2, test was terminated prior to the cleavage with final  $K_J$  value well above  $K_{Jc \text{ (limit)}}$  for this specimen. The ASTM E1921-17 prescribes to use this specimen in  $T_0$  determination by censoring its value with  $K_{Jc \text{ (limit)}}$  as in the Table 1.

Another censoring is prescribed for two specimens that violated  $K_{Jc \Delta a}$  limit. The section 10.2.1 prescribes to use the highest uncensored  $K_{Jc}$  value in this data set as censored value for this violation. In this data set that value is  $119.27 \text{ MPa}\sqrt{\text{m}}$ , see Table 1. Now all uncensored and censored data need to be converted to 1T equivalence and  $T_0$  value can be determined. Following these steps yields  $T_0$  value of  $14^{\circ}\text{C}$ . However, section 10.2.1 gives permission for use  $K_{Jlc}$  value, if available, as censoring value for specimens that violated  $K_{Jc \Delta a}$  limit as well: “The  $K_{Jlc}$  value defined in E1820 can also be used for  $K_{Jc \Delta a}$ , if  $J_{lc}$  is known for the test material”. In the present study, all tests were performed following E1921 procedure and  $J_{lc}$  was not determined. However previous ORNL data (12) reported  $J_{lc}$  value in the irradiated condition at  $150^{\circ}\text{C}$ . It was derived by testing 1T C(T) specimens and it converts to  $K_{Jlc} = 163 \text{ MPa}\sqrt{\text{m}}$ . This value exceeds the  $K_{Jc \text{ (limit)}}$  value and as per section 8.9.2 and 10.2.1 of ASTM E1921-17 standard, the  $K_{Jc \text{ (limit)}}$  value shall be used for censoring purposes (the lower of the two limits). By following this option,  $T_0$  is determined to be  $8^{\circ}\text{C}$ .

### DISCUSSION

As it has been pointed out in the preliminary study with the unirradiated weld specimen (9), the low upper-shelf material may present additional challenge while using these small Mini-CT specimens. The present study with irradiated material confirmed this observation. Indeed, out of available 15 specimens, three specimens did not satisfy validity requirements of E1921. Out of remaining 12 specimens, three specimens violated various  $K_{Jc}$  capacity limits. All these three violations are related to stable crack growth one way or another. It needs to point out that two more specimens are almost exceeded amount stable crack growth prior to the cleavage. As reminder, this was not a blind test case. The whole idea of selecting this material was that large data base is already existed.

Yet, the  $T_0$  values derived from a relatively small number of Mini-CT specimens in this study and (9) are in remarkable agreement with the  $T_0$  values previously reported for a much larger number of conventional fracture toughness specimens. Visual comparison the present Mini-CT and previous conventional specimens data in Figure 1 show very good overall agreement between Mini-CT and conventional specimens data. This is especially the case for the unirradiated data when the difference between previously reported  $T_0$  value ( $-54^{\circ}\text{C}$ , see Fig. 1) and  $T_0$  from Mini-CT ( $-53^{\circ}\text{C}$ , Ref. 9) is only  $1^{\circ}\text{C}$ .

The irradiated data require more detailed consideration though. The previously reported  $T_o$  value from conventional large specimens is 27°C (12). As it was mentioned above, the  $T_o$  from this study for irradiated Mini-CT specimens is either 14°C or 8°C depending on the censoring procedure. While section 10.2.1 does not prescribe the preferred scheme of censoring in the case of violating  $K_{Jc\Delta a}$  limit (the highest uncensored  $K_{Jc}$  or  $K_{Jlc}$ ), it is believed by author that the language of the standard is aimed to select the lowest available value for censoring purpose when several limits are violated. Once again, based on the testing of the irradiated Mini-CT specimens in this study and the unirradiated Mini-CT in (9), it appears that the optimal temperature window for testing of these small specimens is 25-30°C below expected  $T_o$  value.

## SUMMARY

The ability of a small number of Mini-CT specimens to determine the fracture toughness reference temperature,  $T_o$ , of a low upper-shelf material has been examined on the unirradiated and irradiated Linde 80 WF-70 weld from Midland RPV Beltline weld. These Mini-CT specimens were machined from broken halves of previously tested Charpy specimens as part of the Midland beltline weld characterization. The  $T_o$  values derived from a relatively small number of Mini-CT specimens in this study is in very good agreement with the  $T_o$  values previously reported for a much larger number of conventional fracture toughness specimens. At the same time, this study indicates that in real practice it is highly advisable to use a much larger number of specimens than the minimum amount prescribed in ASTM E1921. The selection of censoring criteria in the case of violating  $K_{Jc\Delta a}$  limit in ASTM E1921 needs better clarification in the standard

