NIST Technical Note 2168

# NIST Contribution to the ASTM E08.07.05 Round Robin on Determining the Master Curve Reference Temperature, $T_{0,X}$, at Elevated Loading Rates (ILS #1547)

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National Institute of
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## Abstract

The ASTM Interlaboratory Study (ILS) #1547 (*Determining $T_{0,X}$ at Elevated Loading Rates*) was launched in 2019 under the leadership of MPA Stuttgart (Germany). The aim of this interlaboratory exercise was to generate Master Curve results at elevated loading rates, in order for the *Precision and Bias* section of the ASTM E1921 standard to be updated and expanded with data obtained from specimens other than precracked Charpy samples. The NIST contribution, detailed in this report, consisted of eighty (80) low-temperature fracture toughness tests at elevated loading rates ($dK/dt$ between $10^2$ MPa√m/s - $10^3$ MPa√m/s). Three different specimen types were used: compact tension with 25 mm thickness, single-edge bend with 20 mm thickness and 40 mm width, and precracked Charpy-type. The materials selected for the ILS were Biblis C base and weld materials, and S590QL steel. Test results were analyzed in accordance with ASTM E1921 (Master Curve procedure) for the calculation of the reference temperature $T_{0,X}$. Some of the tests were performed at actuator displacement rates (30 mm/s-40 mm/s) that turned out to be too high for the frequency bandwidth of the force measuring system. The remaining tests were therefore conducted at a displacement rate lower by an order of magnitude (2.5 mm/s-3 mm/s). For the analysis of the individual tests, a macro-enabled spreadsheet was implemented for performing the calculations in accordance with Annex A14 of the ASTM E1820 standard.



## Key words

Fracture toughness; interlaboratory study; loading rate; Master Curve; reference temperature.

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## Glossary

| | |
|---|---|
| 1TC(T) | compact tension specimen with thickness = 25 mm |
| $a/W$ | crack size-to-width ratio in a fracture toughness specimen, where $a$ = crack size and $W$ = specimen width |
| $B$ | specimen thickness (mm) |
| $B_N$ | net thickness of a side-grooved specimen (mm) |
| $BW_{min}$ | minimum required frequency bandwidth for the force measuring system (Hz) |
| CMOD | crack-mouth opening displacement (mm) |
| $dK/dt$ | loading rate, expressed in terms of stress-intensity factor rate (MPa√m/s) |
| $EBW$ | frequency bandwidth of the force measuring system, estimated from the force drop rate after specimen fracture (Hz) |
| $J_c$ | $J$-integral value at cleavage (kJ/m$^2$) |
| $K_{Jc}$ | value of stress-intensity factor at cleavage (unstable fracture) (MPa√m) |
| $K_{Jclimit}$ | maximum $K_{Jc}$ capacity for a specimen according to ASTM E1921 (MPa√m) |
| $K_{med}$ | median value of fracture toughness for a data set (MPa√m) |
| $K_{min}$ | minimum value of stress-intensity factor in the Master Curve analysis, conventionally set at 20 MPa√m (MPa√m) |
| $k_s$ | specimen load-line stiffness (N/m) |
| LLD | load-line displacement (mm) |
| MC | Master Curve |
| $M_{eff}$ | effective mass of the specimen, equal to half of the specimen mass (kg) |
| $N_{out}$ | number of data points falling outside the 5 % and 95 % Master Curve confidence limits |
| $p$ | Master Curve exponent (normally, $p$ = 0.019) |
| PCCv | fatigue precracked Charpy-type specimen |
| $r$ | number of valid data in a Master Curve data set |
| SE(B) | single-edge bend specimen |
| $T_0$ | Master Curve reference temperature, corresponding to a median $K_{Jc}$ = 100 MPa√m for 1T specimens (°C) |
| $T_{0,X}$ | Master Curve reference temperature at elevated loading rates ($dK/dt$ > 2 MPa√m/s), where $X$ = logarithm of the average loading rate for the data set (°C) |
| $T_{0,X}^{est}$ | estimated value of reference temperature according to Eq. (A1.1) of ASTM E1921 (°C) |
| $T_{0IN}$ | conservative estimate of the Master Curve reference temperature for a macroscopically inhomogeneous data set (°C) |
| $t_f$ | time to specimen fracture (s) |
| $t_Q$ | test time, or the observed time to the rate dependent $J_{c(t)}$ (s) |
| $t_w$ | minimum test time according to Annex A1 of ASTM E1820 (s) |
| $\beta$ | sample size uncertainty factor in ASTM E1921 |
| $\Delta_{LL}^{F}$ | final crossover in the force smoothness verification prescribed in A14.7.4 of ASTM E1820 (mm) |
| $\Gamma$ | function used to calculate an estimated value of $T_{0,X}$ according to Eq. (A1.1) of ASTM E1921 |
| $\sigma_{exp}$ | contribution of experimental uncertainties to the overall standard deviation of the reference temperature (°C) |

