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

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ABSTRACT

In this study, we performed fracture toughness testing of ten Eurofer97 steel variants using precracked miniature multi-notch bend bar (M4CVN) specimens based on the Master Curve method in the ASTM E1921 standard. Additional Vickers microhardness and room temperature tensile testing complemented the fracture toughness testing. Compared with standard Eurofer97, the ten variants didn't show a comprehensive improvement of mechanical properties. The Master Curve method was found to yield a reasonable prediction of fracture toughness results obtained from M4CVN specimens with most valid fracture toughness data within the 2% and 98% tolerance boundaries of the Master Curve. The three-parameter Weibull distribution with Weibull exponent b = 4 also yielded excellent prediction of the relationship between fracture toughness results K_{Jc} and the cumulative probability for failure p_f for one steel variant.

Keywords: Eurofer97, Fracture Toughness, Master Curve, Small Specimen Testing Technique, Miniature multi-notch bend bar, Weibull distribution, Fusion

INTRODUCTION

Eurofer97 is the European reference reduced activation ferritic/martensitic (RAFM) steel for the first wall and blanket applications of the DEMO fusion reactor [1-3]. It has favorable properties for fusion applications such as lower radioactivity, superior swelling resistance, and better thermal conductivity [4] and also poses sufficient fracture toughness at the normalized and tempered condition. However, the harsh environment of a fusion reactor, such as neutron irradiation and He/H damage, results in significant degradation of Eurofer97 fracture toughness. Therefore, characterization of fracture toughness property is necessary to ensure long-term safe operation of the fusion reactor. This is especially true for neutron-irradiated specimens which are usually much smaller than standard size specimens due to limited volume of irradiation facilities and advantages with testing small size specimens, such as lower radioactivity and more accurate control of irradiation temperatures. To achieve this goal, we have developed a fracture toughness testing technique using pre-cracked miniature multi-notch bend bar (referred as M4CVN hereafter) specimens based on the Master Curve method in the ASTM E1921 standard [5]. Under the framework of EUROfusion, ten Eurofer97 steel variants were produced to investigate the effects of compositions and heat treatment conditions on Eurofer97 mechanical and microstructural properties as well as materials irradiation behaviors. This paper focuses on the pre-irradiation results on transition fracture toughness of ten Eurofer97 steel variants with complementary results on Vickers microhardness, room temperature yield and tensile strengths.

1. Experimental

Ten Eurofer97 steel variants (code names: H, I, J, K, L, M, N, O, P, and E) were used for testing. The materials compositions and heat treatment conditions (listed in Table 1 and Table 2, respectively) were intentionally varied to study the effects of these two factors on mechanical and microstructural properties. It is worth noting that materials E and M are the standard Eurofer97 heat and the heat treatment condition for material E also represents the standard heat treatment for Eurofer97.

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Table 1 Chemical compositions of Eurofer97 variants (wt%)

	Cr	C	Mn	V	N	W	Ta	Si
H	8.7	.06	.02	.35	.047	1.1	.10	.04
I	8.7	.11	.02	.35	.042	1.1	.09	.04
J	9.0	.11	.39	.22	.022	1.1	.11	<.04
K	7.8	.02	<.03	.22	.022	1.0	.13	<.04
L	9.1	.11	.54	.20	.038	1.1	.12	.03
M	8.8	.11	.53	.20	.019	1.1	.12	.04
N	9.0	.09	.11	<.05	.002	1.0	.09	.04
O	8.8	.06	.50	.30	.070	1.0	.05	.15
P	8.7	.11	.02	.20	.045	1.1	.09	.03
E	8.8	.11	.53	.20	.019	1.1	.12	.04

Table 2. Heat treatment conditions for Eurofer97 variants

H	1000°C/0.5h + WQ + 820°C + AC
I	1000°C/0.5h + WQ + 820°C + AC
J	1250°C/1h + rolling to a final rolling temperature of 850°C in 6 rolling steps with a reduction of 20-30% for each step + AC + 880°C/0.5h + WQ + 750°C/2h + AC
K	1250°C/1h + rolling to a final rolling temperature of 850°C in 6 rolling steps with a reduction of 20-30% for each step + AC + 1050°C/0.25h + WQ + 675°C/1.5h + AC
L	1150°C/0.5h + AQ + 700°C + AC
M	1020°C/0.5h + AQ + 1020°C/0.5h + AQ + 760°C/1.5h + AC
N	920°C/1.5h + AQ + 920°C/1.5h + AQ + 760°C/1h + AC
O	1080°C/1h, cooling to 650°C and rolling to 40% reduction + 760°C/1h + AC
P	1000°C/0.5h + WQ + 820°C + AC
E*	980°C/0.5h + AQ + 760°C + AC

AQ: air quench; WQ: water quench; AC: air cooled
*E had experienced unknown heat treatment prior to the shown heat treatment.

