Proceedings of the ASME 2018 Pressure Vessels and Piping Conference
PVP2018
July 15-20, 2018, Prague, Czech Republic

PVP2018-85065

MASTER CURVE FRACTURE TOUGHNESS CHARACTERIZATION OF EUROFER97 USING MINIATURE MULTI-NOTCH BEND BAR SPECIMENS FOR FUSION APPLICATIONS[1]

Xiang Chen
Oak Ridge National Laboratory
Oak Ridge, TN, USA

Mikhail A. Sokolov
Oak Ridge National Laboratory
Oak Ridge, TN, USA

Yutai Katoh
Oak Ridge National Laboratory
Oak Ridge, TN, USA

Michael Rieth
Karlsruhe Institute of Technology
Karlsruhe, Baden-Württemberg, Germany

Logan N. Clowers
The University of Tennessee
Knoxville, TN, USA

ABSTRACT

Eurofer97 is one of leading candidates of reduced activation ferritic martensitic (RAFM) steels for first wall structural materials of early demonstration fusion power plants. During fusion plant operation, high neutron irradiation damage on first wall materials can cause irradiation embrittlement and reduce the fracture toughness of RAFM steels. Therefore, it is critical to select proper testing techniques to characterize the fracture toughness of RAFM steels with high fidelity. In this manuscript, we present the feasibility study of using pre-cracked miniature multi-notch bend bar specimens (M4CVN) with a dimension of 45mm (length) x 3.3mm (width) x 1.65mm (thickness) to characterize the transition fracture toughness of Eurofer97 steel based on the ASTM E1921 Master Curve method. The testing yielded a provisional Master Curve reference temperature T_{oQ} of -89°C of unirradiated Eurofer97 steel heat J362A in the normalized and tempered condition. The results are within the normal scatter range of Master Curve reference temperature T_0 for Eurofer97 steel, indicating suitability of applying M4CVN specimens for characterizing the transition fracture toughness of Eurofer97 steel.

INTRODUCTION

Eurofer97 is one of leading candidates of reduced activation ferritic martensitic (RAFM) steels for first wall structural materials of early demonstration fusion power plants. During fusion plant operation, high neutron irradiation damage on first wall materials can cause irradiation embrittlement and reduce the fracture toughness of RAFM steels. Therefore, it is critical to select proper testing techniques to characterize the fracture toughness of RAFM steels with high fidelity. This is especially true for testing neutron irradiated specimens which are usually much smaller than standard size specimens due to limited volume of irradiation facilities and low irradiation dose advantage of small size specimens after irradiation. In this study, we present the feasibility study of using pre-cracked miniature multi-notch bend bar specimens (referred as M4CVN hereafter) with a dimension of 45mm (length) x 3.3mm (width) x 1.65mm (thickness) to characterize the transition fracture toughness of Eurofer97 steel based on the ASTM E1921 Master Curve method [1]. We will present the experimental setup and test procedures for M4CVN specimens. We will also compare our fracture toughness results with literature results obtained from larger size specimens to evaluate if the M4CVN specimens have apparent size effects on Master Curve fracture toughness transition temperature, T_0, of unirradiated Eurofer97.

EXPERIMENTAL

The Eurofer97 material used for testing was provided by Karlsruhe Institute of Technology with a heat number of J362A.

[1] Notice: This manuscript has been authored by UT-Battelle, LLC, under contract DE-AC0500OR22725 with the US Department of Energy. The United States Government retains and the publisher, by accepting the article for publication, acknowledges that the United States Government retains a nonexclusive, paid-up, irrevocable, worldwide license to publish or reproduce the published form of this manuscript, or allow others to do so, for United States Government purposes.

The composition of the material is shown in Table 1. The material was normalized at 1000 °C for half an hour, followed by water quench. Then the material went through a tempering heat treatment at 820 °C followed by air cooling.