| | |
|---|---|
| $\sigma_{T0}$ | standard deviation of the Master Curve reference temperature (°C) |
| $\sigma_{ys}$ | room temperature yield strength (MPa) |
| $\sum r_i n_i$ | sum of weight factors, used to validate the Master Curve reference temperature. |

## 1. Introduction

The so-called "Master Curve" (MC) approach [1] allows for establishing the relationship between test temperature and fracture toughness (expressed in terms of stress intensity factor, $K_{Jc}$) in the ductile-to-brittle transition region for ferritic steels that experience cleavage cracking at elastic or elastic-plastic instabilities. The MC has a common shape for all ferritic steels, and its position on the temperature axis is established by the reference temperature $T_0$. This is defined as the temperature at which the median toughness $K_{Jc}$ for 1 in. (25 mm)-thick (1T) specimens is 100 MPa√m.

The statistical relationship between specimen size (thickness) and $K_{Jc}$ fracture toughness is assessed using a weakest-link theory, applied to a three-parameter Weibull distribution of fracture toughness values [2]. The methodology also specifies a limit on $K_{Jc}$ values, relative to specimen size, which ensures sufficiently high constraint conditions along the crack front at fracture.

The MC approach was standardized in 1997 in the ASTM E1921 standard (*Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range*). This test standard has been updated and revised many times since. In particular, the 2015 version introduced Annex A1 (*Special Requirements for Determining the Reference Temperature, $T_{0,X}$, at Elevated Loading Rates*). Before that, determination of the reference temperature, $T_0$, was restricted to quasi-static loading rates, $dK/dt$, between 0.1 MPa√m/s and 2 MPa√m/s. However, Annex A1 only addressed precracked Charpy specimens, tested at impact loading rates by means of an instrumented Charpy machine. The resulting loading rates are typically of the order of $10^5$ MPa√m/s.

Up to the current version of the standard (E1921-20), no provisions exist for tests conducted on standard specimen geometries (compact tension, single-edge notched bend bars, or disk-shaped compact tension) at loading rates higher than the current upper limit of 2 MPa√m/s, in the range $10^2$ MPa√m/s - $10^4$ MPa√m/s.

## 2. Background of the ASTM E08.07 Round-Robin (ILS #1547)

During the ASTM E08.07.05 sub-committee meeting held in November 2018, Uwe Mayer from MPA Stuttgart (Germany) proposed a round-robin on the determination of $T_{0,X}$ at elevated loading rates using standard specimen geometries. This activity was later officially launched as ASTM Interlaboratory Study (ILS) #1547, and the program was officially launched during the May 2019 sub-committee meetings [3].

This ILS aimed at filling a gap in the research on dynamic MC measurements, which until then had been limited to the use of impact-tested precracked Charpy (PCCv) specimens [4,5]. Within such investigations, the small size of the Charpy specimen restricted the possible valid test temperature range below the allowable $T_0 \pm 50$ °C prescribed by ASTM E1921 for standard specimen geometries. Namely, the $K_{Jc}$ limit previously mentioned, which depends on the specimen thickness, greatly limits the temperature range below $T_0$ where PCCv specimens can be tested. An additional limitation ensues from requirements imposed on the frequency bandwidth of the force and displacement systems, which will be discussed later.

