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Abstract

The Fraunhofer Institute for Mechanics of Materials (IWM, Freiburg, Germany) and the Materials Testing Institute University of Stuttgart (MPA Stuttgart, Germany) have recently launched a joint collaboration project titled "Dynamic Mastercurves II" (MC-Dyn II). The project focuses on the measurement of the ductile-to-brittle transition toughness at impact loading rates, in part by means of fatigue precracked and side-grooved Charpy-type specimens (PCC specimens). IWM and MPA invited NIST Boulder to contribute to the project by testing PCC specimens of a reactor pressure vessel (RPV) steel and determining the dynamic Master Curve reference temperature, $T_{0,d}$, in accordance with ASTM E1921-14a. Tests at NIST were performed on an instrumented impact machine at 0 °C and with an impact velocity $v_0 \approx 1.21$ m/s. The calculated reference temperature $T_{0,d}$ = -7.5 °C, corresponding to an average loading rate of 3.3×10^5 MPa√m/s, is in excellent agreement with the results previously published by IWM under similar conditions ($T_{0,d}$ = -7 °C). To the author's knowledge, these are the first tests of this type ever performed at NIST Boulder.

Keywords

Dynamic fracture toughness; dynamic reference temperature; impact loading rates; instrumented Charpy tests; Master Curve; precracked Charpy-type (PCC) specimens; reactor pressure vessel steel.

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1. Introduction

The ASTM E1921 standard, titled "*Standard Test Method for Determination of Reference Temperature, T_0, for Ferritic Steels in the Transition Range*," covers the characterization of the fracture toughness of ferritic steels that experience the onset of cleavage (unstable) crack propagation under elastic or elastic-plastic conditions. Its first edition was published in 1997, and the current version is E1921-14a [1].

The E1921 standard allows the determination of the so-called reference temperature T_0, which represents the temperature at which compact tension, C(T), specimens of 1 in. (25.4 mm) thickness, 1TCT, have a median fracture toughness K_{Jc} = 100 MPa√m [2]. The parameter T_0 is all that is needed to characterize fracture toughness in the ductile-to-brittle transition region, since the shape of the Master Curve is universal for all ferritic steels [3]. The effect of specimen size on fracture toughness is treated by the use of the weakest-link theory [4] applied to a three-parameter Weibull distribution of fracture toughness values. Statistical methods are employed to predict the specific transition toughness curve and specific tolerance bounds for the material tested.

The reference temperature T_0 is dependent on loading rate, and has been shown to increase with increasing loading rate [5]. For reactor pressure vessel (RPV) steels used in the nuclear industry, the so-called ASTM reference curve K_{IR} is used as a lower-bound curve which limits the embrittlement that can be observed and measured [6]. In the nuclear field, elevated loading rates are often used as a surrogate of neutron irradiation for studying embrittlement phenomena of RPV steels.

In 2012, the Fraunhofer Institute for Mechanics of Materials (IWM, Freiburg, Germany) and the Materials Testing Institute University of Stuttgart (MPA Stuttgart, Germany) have collaborated in a joint project aimed at verifying the lower-bound K_{IR} curve for a German RPV steel denominated 22NiMoCr37 (ASTM A508 cl. 2) at elevated loading rates [7]. The results of the project confirmed the conservatism of the ASME K_{IR} curve in comparison to the dynamic fracture toughness values measured on specimens of different configuration (compact tension, single-edge bend) and size at loading rates in the range $dK/dt = 10^5$ MPa√m/s – 10^7 MPa√m/s.

In 2014, IWM and MPA invited NIST to contribute to a follow-up collaborative joint project titled "Dynamic Mastercurves II" (MC-Dyn II), in which Wolfgang Böhme is Project Coordinator and Project Leader at IWM and Uwe Mayer is Project Leader at MPA. NIST was asked to test 25 precracked and side-grooved Charpy-type specimens of 22NiMoCr37 at 0 °C and loading rates on the order of 10^5 MPa√m/s. The specimens had to be tested on an instrumented impact pendulum with a reduced impact speed ($v_0 \approx 1.2$ m/s) and the dynamic reference temperature $T_{0,dyn}$ had to be established in accordance with ASTM E1921-14a.

To the author's knowledge, this is the first time dynamic toughness tests on precracked Charpy-type (PCC) specimens have been performed at NIST Boulder.