Table 1. Chemical composition (wt%) of Eurofer97 heat J362A

Fe	Cr	C	Mn	V	N	W	Ta	Si
Bal.	8.7	0.058	0.022	0.353	0.047	1.07	0.1	0.036

After heat treatment, the Eurofer97 material was machined into M4CVN specimens according to the drawing in Fig. 1 in the L-T orientation. Since some loading portions are shared between neighboring notches, the M4CVN specimen design consumes less material than the standard single notch bend bar specimen and is favorable for more efficient use of irradiation facilities and reducing the specimen dose rate after irradiation.

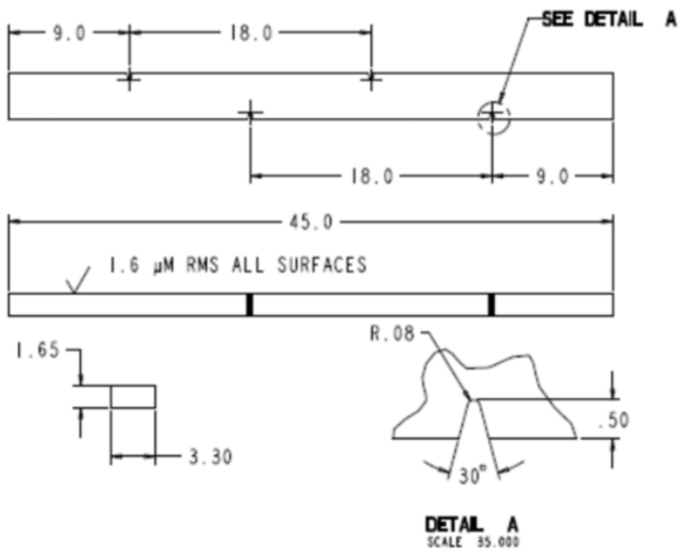


Fig. 1 M4CVN specimen drawing. Unit in millimeter.

The prerequisite of transition fracture toughness testing is having a sharp starting crack in the specimen which is realized by high frequency fatigue precracking in this study. We performed fatigue precracking on M4CVN specimens using a three-point bend type test fixture mounted on a 44.5 kN capacity servo-hydraulic frame as shown in Fig. 2. The span to width ratio of the specimen was kept constant at four. During the fatigue precracking process, the load-line compliance at the machined notch of the specimen was measured by a deflection gauge which yields the real-time crack length using the following equation from Ref. [2]:

$$a/W = 1.0005 - 4.1527U + 9.7477U^2 - 214.2U^3 + 1604.3U^4 - 4633.4U^5 \quad (1)$$
$$U = 1/\{[dE(BB_n)^{1/2}/P]^{1/2} + 1\}$$

where:
a = crack length,
d/P = measured load-line compliance,
E = material Young's modulus,
B_n = specimen net thickness (equals B for non-sidegrooved specimen),
B = specimen thickness.