Several published studies [6-9] provided indications that, when larger specimens (compact or bend) are tested at elevated loading rates, loading rate effects might affect the shape of the Master Curve, the threshold minimum toughness $K_{min}$, and the statistical distribution of the test results. Specifically, some authors have claimed that the slope of the Master Curve should be steeper when high loading rate data are analyzed – which would imply a higher exponent (0.03 instead of 0.019) in the analytical expression of the curve. None of these studies, however, produced a sufficiently large database of results for the outcome to be statistically significant. The ASTM round-robin was therefore set up to fill this gap and eventually contribute to a new or expanded *Precision and Bias* statement for Annex 1 of ASTM E1921.

## 3. Round-Robin Details

Testing for ILS #1547 was to be performed in accordance with ASTM E1820 (*Measurement of Fracture Toughness*), Annex A14 (*Special Requirements for Rapid-Load J-Integral Fracture Toughness Testing*). All Master Curve analyses were to be conducted in accordance with the current version of ASTM E1921.

Specimen geometries included compact tension specimens with 25 mm thickness, 1TC(T), and single-edge notched bend specimens, SE(B), with thickness = 20 mm, width = 40 mm, and length = 220 mm (SE(B) 20/40). Some additional Charpy-type specimens (SE(B) with thickness and width = 10 mm) were extracted from tested SE(B) samples of two materials, fatigue precracked, and tested (PCCv specimens). All specimens were precracked to a nominal crack size-to-specimen width ratio, *a/W*, of 0.5. The geometries of the 1TC(T) and SE(B) 20/40 specimens tested by NIST (all with integral knife edges for the application of a clip-gage) are shown in Appendices A and B.

Three materials were investigated:

1. Base material from the pressure vessel of the never commissioned German Biblis C pressurized water nuclear reactor. Specifically, a forged ring of DIN 22NiMoCr37 steel (corresponding to ASTM A508 Grade 2 Class 1) was utilized for machining the specimens. Its chemical composition [10] is given in **Table 1**.
2. Weld material of the same Biblis C pressure vessel, from the multi-layer beltline welding seam between the upper and lower ring of the vessel. Its chemical composition [11] is given in **Table 1**.
3. High strength structural steel S690QL, equivalent to ASTM A514/A517. The designation S690QL refers to a minimum yield strength of 690 MPa. Its chemical composition [12] is given in **Table 1**.

**Table 1** - Chemical composition in wt % of the materials used in the round-robin.

| Material | C | Si | Mn | P | S | Ni | Cr | Cu | Mo | V | Fe |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Biblis C base | 0.215 | 0.198 | 0.905 | 0.008 | 0.007 | 0.875 | 0.415 | 0.0406 | 0.528 | 0.0072 | Bal. |
| Biblis C weld | 0.054 | 0.169 | 1.190 | 0.013 | 0.007 | 0.937 | 0.041 | 0.041 | 0.554 | 0.0064 | Bal. |
| S690QL | 0.20 | 0.80 | 1.70 | 0.025 | 0.015 | 2.0 | 1.50 | 0.50 | 0.70 | 0.12 | Bal. |

Seven laboratories participated in the round-robin:

- Anderson & Associates (USA)
- Comtes FHT (Czech Republic)
- IWM Fraunhofer (Germany)
- MPA Stuttgart (Germany)

- NASA (USA)
- NIST (USA)
- Naval Surface Warfare Center (USA).

## 4. NIST Experimental Setup

Fracture toughness tests at NIST Boulder were conducted on a universal servo-hydraulic testing machine equipped with a calibrated 250 kN (55 kip) load cell. Load-line displacements (for 1TC(T) specimens) and crack-mouth opening displacements (for SE(B) 20/40 specimens) were measured by means of a clip-gage with a displacement range of 5 mm. Specimens were tested at actuator displacement rates between approximately 2.5 mm/s - 40 mm/s. Specimens were instrumented with a spot-welded K-type thermocouple.