Type SS-J3 miniature tensile specimens with the gauge dimension of 5 (length) x 1.2 (width) x 0.75 (thickness) mm^3 were machined from ten Eurofer97 variants. The head portions of each tensile specimen were used in Vickers microhardness testing prior to tensile testing. The Vickers microhardness measurements were based on the ASTM E384 standard [6] with 1 kgf load and 15 sec dwell time. At least ten measurements were made on each material to obtain the average microhardness value. Afterwards, SS-J3 tensile specimens were used in room temperature tensile testing based on the ASTM E8 standard [7]. The specimen loading direction corresponded to the rolling direction of the raw material. A servohydraulic frame with a calibrated load cell rated for 22.2 kN was used for tensile testing at 10^{-3}/sec strain rate with machine stroke control. At least two specimens per material were tested to yield the average yield and tensile strengths.

M4CVN specimens with a dimension of 45 (length) x 3.3 (width) x 1.65 (thickness) mm^3 were used for Master Curve fracture toughness testing. The specimen was specifically developed from the Oak Ridge National Laboratory (ORNL) Fusion Materials Program and has four notches per specimen. Despite its small size, the M4CVN specimen follows the same size ratio of a bend bar specimen in ASTM E1921. Due to shared loading portions between neighboring notches, the M4CVN specimen consumes significantly less material than the standard single notch bend bar specimen and is favorable for neutron irradiation testing. Each notch on the M4CVN specimen was fatigue precracked to a crack size to width ratio of ~0.5 before testing. In order to obtain the Master Curve reference temperature, at least 14 notches were tested per material. Detailed descriptions for the testing procedures, fixture design, and analysis procedures can be found in [8] and are omitted here for simplicity.

2. Results and Discussion

2.1 Vickers Microhardness and Yield/Tensile Strengths

Vickers microhardness and yield/tensile strengths of ten Eurofer97 variants are shown in Figs. 1 and 2, respectively. The standard Eurofer97 Vickers hardness [9] and yield/tensile strengths [10] are also shown in the same figure for comparison. Most steel variants showed similar Vickers hardness as the standard Eurofer97 except for material H with slightly lower hardness and materials K and L with significantly higher hardness. In tensile testing, most steel variants showed lower yield and tensile strengths than the standard Eurofer97 with the exceptions of materials M and O showing similar strengths and materials K and L showing significantly higher strengths. Comparing Vickers hardness to tensile results shows a close correlation between the two with a linear relationship in Fig. 3.

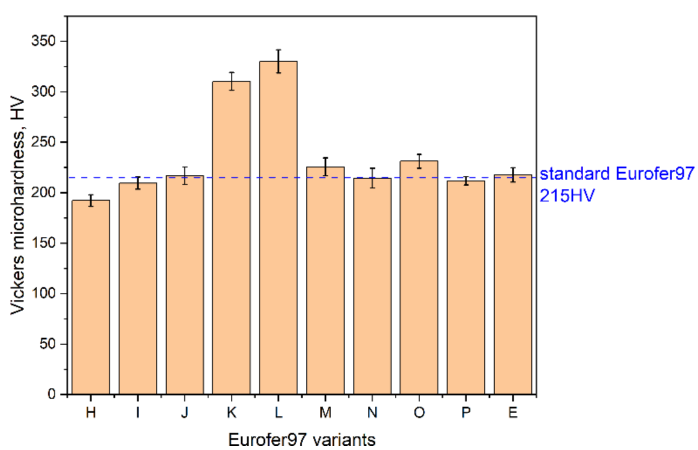


FIGURE 1: Vickers microhardness of ten Eurofer97 variants in comparison with standard Eurofer97. Error bars correspond to ± one standard deviation