2. Material and Experimental

The material tested is a German RPV steel denominated 22NiMoCr37, which corresponds to ASTM A508 cl. 2. Its measured chemical composition is given in Table 1, and Table 2 provides basic mechanical properties as reported in [7,8].

Table 1 – Chemical composition of 22NiMoCr37 steel, wt %.

C	Si	Mn	P	S	Mo	Ni	Cr	Cu	V	Co	Al
0.18	0.15	0.82	0.005	0.008	0.54	0.96	0.39	0.08	<0.01	0.014	0.016

Table 2 – Basic mechanical properties of 22NiMoCr37 [7,8].

Tensile properties at 20 °C	Yield strength, R_{el} = 430 MPa Tensile strength, R_m = 587 MPa
Charpy properties T_{XJ} (temperatures corresponding to X J absorbed energy)	T_{28J} = -40 °C T_{41J} = -31 °C T_{68J} = -17 °C
Nil Ductility Reference Temperature[1]	RT_{NDT} = -20 °C
Quasi-static Reference Temperature[2]	T_0 = -68 °C

The Charpy-type specimens for this project were extracted from the beltline of the reactor pressure vessel of Biblis C, which was never in operation. The specimens were machined in T-S orientation, with the crack propagating through the thickness from the inner diameter and the fatigue precrack tip located in the region between 2/3 and 3/4 of the vessel thickness.

In order to facilitate the initiation of the fatigue precrack, a narrow EDM[3] slot was machined instead of a conventional Charpy notch (45° notch angle with 2 mm notch depth). Twenty-five specimens were fatigue precracked at IWM Freiburg, than side-grooved to 80 % of the original thickness at MPA Stuttgart, and finally shipped to NIST[4]. Table 3 reports the average values and standard deviations for the specimen dimensions (width W, thickness B, net thickness B_N, and initial crack size a_0), all measured after the specimens had been tested. The average value of the ratio between initial crack size and specimen width is $a_0/W = 0.330 \pm 0.007$.

Table 3 – Average values and standard deviations of specimen dimensions.

	W (mm)	B (mm)	B_N (mm)	a_0 (mm)
Average	10.00	9.99	8.07	3.30
Stand. dev.	0.037	0.025	0.055	0.077

Tests were conducted on an instrumented pendulum machine with 406.5 J energy capacity and 134˚ fall angle at full swing, corresponding to a maximum impact speed of 5.47 m/s. The

[1] Maximum temperature where a standard drop-weight specimen breaks when tested according to the provisions of ASTM E208-06(2012). This temperature is used for the establishment of the ASME K_{IR} lower bound curve.
[2] Corresponding to a quasi-static loading rate on the order of 1 MPa√m/s.
[3] Electro-Discharge Machining.
[4] At the time of testing, NIST was not equipped for fatigue precracking of Charpy-type specimens.

instrumented striker conforms to the geometry of the ASTM E23-12b standard, having a striking edge radius of 8 mm. Impact tests on PCC specimens were performed at reduced speed, which was obtained by dropping the pendulum from a lower angle $\alpha \approx 23.6^\circ$, resulting in an approximate impact speed of 1.21 m/s and a potential energy of 20 J. Average values for the test parameters are given in Table 4 along with standard deviations.

Table 4 – Average values and standard deviations of test parameters (fall angle, potential energy and impact speed).

	α (°)	E_p (J)	v_0 (m/s)
Average	23.58	20.01	1.2137
Stand. dev.	0.025	0.042	0.001

Data from the instrumented striker, amplified with a gain factor of approximately 200, were acquired at a sampling rate of 2 MHz, and transferred to a personal computer via an analog-to-digital converter unit. The electric output (mV) from the strain gages applied to the striker was converted into force (kN) by means of the static calibration of the instrumented striker (Figure 1). No further correction or adjustment was applied to the instrumented data.