All tests were performed at cryogenic temperatures between -5 °C and -75 °C, inside an environmental chamber that uses liquid nitrogen to cool down specimens.

The experimental setups used for testing 1TC(T) and SE(B) 20/40 specimens are shown in **Figure 1** and **Figure 2**, respectively.

**Figure 1** - NIST setup for testing 1C(T) specimens. The yellow wire is the thermocouple.


**Figure 2** - NIST setup for testing SE(B) 20/40 specimens. The yellow wire is the thermocouple.

## 5. Individual Test Analyses

As previously mentioned, fracture toughness tests and fracture toughness calculations were conducted in accordance with the provisions of Annex A14 of ASTM E1820 (*Rapid-Load J-Integral Fracture Toughness Testing*).

A Microsoft Excel[1] macro-enabled spreadsheet was used for calculations and validity checks. Its use is described in Appendix C.[2]

With respect to a fracture toughness test conducted at quasi-static rates, the analysis of a rapid-load *J*-integral test features the following notable differences.

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[1] Certain commercial equipment, instruments, software packages, or materials are identified in this document in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

[2] A copy of the spreadsheet can be requested free of charge from the author (enrico.lucon@nist.gov).

- The time/force data points acquired after specimen cleavage and before the force returns to zero (force drop) are linearly fitted in order to calculate the fall time, $t_f$(s):

$$t_f = A(F_{10\%} - F_{90\%}) \ , \tag{1}$$

with $A$ = slope of the fitting line (s/kN), $F_{10\%}$ = 10 % of the force at fracture (kN), and $F_{90\%}$ = 90 % of the force at fracture (kN). The fall time is used to determine the estimated bandwidth of the measuring system, given by:

$$EBW = \frac{0.35}{t_f} \quad . \tag{2}$$

*EBW* is the natural frequency of the whole test system, including the specimen and the specimen fixtures.

- *EBW* must be compared with the minimum acceptable frequency bandwidth for the signal conditioners, $BW_{min}$. This depends on the test time $t_Q$ (s), which is defined as the observed time to the rate-dependent $J$-integral at fracture, $J_c$. It is calculated by linearly fitting $J$-integral data as a function of test time between $0.5J_c$ and $J_c$, and extrapolating the linear fit to the x-axis, as shown in **Figure 3**.

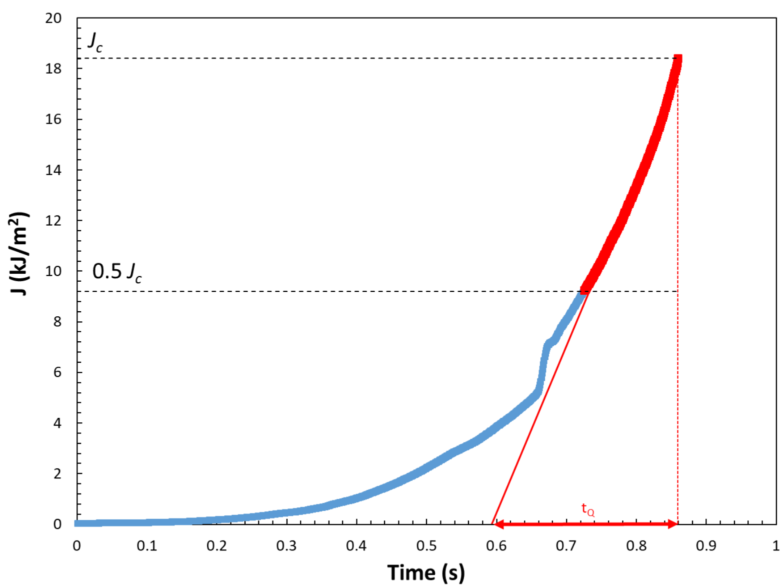


**Figure 3** - Determination of test time $t_Q$.

According to section A14.5.6.1 of ASTM E1820, the frequency bandwidth of the signal conditioners shall be in excess of $10/t_Q$ for force signals and $2/t_Q$ for displacement signals. If the estimated bandwidth of the test system is lower than the recommended minimum, an erroneously high value of maximum force (overshoot) may be measured, which would affect the elastic component of the $J$-integral near crack initiation.