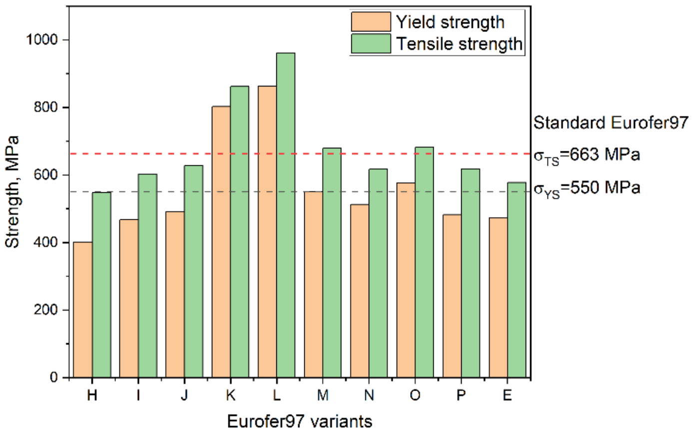


FIGURE 2: Yield and tensile strengths of ten Eurofer97 variants in comparison with standard Eurofer97

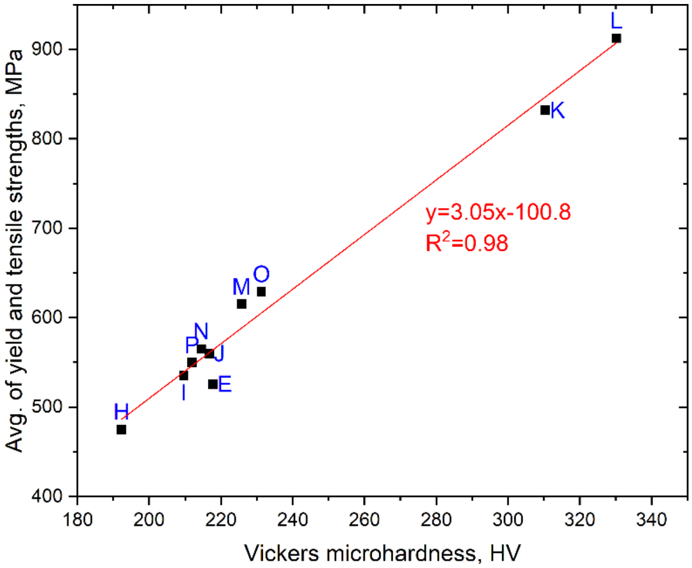


FIGURE 3: Correlation between the Vickers microhardness and tensile results for ten Eurofer97 variants