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## REFERENCES

1. T. Tobita, Y. Nishiyama, T. Ohtsu, M. Udagawa, J. Katsuyama, and K. Onizawa, "Fracture Toughness Evaluation of Reactor Pressure Vessel" PVP2013-97897, in Proceedings of the ASME 2013 Pressure Vessels and Piping Conference, 2013.
2. N. Miura and N. Soneda, "Evaluation of Fracture Toughness by Master Curve Approach using Miniature C(T) Specimens," PVP2010-25862, in Proceedings of the ASME 2010 Pressure Vessels and Piping Conference/ K-PVP Conference, 2010.
3. K. Yoshimoto, T. Hirota, H. Sakamoto, T. Sugihara, S. Sakaguchi, and T. Oumaya, "Applicability of Miniature C(T) Specimen to Evaluation of Fracture Toughness for Reactor Pressure Vessel Steel," PVP2013-97840, in Proceedings of the ASME 2013 Pressure Vessels and Piping Conference, 2013.
4. M. Yamamoto, A. Kimura, K. Onizawa, K. Yoshimoto, T. Ogawa, A. Chiba, T. Hirano, T. Sugihara, M. Sugiyama, N. Miura and N. Soneda, "A Round Robin Program of Master Curve Evaluation Using Miniature C(T) Specimens -First Round Robin Test on Uniform Specimens of Reactor Pressure Vessel Material," PVP2012-78661, In Proceedings of the ASME 2012 Pressure Vessels and Piping Conference, 2012.
5. M. Yamamoto, K. Onizawa, K. Yoshimoto, T. Ogawa, Y. Mabuchi, M. Valo, M. Lambrecht, H.W. Viehrig, N. Miura and N. Soneda, "International Round Robin Program on Master Curve Reference Temperature Evaluation Utilizing Miniature C(T) Specimen", ASTM STP 1576, 2014.
6. M. Yamamoto, K. Onizawa, K. Yoshimoto, T. Ogawa, Y. Mabuchi, and N. Miura, "Round Robin Program of Master Curve Evaluation Using Miniature C(T) Specimens - 2nd Report: Fracture Toughness Comparison in Specified Loading Rate Condition," PVP2013-97936, In Proceedings of the ASME 2013 Pressure Vessels and Piping Conference, 2013.
7. M. Yamamoto, A. Kimura, K. Onizawa, K. Yoshimoto, T. Ogawa, Y. Mabuchi, H.W. Viehrig, N. Miura and N. Soneda, "A Round Robin Program of Master Curve Evaluation Using Miniature C(T) Specimens -3rd Report: Comparison of  $T_o$  under Various Selections of Temperature Conditions," PVP2014-28898, In Proceedings of the ASME 2014 Pressure Vessels and Piping Conference, 2014.
8. M. Yamamoto and N. Miura, "Applicability of Miniature C(T) Specimens for the Master Curve Evaluation of RPV Weld Metal," PVP2015-45545, Proceedings of the ASME 2015 Pressure Vessels and Piping Conference, 2015.
9. M.A. Sokolov, "Use of Mini-CT specimens for Fracture Toughness Characterization of Low Upper-Shelf Linde 80 Weld," PVP2017-65904, Proceedings of the ASME 2017 Pressure Vessels and Piping Conference, 2017.
10. R. K. Nanstad, D. E. McCabe, R. L. Swain, M. K. Miller, "Chemical Composition and RTNDT Determinations for Midland Weld WF-70," NUREG/CR-5914 (ORNL-6740), 1992.
11. D. E. McCabe, R. K. Nanstad, S. K. Iskander, R. L. Swain, "Unirradiated Material Properties of Midland Weld WF-70," NUREG/CR-6249 (ORNL/TM-12777), 1994.
12. D. E. McCabe, R. K. Nanstad, S. K. Iskander, D. W. Heatherly, R. L. Swain, "Evaluation of WF-70 weld

metal from the Midland Unit 1 Reactor Vessel,” NUREG/CR-5736 (ORNL/TM-13748), 2000.

13. R. K. Nanstad, D. E. McCabe, and R. L. Swain, “Evaluation of Variability in Material Properties and Chemical Composition for Midland Reactor Weld WF-70,” in 18th International Symposium on Effects of Radiation on Materials, ASTM STP 1325, 1996, pp 125-156.
14. D. E. McCabe, R. K. Nanstad, and M. A. Sokolov, “Effects of Irradiation and Thermal Annealing on

Fracture Toughness of Midland Reactor Weld WF-70,” in 19th International Symposium on Effects of Radiation on Materials, ASTM STP 1366, 1998, pp 306-319

15. W. Server, M.A. Sokolov, M. Yamamoto, and R. Carter, “Inter-laboratory comparisons and preliminary analyses of Mini-C(T) specimen testing of an irradiated Linde 80 Weld metal”, PVP2018-84950, Proceedings of the ASME 2018 Pressure Vessels and Piping Conference, 2018.