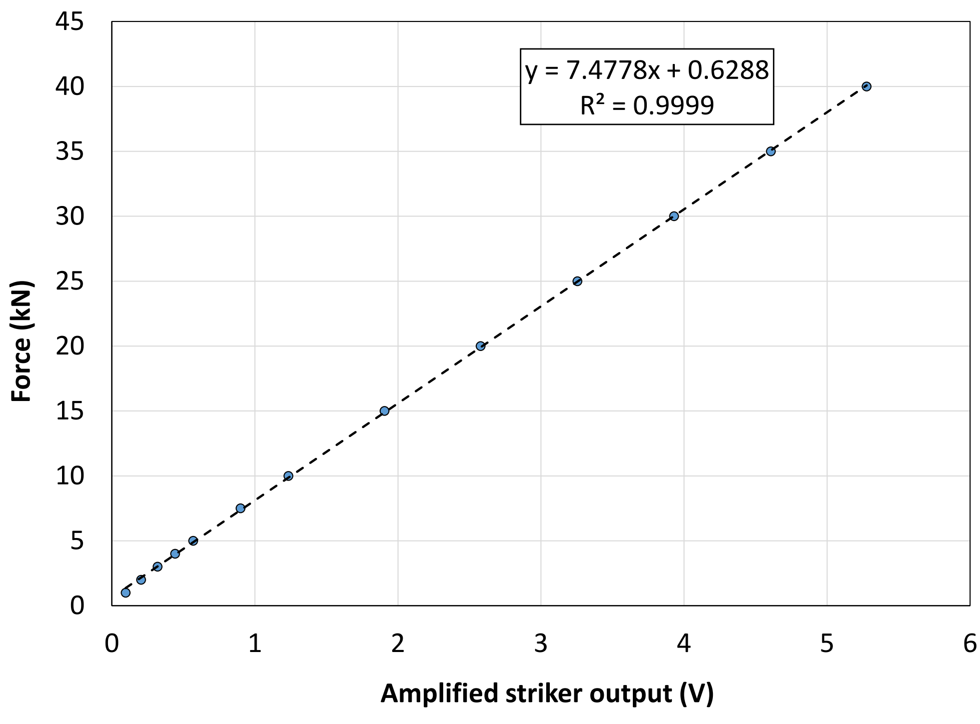

Figure 1 - Static calibration of the instrumented striker used for the tests.

3. Calculations

3.1 Individual test analysis

The raw data file of each test, containing time and force data, was first processed to obtain velocity *v(t)*, displacement *s(t)*, and absorbed energy *W(t)* in accordance with ASTM E2298-13 [9]:

$$v(t) = v_0 - \frac{1}{m} \int_{t_0}^{t} F(t)dt \quad (1)$$

$$s(t) = \int_{t_0}^{t} v(t)dt \quad (2)$$

$$W(s) = \int_{s_0}^{s} F(s)ds \quad (3)$$

where t_0 , v_0, and s_0 are time, velocity, and displacement corresponding to the start of the test.

The points corresponding to general yield (*gy*) and unstable fracture (*bf*) were then visually identified on the force/displacement test record, as well as the initial linear elastic portion of the curve, which was fitted by a straight line (excluding the first inertia peak – see Figure 2).

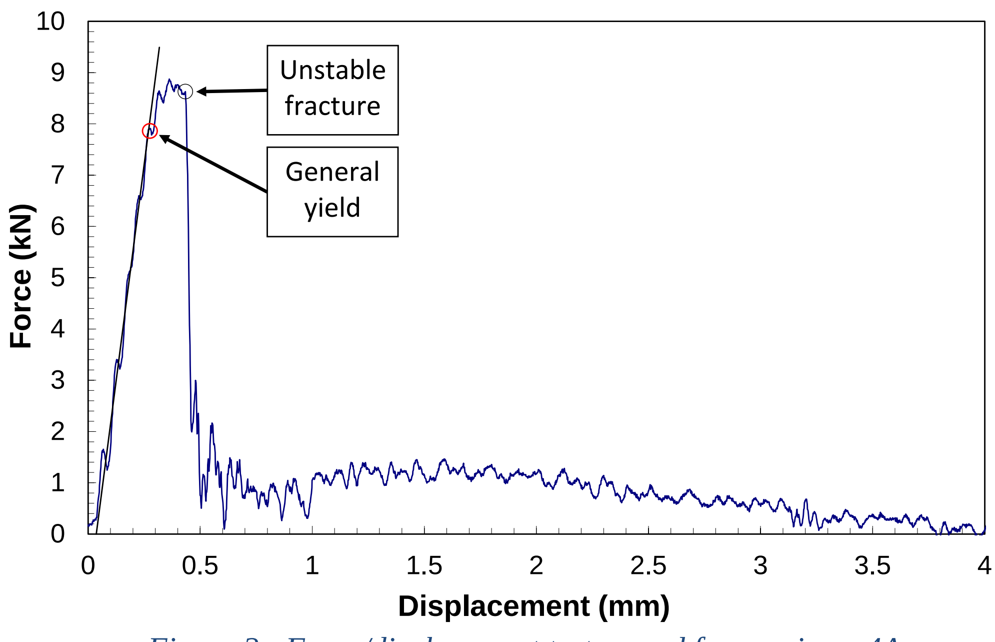

Figure 2 - Force/displacement test record for specimen 4A.