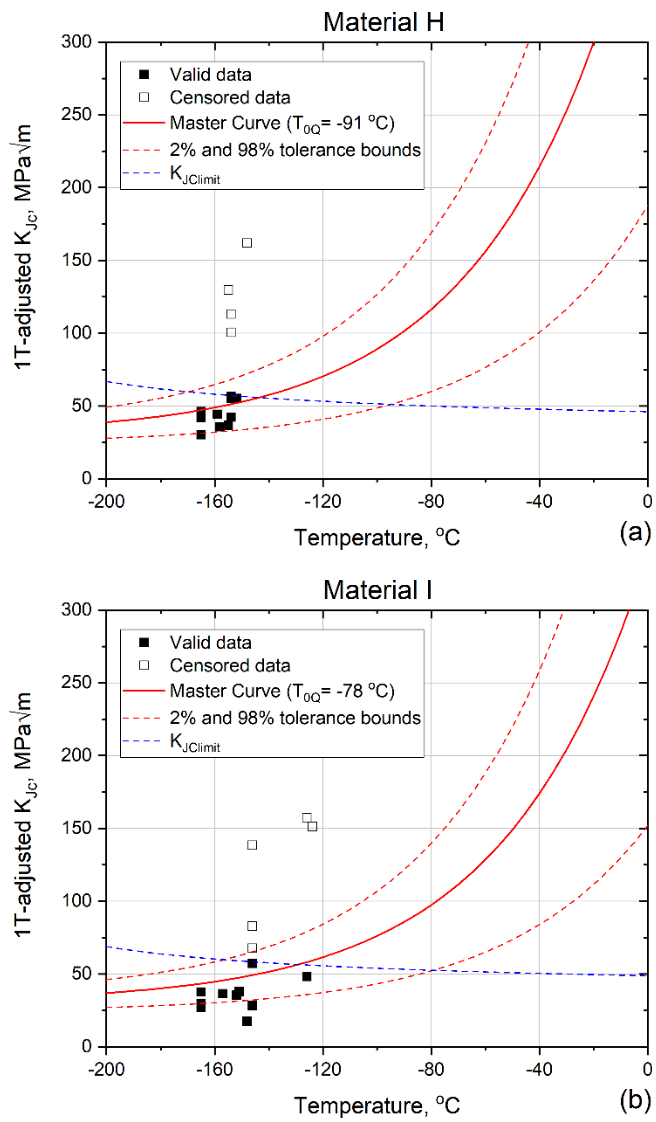
2.2 Fracture Toughness

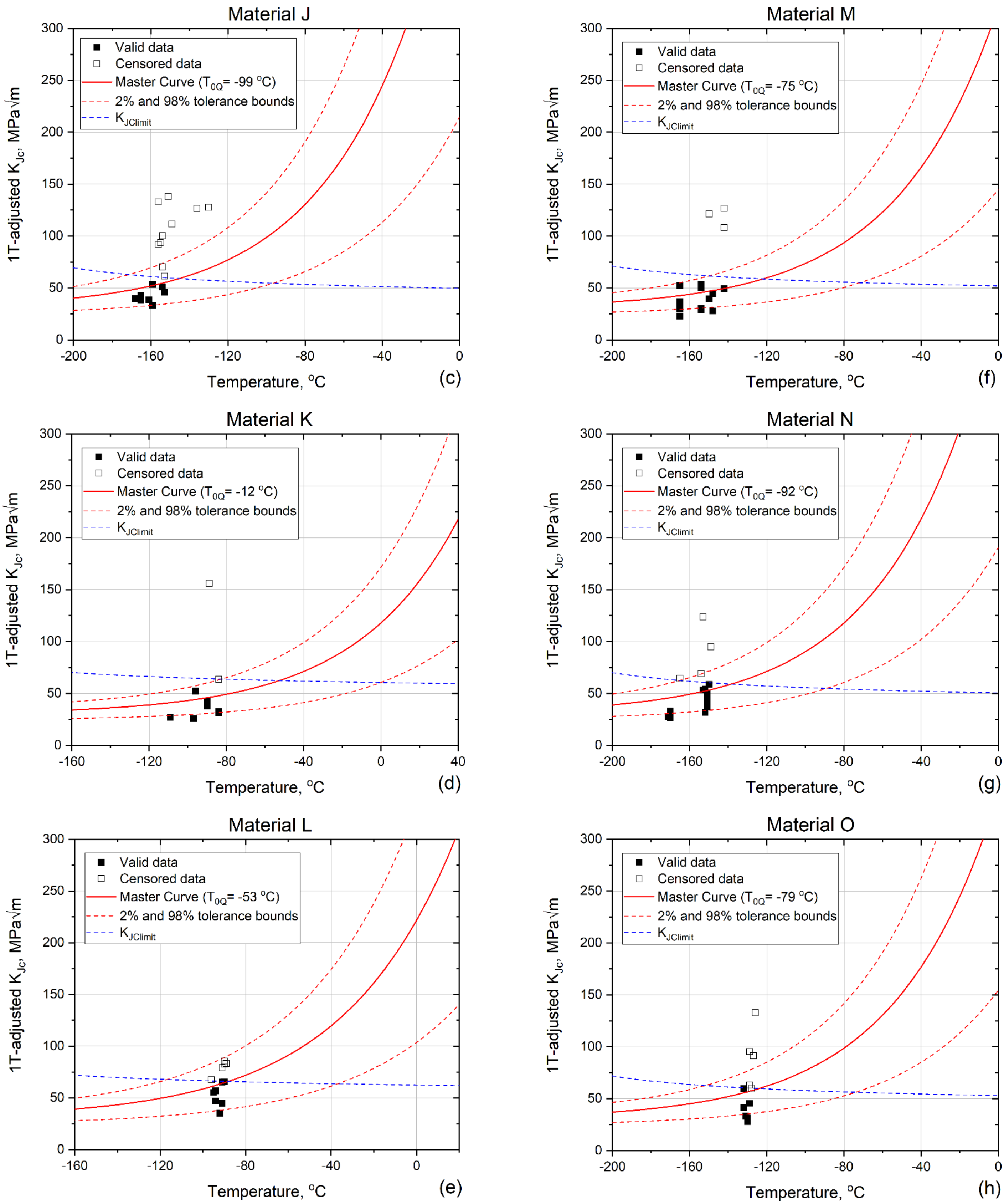
The 1T-adjusted transition fracture toughness results are shown in Figs. 4(a)-(j) for ten Eurofer97 steel variants. Due to the small size of M4CVN specimens, the fracture toughness capacity ($K_{Jclimit}$) given by Eq. (1) was very low.