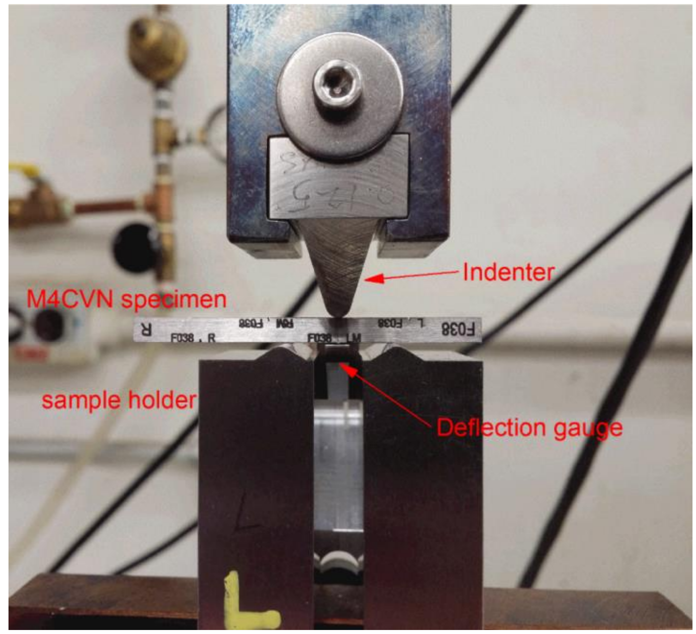


Fig. 2 Fatigue precracking test fixture for a M4CVN specimen

We applied the fatigue precracking procedure in accordance with ASTM E1921 Standard. The standard sets limitations on both the maximum fatigue force (P_m) as well as the allowable maximum stress intensity factor (K_{max}) during the fatigue precracking process. P_m for the M4CVN specimen used in this study is defined per ASTM E1921:

$$P_m = \frac{0.5Bb_0^{\,2}\sigma_Y}{S} \quad (2)$$

where:
B = specimen thickness,
b_0 = length for the initial uncracked ligament,
σ_Y = average of material yield and tensile strengths,
S = span distance.

After substituting the specimen dimensions and typical Eurofer97 yield strength (560 MPa) and tensile strength (670 MPa) into Eq. (2), P_m for the M4CVN specimen is approximately 302.5 N (68 lbs). During fatigue precracking, we applied a constant stress intensity of 12.1 MPa√m which was well below the allowable K_{max} per ASTM E1921. In addition, the maximum fatigue force was also less than P_m. The fatigue precracking frequency was 45 Hz and the minimum to maximum fatigue force ratio, R-ratio, was kept constant at 0.1. In general, it took approximately 260 thousand cycles for completing fatigue precracking of one notch to a crack size to width (a/W) ratio of 0.5.

The test frame used for fracture toughness testing was a 222.4 kN capacity servo-hydraulic frame with a calibrated 4.45 kN capacity load cell. Fig. 3 illustrates the general layout of the experimental setup. We used liquid nitrogen to control testing temperatures which were measured directly from type-T thermocouple wires spot welded to specimens. The environment chamber enclosed specimens and the test setup was used to maintain a relatively stable temperature during testing. As shown in Fig. 4, the M4CVN specimen test setup consists of a specimen indenter and a specimen fixture. The deflection gauge attached to the specimen fixture was used to measure the load-line displacement of the specimen. The push bar can slide left and right and is used to push the specimen against the positioning block such that the specimen notch is aligned with the specimen indenter and the deflection gauge.

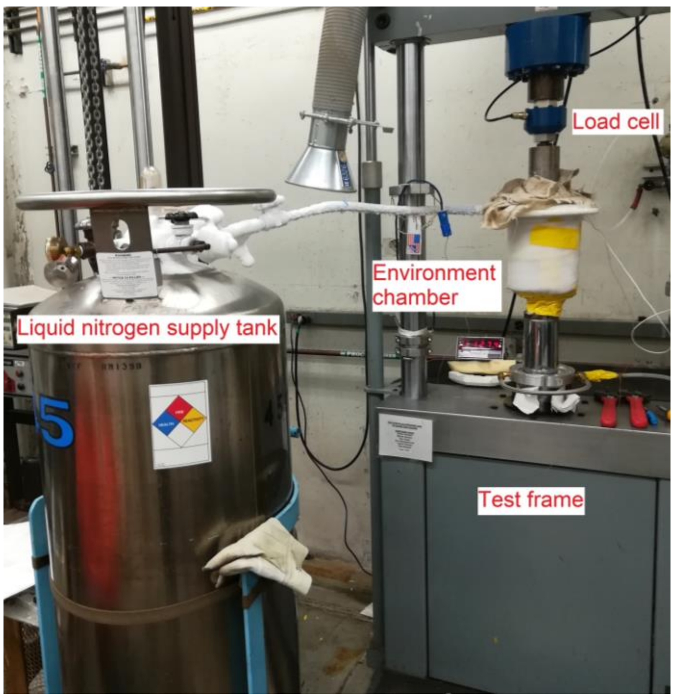
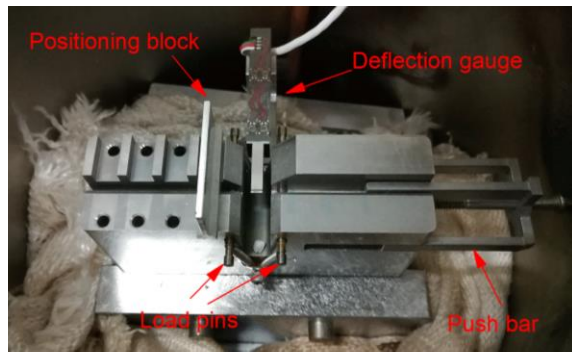