The following validity conditions have to be fulfilled for the test to be valid [10]:

(a) The potential energy (E_p) must be greater than three times the absorbed energy at maximum force (W_m), to avoid an excessive decrease of hammer speed during the test:

$$E_p > 3W_m \ . \quad (4)$$

(b) The time corresponding to unstable fracture (t_f) must be greater than three times the period of oscillation (τ) of the specimen/striker system, to ensure that inertial oscillations have sufficiently dampened and do not prevent a reliable identification of the initiation of unstable fracture:

$$t_f > 3\tau \quad . \tag{5}$$

Both requirements, eqs. 4 and 5, were satisfied by all the tests performed.

In accordance with ASTM E1921-14a, the stress intensity factor at unstable fracture, K_{Jc}, is calculated from the corresponding value of J-integral, J_c, using:

$$K_{Jc} = \sqrt{\frac{J_c E}{1-\nu^2}} \tag{6}$$

where E = 212 GPa is the Young's modulus at the test temperature (0 ˚C), and ν is Poisson's ratio (0.3).

The value J_c is calculated as the sum of an elastic (J_{el}) and a plastic (J_{pl}) component, respectively, given by:

$$J_{el} = \frac{K_c^2\left(1-\nu^2\right)}{E} \tag{7}$$

with:

$$K_c = \frac{F_{bf}\,S}{\sqrt{BB_N}\,W^{1.5}}\, f\left(\frac{a_0}{W}\right) \tag{8}$$

(F_{bf} = force at unstable fracture; S = 42 mm, span or distance between the machine supports; the elastic function $f(a_0/W)$ is given in E1921-14a), and:

$$J_{pl} = \frac{1.9 \cdot W_{bf,pl}}{B_N\left(W - a_0\right)} \tag{9}$$

where the plastic part of the absorbed energy at unstable fracture is:

$$W_{bf,pl} = W_{bf} - \frac{C_0 F_{bf}^2}{2} \tag{10}$$

with W_{bf} = absorbed energy at unstable fracture and C_0 = reciprocal of the initial elastic slope in mm/kN (Figure 2).

The value K_{Jc} calculated via eqs. 6-10 needs to be finally validated by comparison with the maximum specimen capacity, expressed as:

$$K_{Jc(\text{limit})} = \sqrt{\frac{E\left(W - a_0\right)\sigma_{YS}}{30\left(1-\nu^2\right)}} \tag{11}$$

where σ_{YS} is the material's yield strength at the test temperature and relevant loading rate. For our analyses, a dynamic yield strength value σ_{YS} = 558 MPa was used, based on dynamic tensile tests performed at MPA Stuttgart.

If $K_{Jc} < K_{Jc(limit)}$, the value can be used "as is" in the subsequent Master Curve analysis. If $K_{Jc} \geq K_{Jc(limit)}$, the value is censored and replaced with $K_{Jc(limit)}$ in the Master Curve analysis. As will be seen in the following, only two of the 24 tests performed yielded a K_{Jc} value above the specimen measuring capacity.

For each individual test, the stress intensity rate at fracture is obtained by dividing the K_{Jc} value by the corresponding time to fracture t_f.

3.2 Master Curve analysis

Each individual K_{Jc}, calculated according to eqs. 6-10, is size-adjusted with respect to the thickness of a 25 mm-thick C(T) specimen (1TCT[5]):

$$K_{Jc(1TCT)} = 20 + \left(K_{Kc(PCC)} - 20\right)\left(\frac{B_{PCC}}{B_{1TCT}}\right)^{1/4} \tag{12}$$

where B_{PCC} and B_{1TCT} = 25 mm are, respectively, the gross thicknesses (neglecting any side-grooves) of the test specimen and the reference 1TCT specimen.

As mentioned above, any K_{Jc} value which exceeds the specimen capacity given by eq. 11 is censored and replaced with $K_{Jc(limit)}$ in subsequent analyses.