- Moreover, the test time $t_Q$ must be compared with $t_w$, the minimum test time to the rate-dependent $J_c$ [13]. Test times, $t_Q$, lower than $t_w$ will lead to inaccurate J integral results since large kinetic energy components will be present. The minimum test time, in s, is defined as:

$$t_w = \frac{2\pi}{\sqrt{\frac{k_s}{M_{eff}}}} \quad , \tag{3}$$

where: $k_s$ = specimen load-line stiffness (N/m), and

$M_{eff}$ = effective mass of the specimen, taken as half of the specimen mass (kg).

The initial specimen stiffness, $k_s$, is calculated by linearly fitting force vs. time data between 20 % and 50 % of the maximum force measured in the test. As mentioned above, ASTM E1820 requires that $t_Q > t_w$.

- Furthermore, Annex A14 of the E1820 standard prescribes a "force smoothness verification", whereby force and displacement data between 0.3 $\Delta_{LL}^F$ and 0.8 $\Delta_{LL}^F$ ($\Delta_{LL}^F$= value of displacement where the test record departs from linearity, or "final crossover") should remain within two lines, which are parallel to the elastic compliance (inverse of stiffness) with an offset of ± 10 % of the maximum force (**Figure 4**).

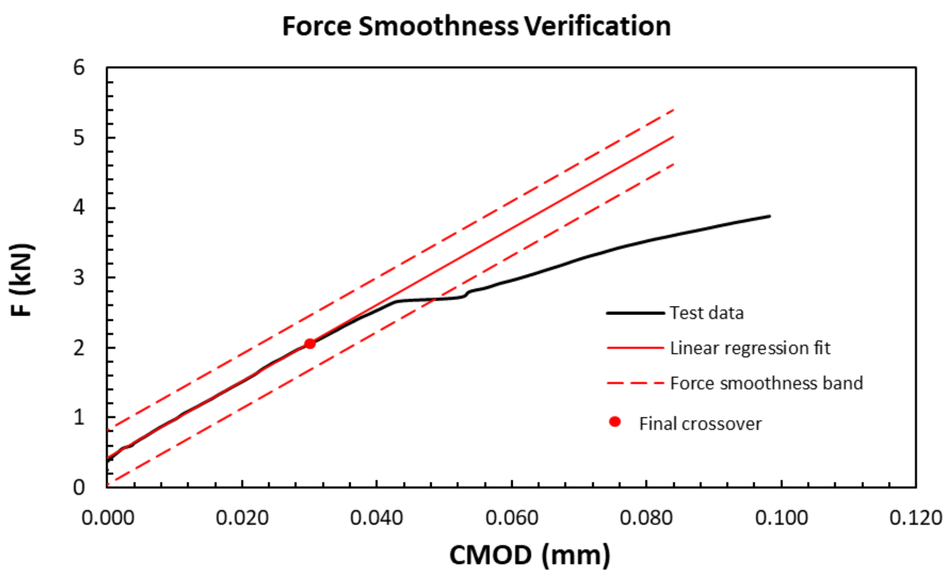


**Figure 4** - Force smoothness verification.

- A final test validity check is performed by comparing the initial crack size estimated from the elastic compliance, $1/k_s$, with the value measured on the fracture surface. The two values shall agree within ± 10 % for the data set to be qualified.

## 6. Master Curve Analyses

Test results ($K_{Jc}$ values) obtained from a specific material and specimen type were analyzed in accordance with the current version of ASTM E1921. Since tests were performed at different temperatures, the multi-temperature analysis (reference method) of section 10.3 was applied for the determination of the reference temperature, $T_{0,X}$.

For each material tested, different data sets were considered and analyzed:

- All tests performed on a specific material.
- Tests performed on a specific specimen type.

- Tests performed at a specific loading rate (actuator displacement rate). For this purpose, we will refer to "higher" rate tests for rates ≥ 30 mm/s and "lower" rate tests for rates ≤ 3 mm/s.