Fig. 3 General layout of the fracture toughness test setup

(a)



(b)

Fig. 4 M4CVN specimen test setup: specimen indenter in (a) and specimen fixture in (b)

We performed fracture toughness testing according to the ASTM E1921 Master Curve method. The test temperatures were selected by balancing between obtaining as high fracture toughness results as possible and still within the fracture toughness capacity limit $K_{Jclimit}$ given in Eq. (3):

$$K_{Jc\,limit} = \sqrt{\frac{E b_o \sigma_{YS}}{30(1-v^2)}} \quad (3)$$

where:
E = material Young's modulus at the test temperature,
b_0 = length for the initial uncracked ligament,
σ_{YS} = material yield strength at the test temperature,
v = Poisson's ratio.

Each specimen notch was tested until cleavage and then the crack length was measured from the fracture surface. The elastic-plastic equivalent stress intensity factor, K_{Jc}, was derived from the J-integral at the onset of cleavage fracture and size-adjusted to 1T value based on the statistical weakest-link theory:

$$K_{Jc(1T)} = 20 + [K_{JC(o)} - 20](\frac{B_0}{B_{1T}})^{1/4} \quad (4)$$

where:
$K_{Jc(1T)}$ = K_{Jc} for a specimen thickness of one inch (B_{1T}=25.4 mm),
$K_{Jc(o)}$ = K_{Jc} for a specimen thickness of B_o (B_o=1.641 mm for M4CVN specimens).

We then calculated the Master Curve provisional reference temperature T_{oQ} using multi-temperature analysis method in Eq. (5) and K_{Jc} data were censored against both the fracture toughness capacity limit $K_{Jclimit}$ and the slow stable crack growth limit $K_{Jc\Delta a}$.

$$\sum_{i=1}^{N} \delta_i \frac{\exp[0.019(T_i - T_{oQ})]}{11.0 + 76.7\exp[0.019(T_i - T_{oQ})]} - \sum_{i=1}^{N} \frac{(K_{Jc(i)} - 20)^4 \exp[0.019(T_i - T_{oQ})]}{\{11.0 + 76.7\exp[0.019(T_i - T_{oQ})]\}^5} = 0 \quad (5)$$

where:
N = number of specimens tested,
T_i = test temperature corresponding to $K_{Jc(i)}$,
$K_{Jc(i)}$ = either a valid K_{Jc} datum or a datum replaced with a censoring value,
δ_i = 1.0 if the datum is valid or zero if the datum is a censored value,
T_{oQ} = Master Curve provisional reference temperature solved by iteration.

RESULTS AND DISCUSSION

The fracture toughness results of Eurofer97 heat J362A in the normalized and tempered condition are summarized in Table 2 and illustrated in Fig. 5. From Eq. (5), we calculated the Master Curve provisional reference temperature, T_{oQ}, as -89 °C. Further, we derived the Master Curve in Fig. 5 using the following equation:

$$K_{Jc(med)} = 30 + 70\exp[0.019(T - T_{oQ})] \quad (6)$$

where:
$K_{Jc(med)}$ = median fracture toughness for a multi-temperature data set from 1T size specimen,
T = test temperature,
T_{oQ} = Master Curve provisional reference temperature.

The tolerance bounds were calculated using the equation below:

$$K_{Jc(0.xx)} = 20 + [\ln(\frac{1}{1 - 0.xx})]^{1/4} \{11 + 77\exp[0.019(T - T_{oQ})]\} \quad (7)$$

where:
0.xx = selected cumulative probability level, e.g., for the 2% tolerance bound, 0.xx=0.02.