A provisional dynamic reference temperature value, T_{0Q}, is calculated by iteratively solving the following equality [12,13]:

$$\sum_{i=1}^{N} \delta_i \frac{\exp\left[0.019\left(T_i - T_{0Q}\right)\right]}{11.0 + 76.7 \cdot \exp\left[0.019\left(T_i - T_{0Q}\right)\right]} - \frac{\left(K_{Jc(i)} - 20\right)^4 \cdot \exp\left[0.019\left(T_i - T_{0Q}\right)\right]}{\left\{11.0 + 76.7 \cdot \exp\left[0.019\left(T_i - T_{0Q}\right)\right]\right\}^5} = 0 \tag{13}$$

where: N = number of specimens tested;

T_i = test temperature;

$K_{Jc(i)}$ = side-adjusted test result or censored value;

δ_i = 1 if the datum is valid or 0 if it's a censored value.

The provisional value T_{0Q} is validated as the dynamic reference temperature of the material at the specific loading rate if:

$$\sum_{i=1}^{3} r_i n_i \geq 1 \tag{14}$$

where r_i is the number of valid tests within the i-th temperature range ($T - T_0$), and n_i is the specimen weighting factor for the same temperature range as shown in Table 5.

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[5] 1TCT = 1 in.-thick C(T) specimen. The thickness is rounded from 1 in. = 25.4 mm to 25 mm.

Table 5 – Weighting factors for Master Curve analysis.

(T – T_0) range (°C)	**Weighting factor n_i**
50 to -14	1/6
-15 to -35	1/7
-36 to -50	1/8

For the tests performed, the test temperature (0 °C) lies within the first range in Table 5, so each one of the 23 valid tests contributes by 1/6. The relationship expressed by eq. 14 is therefore satisfied, and T_{0Q} is validated as $T_{0,d}$.

The toughness/temperature transition curve (Master Curve) is given by:

$$K_{Jc(med)} = 30 + 70 \cdot \exp[0.019(T - T_0)] \quad (15)$$

where $K_{Jc(med)}$ is the median K_{Jc} toughness for 1TCT specimens. Upper and lower tolerance bounds (typically corresponding to 5 % and 95 % cumulative fracture probability) can be calculated by means of:

$$K_{Jc(0.xx)} = 20 + \left[\ln\left(\frac{1}{1 - 0.xx}\right)\right]^{1/4} \{11 + 77 \cdot \exp[0.019(T - T_0)]\} \quad (16)$$

where 0.xx = 0.05 and 0.95 for the 5 % and 95 % tolerance bounds, respectively.

4. Results

Out of 25 PCC specimens of 22NiMoCr37 shipped to NIST, 24 analyzable data sets were obtained. During the test on specimen B14, the data acquisition system did not trigger and the instrumented data were lost.

Only two of the 24 tested specimens (D3) provided K_{Jc} values which exceeded $K_{Jc(limit)}$ given by eq. 11 and were therefore replaced with the limit values in the analyses. The summation in eq. 14 equals 3.677, based on 22 valid results. Table 6 reports individual values of $K_{Jc(PCC)}$, $K_{Jc(limit)}$, $K_{Jc(1TCT)}$, and dK/dt.

Table 6 – Individual test results. NOTE: invalid tests marked in red italic font.

Specimen ID	$K_{Jc(PCC)}$ (MPa√m)	$K_{Jc(limit)}$ (MPa√m)	$K_{Jc(1TCT)}$ (MPa√m)	dK/dt (10^5 MPa√m/s)
L2	85.3	172.6	71.9	3.09
E1	94.2	170.2	78.9	3.36
G7	107.2	172.2	89.3	3.25
D7	110.9	172.0	92.3	3.40
M12	112.2	170.7	93.3	3.35
M10	117.3	171.8	97.3	3.26
4A	118.7	172.0	98.5	3.25
M1	118.8	171.5	98.5	3.41
E10	119.1	171.9	98.8	3.46
K3	125.1	171.4	103.6	3.43
G3	131.7	170.0	108.7	3.43
L10	137.2	169.8	113.2	3.42
N14	137.9	170.1	113.7	3.41
L14	140.3	172.5	115.7	3.32
N6	144.4	170.0	118.9	3.37
12A	147.3	170.9	121.1	3.17
K7	147.6	170.9	121.6	3.35
M4	150.6	171.3	123.9	3.16
M2	150.9	172.0	124.1	3.25
D11	160.5	170.7	131.5	3.24
H9	166.5	171.3	136.6	3.15
E13	172.0	172.0	140.2	3.10
B2	*175.7*	*169.4*	*138.1*	*3.06*
D3	*203.0*	*169.6*	*138.4*	*2.91*

The average stress intensity rate for the 24 tests analyzed was 3.3 × 10^5 MPa√m ± 4.4 %.