$$K_{Jclimit} = \sqrt{\frac{E b_0 \sigma_{YS}}{30(1-v^2)}} \quad (1)$$

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.

In order to obtain more fracture toughness results within $K_{Jclimit}$ and avoid generating too many censored data, most testing temperatures were more than 50 °C lower than the derived provisional Master Curve reference temperature, T_{0Q}. Based on the current ASTM E1921 standard, this violated the minimum testing temperature requirement and therefore the provisional value cannot be qualified as Master Curve reference temperature, T_0. Nonetheless, most valid fracture toughness data in Fig. 4 are bounded by the 2% and 98% tolerance boundaries of the Master Curve. Further, the derived T_{0Q} for material H, which was reported with similar Charpy impact property as standard Eurofer97 [11], was within the scatter band range of standard Eurofer97 T_0 obtained from larger size specimens [8]. Indeed, based on our previous studies on RAFM steel F82H, which has similar mechanical properties and microstructure as Eurofer97, we found the Master Curve transition reference temperature determined by the small size bend bar specimens is essentially same as that determined by conventional larger size specimens [12-14].





Material J
Valid data
Censored data
Master Curve (T0Q= -99 °C)
2% and 98% tolerance bounds
KJClimit
1T-adjusted KJc, MPa√m
Temperature, °C
(c)
Material M
Valid data
Censored data
Master Curve (T0Q= -75 °C)
2% and 98% tolerance bounds
KJClimit
1T-adjusted KJc, MPa√m
Temperature, °C
(f)
Material K
Valid data
Censored data
Master Curve (T0Q= -12 °C)
2% and 98% tolerance bounds
KJClimit
1T-adjusted KJc, MPa√m
Temperature, °C
(d)
Material N
Valid data
Censored data
Master Curve (T0Q= -92 °C)
2% and 98% tolerance bounds
KJClimit
1T-adjusted KJc, MPa√m
Temperature, °C
(g)
Material L
Valid data
Censored data
Master Curve (T0Q= -53 °C)
2% and 98% tolerance bounds
KJClimit
1T-adjusted KJc, MPa√m
Temperature, °C
(e)
Material O
Valid data
Censored data
Master Curve (T0Q= -79 °C)
2% and 98% tolerance bounds
KJClimit
1T-adjusted KJc, MPa√m
Temperature, °C
(h)