Table 2. Fracture toughness of Eurofer97 heat J362A

Specimen ID	Test temperature (°C)	1T-K_{Jc} (MPa√m)	Note	T_{oQ} (°C)
H006L	-165	30.3		-89
H006LM	-154	113.2	censored	
H006RM	-165	41.7		
H006R	-165	46.1		
H002L	-159	44.2		
H002RM	-154	100.5	censored	
H002R	-158	35.7		
H003L	-154	55.2		
H003LM	-154	26.6		
H003RM	-154	42.3		
H003R	-152	55.0		
H004L	-154	56.6		
H004LM	-155	36.7		
H004RM	-155	129.7	censored	
H004R	-148	161.8	censored	

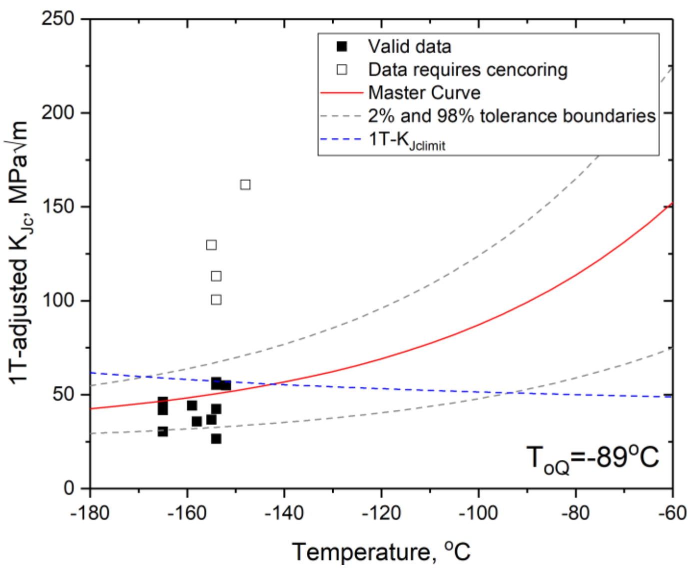


Fig. 5 Master Curve fracture toughness results of Eurofer97 heat J362A

As shown in Fig. 5, many valid data are lower than the median fracture toughness value predicted by the Master Curve although most valid data are still bounded by the 2% and 98% tolerance boundaries of the Master Curve. When the median fracture toughness approached the fracture toughness capacity limit, larger scattering in the fracture toughness results with existence of censored data was observed. Due to the small size of M4CVN specimens, fracture toughness capacity limit $K_{Jclimit}$

of the specimen is very low, which forces the testing in the lower shelf region. This also renders the testing temperatures being more than 50°C lower than the derived T_{oQ}, and hence the T_{oQ} cannot be qualified as T_0 per ASTM E1921.

Despite the fact that we only obtained a provisional Master Curve reference temperature in this study, our T_{oQ} results are within the normal scatter range of Master Curve reference temperature T_0 obtained with larger size specimens for Eurofer97 steel in literature (Table 3). The literature results cover heat 83697, a standard heat for Eurofer97 steel. The Eurofer97 heat (heat J362A) studied in this work, although different from the standard heat, has been reported with similar Charpy impact property as heat 83697 [10]. Therefore, we expect a similar transition fracture toughness property of this heat to the standard heat. The fact that our T_{oQ} results are in line with T_0 results obtained with larger size specimens for Eurofer97 steel proves suitability of applying M4CVN specimens for characterizing the transition fracture toughness of Eurofer97 steel. Indeed, based on our previous work [11-13], we do not anticipate apparent specimen size effects of M4CVN specimens on transition fracture toughness of RAFM steels, such as Eurofer97 and F82H.