The reference temperature, calculated by iteratively solving the equality in eq. 13 and validated by the condition expressed in eq. 14, is:

$$T_0 = -7.5\ °\mathrm{C} \quad .$$

The uncertainty of the calculated reference temperature is given by:

$$\sigma = \sqrt{\frac{\beta^2}{r} + \sigma_{exp}^2} \tag{17}$$

where: β = sample size uncertainty factor;
r = number of valid K_{Jc} results;
σ_{exp} = contribution of experimental uncertainties (if standard calibration practices are followed, σ_{exp} = 4 °C).

The value of the sample size uncertainty factor depends on the median toughness of the data set, which is expressed as [14]:

$$K_{Jc(med)}^{eq} = \frac{1}{r}\sum_{i=1}^{r} 30 + 70 \cdot \exp\left[0.019\left((T_i - T_0)\right]\right. \quad (18)$$

For the data set under consideration, eq. 18 yields $K_{Jc(med)}^{eq} = 110.7$ MPa√m, and therefore [15] β = 18 °C. Substituting in eq. 17, the uncertainty of the calculated reference temperature is therefore σ = 5.6 °C.

The size-adjusted experimental data are shown in Figure 3 with the corresponding dynamic Master Curve (corresponding to 50 % fracture probability), as well as the 5 % and 95 % tolerance bounds.

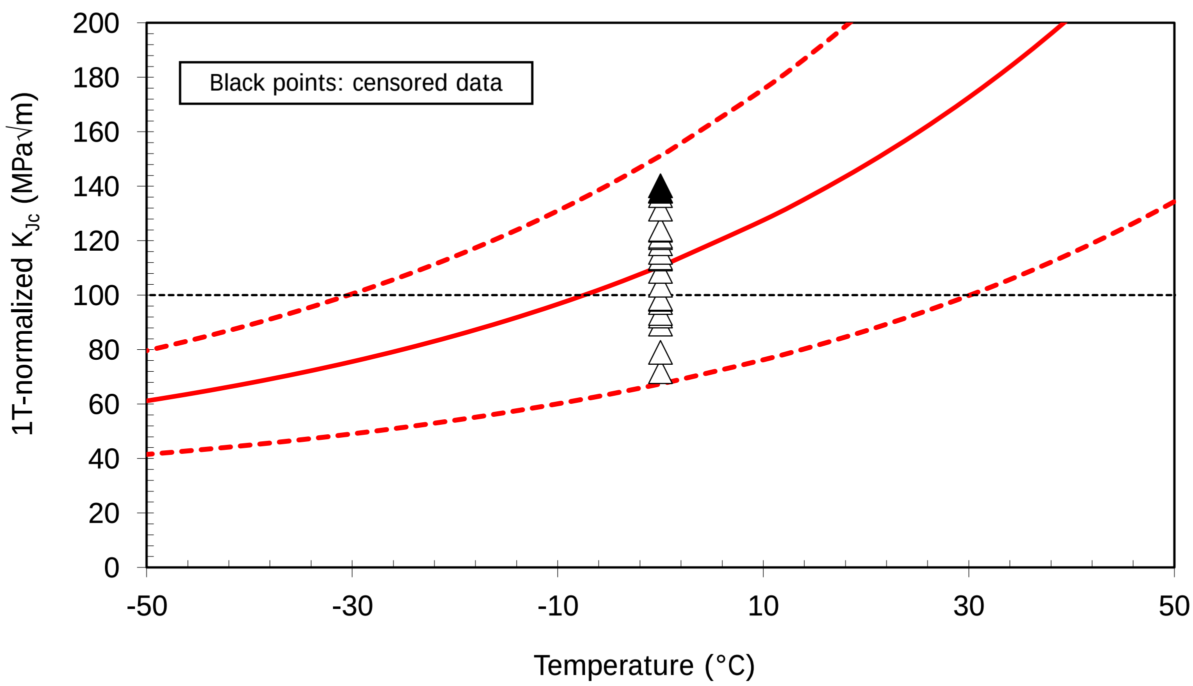

Figure 3 – Experimental data and dynamic Master Curves corresponding to 50 % (solid curve), 95 % (upper dashed curve), and 5 % (lower dashed curve) fracture probabilities.