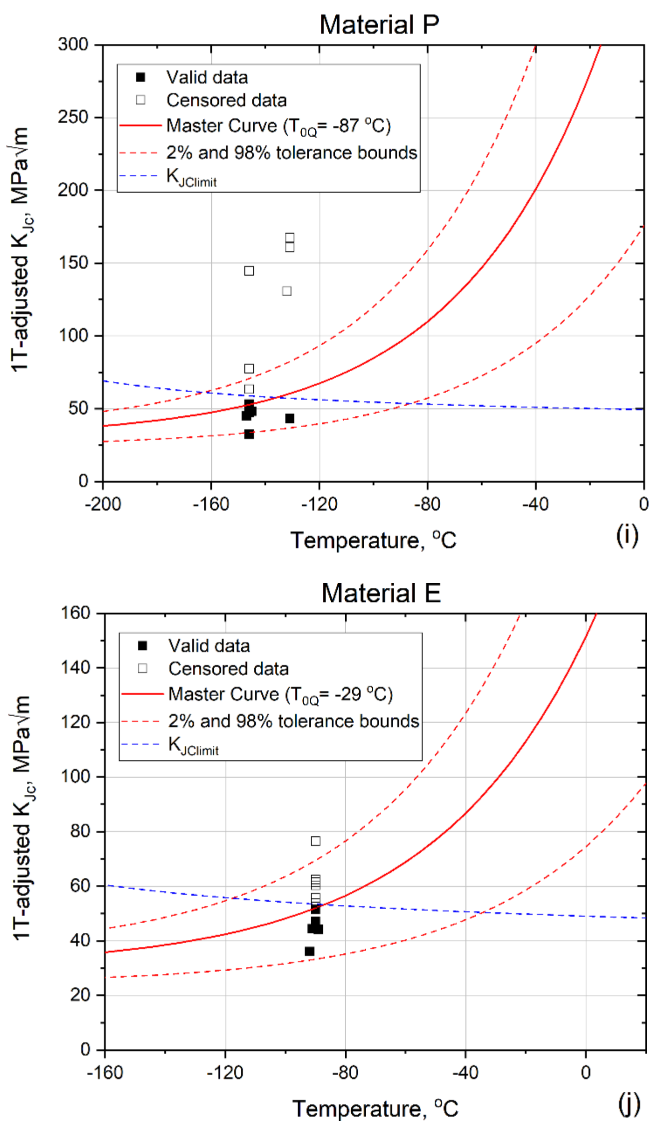


FIGURE 4: Master Curve fracture toughness results for ten Eurofer97 variants

Since the Master Curve method in ASTM E1921 assumes a Weibull distribution of test data, it is justifiable to check how well the three-parameter Weibull model can fit the relationship between fracture toughness results K_{Jc} and the cumulative probability for failure p_f. The fitting should be applied to testing at a single temperature and only material E qualified in this case. Hence, fracture toughness results from material E are evaluated herein. The definition for p_f is the probability for failure at or before K_{Jc} for an arbitrarily chosen specimen from the population of specimens. In Weibull distribution, this can be described by:

$$p_f = 1 - \exp[-(\frac{K_{Jc(i)} - K_{min}}{K_0 - K_{min}})^b] \quad (2)$$

where:
$K_{Jc(i)}$ = 1T-adjusted cleavage fracture toughness,
K_{min} = constant equals 20 MPa√m,
K_0 = scale parameter,
b = Weibull exponent.

Rearranging Eq. (2) yields a linear relationship as following:

$$Y = bX + Y_0 \quad (3)$$

where:
X = $\ln(K_{Jc(i)}-20)$,
Y = $\ln\{\ln[1/(1- p_f)]\}$,
Y_0 = $-b\ln(K_0-20)$.

In order to perform the fitting, fracture toughness results from material E are used to calculate p_f using [15]:

$$p_f = \frac{i-0.3}{N+0.4} \quad (4)$$

where:
i = rank of each measured fracture toughness value,
N = total number of uncensored and censored data.

Further, the scale parameter K_0 is calculated as:

$$K_0 = [\sum_{i=1}^{N} \frac{(K_{Jc(i)}' - 20)^4}{r}]^{1/4} + 20, \text{ MPa}\sqrt{m} \quad (5)$$

where:
$K_{Jc(i)}'$ = 1T-adjusted valid data or 1T-adjusted censored data if the test needs to be censored due to exceeding either $K_{Jclimit}$ or ductile crack growth limit,
r = number of uncensored data.

Then, $K_{Jc(i)}$ and p_f from material E dataset plugged into X and Y in Eq. (3) and the result is shown in Fig. 5. In general, all data points follow a linear correlation as expected. The linear fitting of valid data yielded a slope of 4.02 which matches the Weibull exponent b = 4 for the Master Curve method in ASTM E1921. In addition, the intercept in Fig. 5 also matches the calculated Y_0 in Eq. (3), namely -14.20 vs. -14.22. The results confirm that the three-parameter Weibull distribution with Weibull exponent b = 4 can successfully describe the relationship between fracture toughness results K_{Jc} and the cumulative probability for failure p_f for material E. Therefore, M4CVN specimens tested with the Master Curve method in ASTM E1921 should be suitable for evaluating the fracture toughness for such type of material.