Table 3. Eurofer97 Master Curve transition temperature T_0 results in literature

<table>
<tr><th>Specimen type</th><th>Orientation</th><th>Heat #</th><th>T_0 (°C)</th><th>Ref.</th></tr>
<tr><td>PCCVN*</td><td>L-T</td><td rowspan="6">83697</td><td>-121</td><td>[3]</td></tr>
<tr><td>0.35T C(T)**</td><td>Mostly L-T, some T-L</td><td>-76 or -89</td><td>[4-6]</td></tr>
<tr><td>PCCVN</td><td>L-T</td><td>-113</td><td rowspan="3">[7]</td></tr>
<tr><td>KLST***</td><td>L-T</td><td>-99</td></tr>
<tr><td>0.5T C(T)</td><td>T-L</td><td>-129</td></tr>
<tr><td>0.2T C(T) or 0.4T C(T)</td><td>L-T</td><td>-95 or -112</td><td>[8, 9]</td></tr>
</table>

*PCCVN: precracked Charpy v-notch specimen,
**C(T): compact tension specimen; the number in front of “T” is specimen thickness in unit of inch,
***KLST: one type of miniaturized Charpy specimen (3 x 4 x 27 mm).

CONCLUSIONS

In this study, we present the feasibility study of using pre-cracked miniature multi-notch bend bar specimens with a dimension of 45mm (length) x 3.3mm (width) x 1.65mm (thickness) to characterize the transition fracture toughness of Eurofer97 steel based on the ASTM E1921 Master Curve method. The testing yielded a provisional Master Curve reference temperature T_{oQ} of -89°C of unirradiated Eurofer97 steel heat J362A in the normalized and tempered condition. The results are within the normal scatter range of Master Curve reference temperature T_0 for Eurofer97 steel, indicating suitability of applying M4CVN specimens for characterizing the transition fracture toughness of Eurofer97 steel.

ACKNOWLEDGEMENTS

This research was sponsored by the Office of Fusion Energy Sciences, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC. The authors would like to thank Eric Manneschmidt and Ronald Swain for performing the experimental work. The authors are also grateful for Xunxiang Hu and Kevin Field for their technical review of this manuscript.

REFERENCES

1. ASTM E1921-17a: Standard Test Method for Determination of Reference Temperature, T_0, for Ferritic Steels in the Transition Range, ASTM International, West Conshohocken, PA, 2017.
2. J. Underwood et al., ASTM STP 1114 (1991) p197.
3. E. Lucon et al., J. Nucl. Mat., 329-333 (2004) p1078.
4. R. Bonade et al., Eng. Frac. Mech., 75 (2008) p3985.
5. R. Bonade, Constitutive Behavior and Fracture Properties of Tempered Martensitic Steels for Nucl ear Applications: Experiments and Modeling, Ph.D. Thesis, doi:10.5075/epfl-thesis-3405, 2006.
6. R. Bonade et al., J. Nucl. Mat., 367-370 (2007) p581.
7. M. Serrano et al., ASTM STP 1502 (2009) p114.
8. J. Rensman, NRG Irradiation Testing: Report on 300 °C and 60 °C Irradiated RAFM Steels, NRG Petten, 20023/05.68497/P, (2005).
9. E. Gaganidze, Assessment of Fracture Mechanical Experiments on Irradiated EUROFER97 and F82H Specimens, Final Report for Task TW5-TTMS 001-D14, 2007.
10. J. Hoffmann et al., Eurofer Improvement by Optimized Chemical Compositions, The 18th International Conference on Fusion Reactor Materials, Aomori, Japan, 2017.
11. M. Sokolov and H. Tanigawa, DOE/ER-0313/41, Fusion Reactor Materials Program Semiannual Progress Report, December 31, 2006, p83.
12. M. Sokolov and H. Tanigawa, DOE/ER-0313/33, Fusion Reactor Materials Program Semiannual Progress Report, December 31, 2002, p105.
13. X. Chen et al., Transition Fracture Toughness Characterization of Eurofer97 Steel using Pre-Cracked Miniature Multi-notch Bend Bar Specimens, ORNL/LTR-2017/532, November 30, 2017.