5. Discussion

The value obtained at NIST for the dynamic reference temperature of 22NiMoCr37 by testing PCC specimens at an average loading rate of 3.28 × 10^5 MPa√m/s, $T_{0,d}$ = -7.5 ˚C ± 5.6 ˚C, is in excellent agreement with the value reported in [7,8] for PCC specimens (SE(B) 10 × 10) tested at -20 °C at 2 × 10^5 MPa√m/s ($T_{0,d}$ = -7 °C).

With respect to the quasi-static value of the reference temperature reported in [8], T_0 = -68 °C, the increase in T_0 caused by the increase in loading rate is 62 °C.

It has been contended [8] that dynamic fracture toughness could be limited by crack arrest effects, and therefore a lower-bound dynamic Master Curve (such as the 5 % tolerance bound) should be limited by the ASME K_{IR} reference curve [6]. In reactor safety assessment, the K_{IR} curve is deemed to represent a lower bound for fracture toughness properties, particularly for accidental situations when high loading/strain rates are encountered. On this topic, the German government recently funded a collaborative research project between IWM and MPA to investigate the correlation between dynamic crack initiation and crack arrest: it was found that the K_{IR} curve effectively bounds dynamic fracture toughness properties measured from PCC specimens tested at impact loading rates [7]. Our results confirm the outcome of the German project, as shown in Figure 4: all experimental data, and the relevant 5 % Master Curve, fall above the K_{IR} curve, which is given by:

$$K_{IR} = 26.78 + 1.223 \cdot \exp[0.0145(T - RT_{NDT} + 160)] \tag{19}$$

with K_{IR} in ksi√in. and temperatures T and RT_{NDT} in ˚F. RT_{NDT} for the 22NiMoCr37 steel is -20 ˚C or -4 ˚F, according to Table 2 [8].

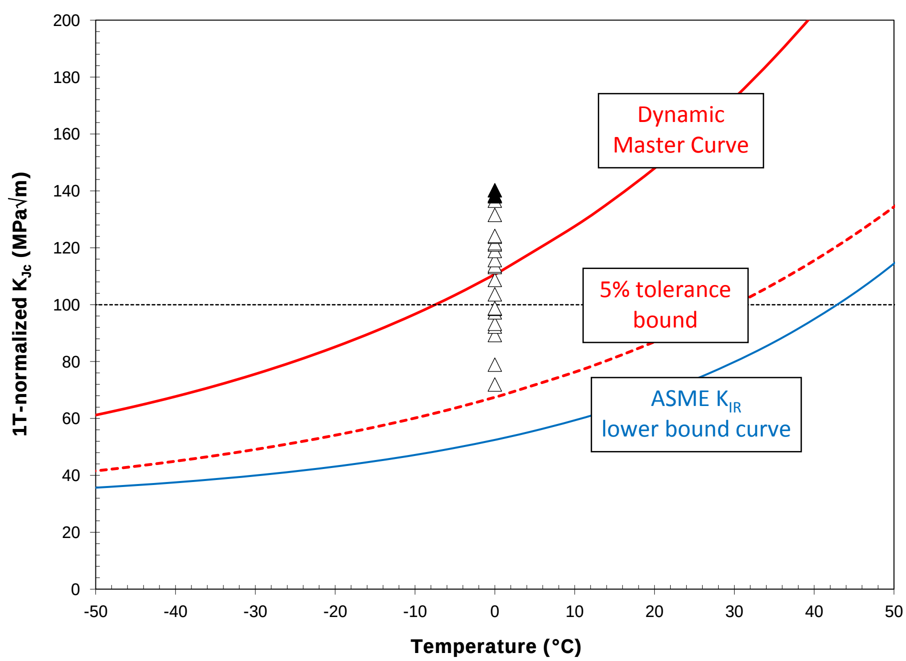

Figure 4 – Comparison between dynamic Master Curves and ASME K_{IR} lower-bound curve.

Other researchers have claimed that high loading rate data might not be represented satisfactorily by the Master Curve equation, eq. 15, which is typically used for quasi-static test data. In particular, Schindler and Kalkhof [16] claimed that the dynamic Master Curve is steeper, and proposed the following expression for the median Master Curve equation:

$$K_{Jc(med)} = 30 + C \cdot \exp[p(T - T_0)] \quad , \tag{20}$$

where the rate-dependent form factor p equals 0.019 for quasi-static loading rates, but can increase up to 0.04 for impact loading rates on the order of 10^5 MPa√m. This would happen as a result of adiabatic heating at the crack tip caused by elevated loading rates, which tends to counteract the effect of dynamic embrittlement. Böhme *et al.* [8] used $p = 0.03$ for their dynamic Master Curve evaluations. The results obtained by comparing eq. 15, corresponding to $p = 0.019$, and eq. 20 with $C = 70$ MPa√m and $p = 0.03$, are depicted in Figure 5 together with our experimental data after size adjustment. The reference temperature changes only by 2.2 ˚C ($T_{0,p=0.03}$ = -5.3 ˚C).