Additionally, each data set was screened for homogeneity according to the SINTAP procedure [14], described in section 10.6 of ASTM E1921. For any data set failing the screening criterion, a simplified method for the treatment of potentially inhomogeneous data sets was applied, in accordance with Annex X5 of E1921. A "generally conservative estimate of the reference temperature", $T_{0IN}$, is obtained from this approach.

## 7. NIST Test Results

### 7.1. Test Matrix

The test matrix for NIST participation in the round-robin is illustrated in **Table 2**. Overall, 80 low-temperature fracture toughness tests were performed in the period 2019-2021.

**Table 2** - NIST test matrix.

| Material | Specimen geometry | Rate (mm/s) | # of specimens tested |
|---|---|---|---|
| Biblis C base | 1TC(T) | 30<br>3 | 10<br>9 |
| | SE(B) 20/40 | 30<br>3 | 8<br>12 |
| | PCCv | 3 | 10 |
| Biblis C weld | SE(B) 20/40<br>PCCv | 3 | 10<br>5 |
| S690QL | 1TC(T) | 2.5<br>34<br>42 | 14<br>1<br>1 |

### 7.2. Choice of Test Rate

The actuator displacement rate has a direct effect on the fall time, the estimated bandwidth of the test system, the test time, and the minimum required bandwidth. For the NIST test system, and in particular its signal conditioner and data acquisition system, "higher" actuator displacement rates on the order of 30 mm/s (close to the value initially recommended to participants by the round-robin coordinator, U. Mayer [3]) resulted in minimum required bandwidths between 150 Hz and 2400 Hz. For the tests performed at NIST, estimated bandwidths were an order of magnitude lower, in the range 40 Hz to 75 Hz, irrespective of test rate or specimen type. Consequently, force values recorded in higher rate tests should be considered inaccurate.

On the other hand, for lower rate tests (actuator displacement rates = 2.5 mm/s - 3 mm/s), the minimum required bandwidths decrease by an order of magnitude to between 20 Hz - 115 Hz. Note that the minimum bandwidth is lower for "less brittle" tests, and higher for "more brittle" tests. For these lower rate tests and in case of "more brittle" behavior, it is still generally the case that $EBW < BW_{\min}$. However, based on a technical discussion with the round-robin coordinator, U. Mayer [15], it has been assumed that test results can be considered reliable if the estimated bandwidth is within 50 % to 100 % of the minimum value required by ASTM E1820.

## 7.3. Tests on Biblis C Base Material

### 7.3.1. Tests on 1TC(T) Specimens

#### 7.3.1.1. Higher Rate Tests

Ten specimens were tested between -40 °C and 0 °C at actuator displacement rates between 30.05 mm/s – 30.13 mm/s, corresponding to load-line displacement rates between 30.80 mm/s – 30.95 mm/s.[3] A summary of individual test results is provided in **Table 3**.

**Table 3** - Results obtained from higher rate tests on 1TC(T) specimens of Biblis C base material. (N/A = not available.)

| Specimen id | $T$ (°C) | Actuator rate (mm/s) | Load-line rate (mm/s) | Loading rate, $dK/dt$ (MPa√m/s) | $K_{Jc}$ (MPa√m) | $EBW$ (Hz) | $BW_{\min}$ (Hz) | $\frac{EBW}{BW_{min}}$ |
|---|---|---|---|---|---|---|---|---|
| L1.1 AA2 | -40 | 30.07 | 30.81 | 1.76E+03 | 67.09 | 64.1 | 1209.1 | **5%** |
| L1.1 AA1 | -40 | 30.13 | 30.95 | 8.34E+02 | 23.63 | 73.7 | 2397.1 | **3%** |
| L1.1 BB2 | -30 | 30.13 | 30.84 | 1.36E+03 | 46.46 | 65.9 | 2217.5 | **3%** |
| L1.1 AC3 | -20 | 45.18 | 18.81 | 1.50E+03 | 61.41 | N/A | N/A | N/A |
| L1.1 AA3 | -20 | 30.05 | 30.89 | 1.58E+03 | 61.87 | 63.0 | 1623.4 | **4%** |
| L1.1 BB6 | -15 | 30.06 | 30.94 | 1.42E+03 | 52.60 | 59.6 | 1673.6 | **4%** |
| L1.1 BB5 | -10 | 30.10 | 30.77 | 2.08E+03 | 103.83 | 52.6 | 611.2 | **9%** |
| L1.1 BB1 | -10 | 30.10 | 30.86 | 2.07E+03 | 117.20 | 52.2 | 472.9 | **11%** |
| L1.1 AC1 | -2 | 30.08 | 30.80 | 2.26E+03 | 141.40 | 51.1 | 371.5 | **14%** |
| L1.1 AC2 | 0 | 30.09 | 30.54 | 2.12E+03 | 109.92 | 52.8 | 532.8 | **10%** |