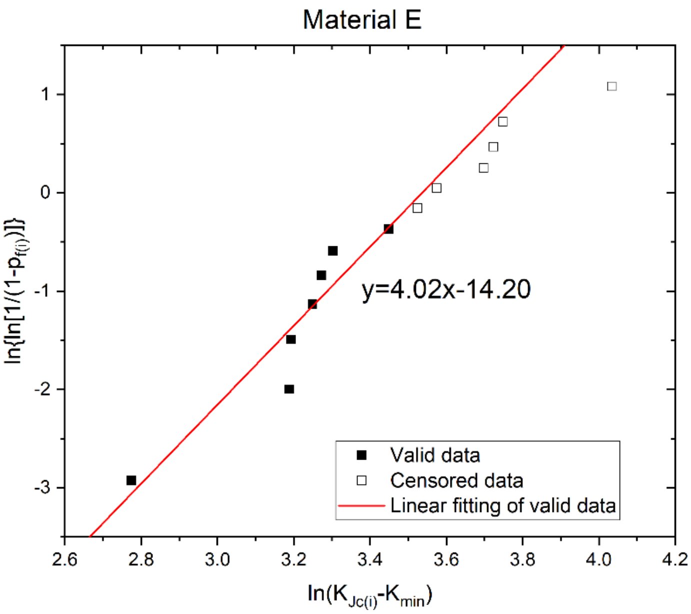


FIGURE 5: Weibull fitting of fracture toughness data for Material E

Lastly, the calculated provisional Master Curve reference temperature, T_{0Q}, is summarized in Fig. 6 with comparison to literature data on standard Eurofer97 reference temperature T_0 [16-22]. Materials K, L, and E showed higher reference temperatures than the standard Eurofer97 whereas other materials had reference temperatures within the upper scatter band of the standard Eurofer97 T_0 range.

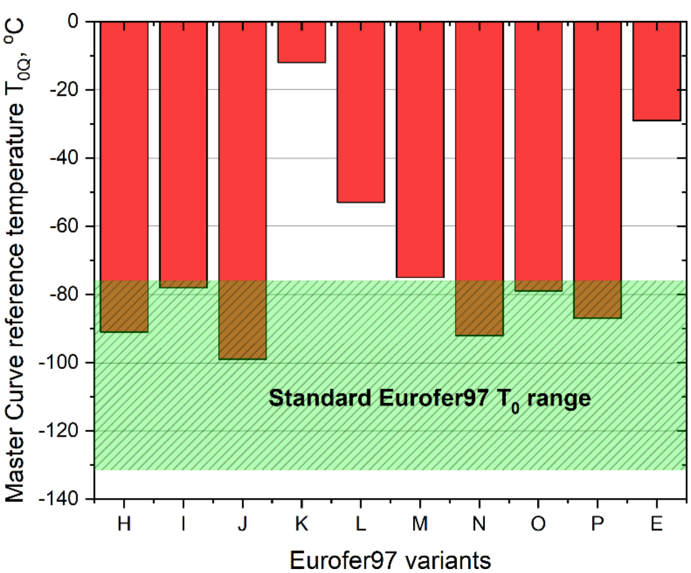


FIGURE 6: Provisional Master Curve reference temperature, T_{0Q}, for ten Eurofer97 variants

CONCLUSIONS

In this study, we present the pre-irradiation results on transition fracture toughness of ten Eurofer97 steel variants with complementary results on Vickers microhardness, room temperature yield and tensile strengths. Compared with standard Eurofer97, the ten variants didn't show a comprehensive improvement of mechanical properties. In particular, materials K and L showed poorer fracture toughness than the standard Eurofer97 despite their higher hardness and strengths. Material E showed worse tensile and fracture toughness properties than the standard Eurofer97. Materials M and O showed similar tensile and fracture toughness properties as the standard Eurofer97. Materials H, I, J, N and P had similar fracture toughness but lower tensile strengths than the standard Eurofer97.

The Master Curve method was found to yield a reasonable prediction of fracture toughness results obtained from M4CVN specimens with most valid fracture toughness data within the 2% and 98% tolerance boundaries of the Master Curve. The three-parameter Weibull distribution with Weibull exponent b = 4 also yielded excellent prediction of the relationship between fracture toughness results K_{Jc} and the cumulative probability for failure, p_f, for one steel variant.

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