With only one experimental data set, it's hard to confirm or refute Schindler's claim. We note that all the experimental data points are bounded by both the "conventional" 5 % Master Curve and the alternative 5 % confidence bound with $p = 0.03$.

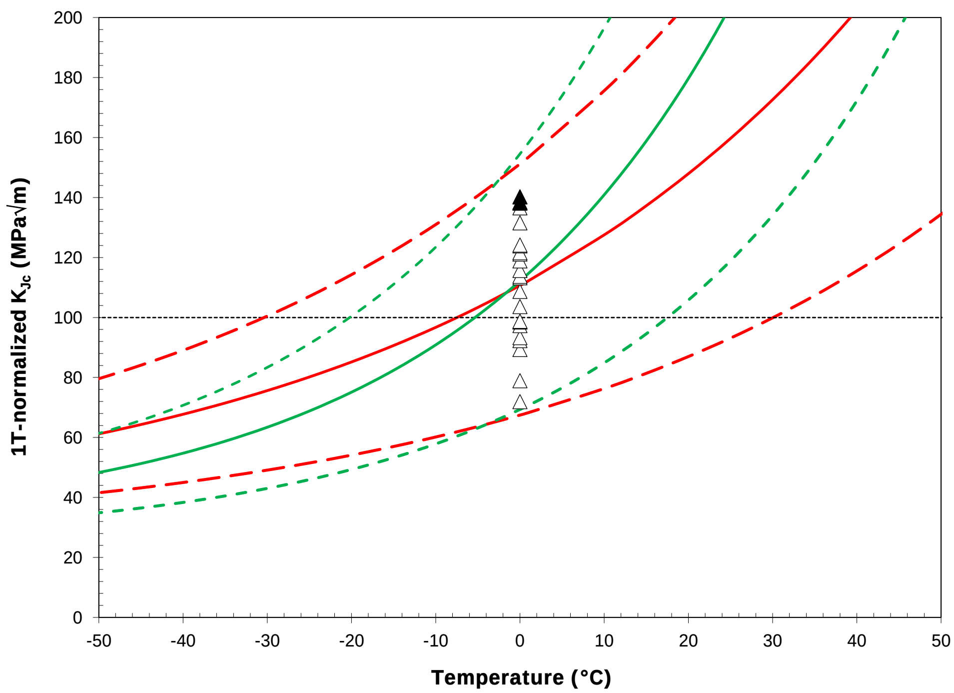

Figure 5 – Comparison between experimental data and Master Curves corresponding to eq. 15 (red curves) and to eq. 20 (green curves), that is p = 0.0019 and p = 0.03 respectively.

6. Conclusions

The dynamic fracture toughness of a German pressure vessel steel, denominated 22MoNiCr37, was characterized in the ductile-to-brittle transition region by testing 25 precracked and side-grooved Charpy-type (PCC) specimens by means of an instrumented impact machine. Tests were performed at 0 ˚C with an impact velocity of 1.21 m/s, corresponding to a stress intensity factor rate (loading rate) on the order of 3×10^5 MPa√m/s. The fracture toughness values corresponding to unstable fracture, K_{Jc}, were statistically evaluated in accordance with the Master Curve methodology (ASTM E1921-14a).

The value obtained for the dynamic reference temperature, corresponding to a median dynamic fracture toughness of 100 MPa√m for 1TCT specimens, was $T_{0,d}$ = -7.5 ˚C, with a standard deviation of 5.6 ˚C. Our results, which represent the NIST contribution to a cooperative project with IWM Freiburg and MPA Stuttgart (Germany), are in excellent agreement with results previously published by IWM.

As far as the author was able to ascertain, these are the first dynamic fracture toughness tests ever performed at NIST Boulder and the results look extremely promising in view of a future application of this methodology to other materials of interest to NIST, such as pipeline steels, ultra-high-energy steels, welded joints, etc.

Acknowledgments

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