For all tests performed, the estimated bandwidth is less than 15 % of the minimum bandwidth required by ASTM E1820 Annex A14.

A valid[4] reference temperature, $T_{0,3}$ = -1.9 °C, was obtained by analyzing test results. Details of the Master Curve analyses (summarized in **Table 4**) are given in Appendix D.

Based on the SINTAP screening criterion of ASTM E1921, the material is judged to be potentially inhomogeneous, with a conservative estimated reference temperature $T_{0IN}$ = 12.8 °C. The adjusted Master Curve is shown in **Figure 5**. Four data points (40 % of the tested specimens) fall outside the 5 % and 95 % confidence limits, supporting the assumption that higher rate tests provide unreliable results. Furthermore, one of the tests performed at -40 °C yielded a suspiciously low $K_{Jc}$ value of 23.63 MPa√m, which is very close to the lower limit of toughness $K_{\min}$ = 20 MPa√m.

**Table 4** - Master Curve analysis results from higher rate tests on 1TC(T) specimens of Biblis C base material.

| Number of tests | Valid tests | $T_{0,3}$ (°C) | $\sum r_i n_i$ | Average loading rate (MPa√m/s) | $K_{med}$ (MPa√m) | $\sigma_{T0}$ (°C) |
|---|---|---|---|---|---|---|
| 10 | 10 | -1.9 | 1.5 | 1.70E+3 | 82.5 | 7.2 |

[3] Rates were calculated by linearly fitting displacement values as a function of time after inertial effects (time oscillations) have died down.
[4] The reference temperature is considered valid if the number of valid tests is at least 6.

LEGEND $\sum r_i n_i$ = sum of weighting factors for valid tests (needs to be ≥ 1 for $T_{0,X}$ to be valid).
$K_{med}$ = median value of fracture toughness for the data set.
$\sigma_{T0}$ = standard deviation of the calculated reference temperature.

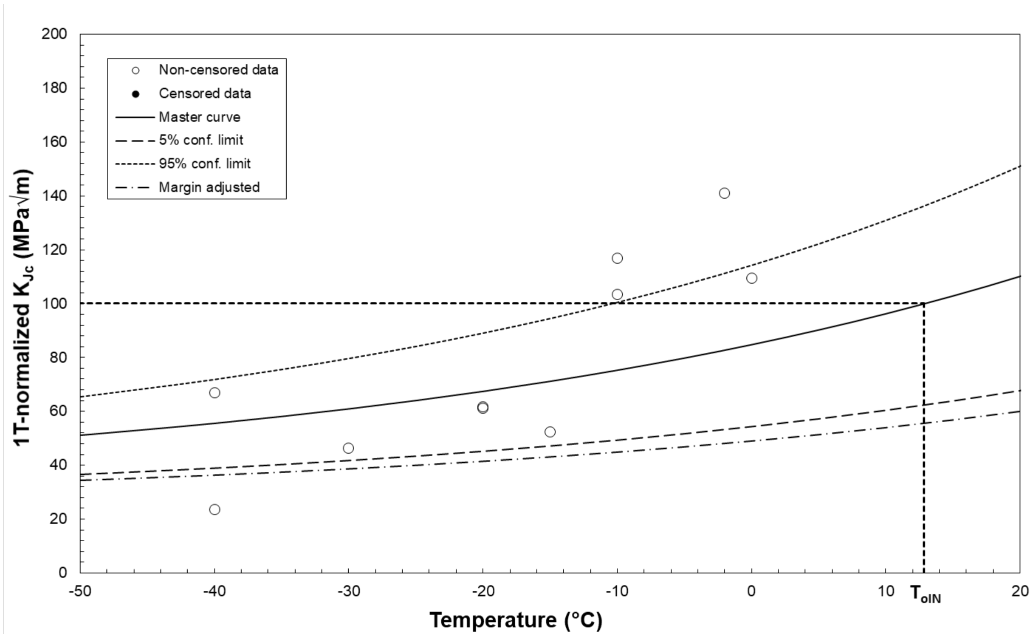


**Figure 5** – Inhomogeneous Master Curve analysis of the tests performed at higher rates on 1TC(T) specimens of Biblis C base material.

### 7.3.1.2. Lower Rate Tests

Nine specimens were tested between -20 °C and 0 °C at actuator displacement rates between 3.01 mm/s – 5.10 mm/s, corresponding to load-line displacement rates between 0.98 mm/s – 3.80 mm/s. A summary of individual test results is provided in **Table 5**.

For all tests performed, the estimated bandwidth is 57 % or more of the minimum bandwidth required by ASTM E1820 Annex A14; therefore, these tests can be considered reliable.

A valid reference temperature, $T_{0,2}$ = -25.9 °C, was obtained (24 °C below the value calculated for higher rate tests). The average loading rate is one order of magnitude lower than the tests conducted at higher rates. Details of the Master Curve analyses (summarized in **Table 6**) are given in Appendix E.

**Table 5** - Results obtained from lower rate tests on 1TC(T) specimens of Biblis C base material.

| Specimen id | *T* (°C) | Actuator rate (mm/s) | Load-line rate (mm/s) | Loading rate, *dK/dt* (MPa√m/s) | $K_{Jc}$ (MPa√m) | *EBW* (Hz) | $BW_{min}$ (Hz) | $\frac{EBW}{BW_{min}}$ |
|---|---|---|---|---|---|---|---|---|
| L1.3-AA5 | -20 | 5.03 | 1.67 | 3.21E+02 | 78.94 | 49.2 | 86.0 | **57%** |
| L1.3-BB7 | -20 | 3.01 | 1.03 | 2.00E+02 | 86.94 | 46.8 | 51.2 | **91%** |
| L1.2-BB7 | -18 | 3.01 | 0.98 | 2.02E+02 | 91.38 | 48.1 | 48.7 | **99%** |
| L1.3-BB1 | -10 | 5.03 | 1.67 | 3.49E+02 | 120.03 | 47.7 | 75.1 | **64%** |
| L1.2-BB3 | -10 | 5.06 | 2.11 | 2.11E+02 | 97.59 | 48.4 | 46.9 | **103%** |
| L1.3-AC4 | -5 | 3.01 | 1.22 | 2.31E+02 | 132.55 | 45.5 | 41.6 | **109%** |
| L1.3-AA2 | 0 | 5.10 | 3.80 | 4.59E+02 | 207.47 | 46.3 | 57.9 | **80%** |
| L1.2-AC2 | 0 | 3.01 | 1.27 | 2.77E+02 | 172.95 | 47.1 | 40.9 | **115%** |
| L1.3-AC6 | 0 | 3.01 | 1.46 | 2.71E+02 | 181.55 | 44.6 | 38.9 | **115%** |

**Table 6** - Master Curve analysis results from lower rate tests on 1TC(T) specimens of Biblis C base material.

| Number of tests | Valid tests | $T_{0,2}$ (°C) | $\sum r_i n_i$ | Average loading rate (MPa√m/s) | $K_{med}$ (MPa√m) | $\sigma_{T0}$ (°C) |
|---|---|---|---|---|---|---|
| 9 | 9 | -25.9 | 1.5 | 2.80E+2 | 127.2 | 7.2 |

The Master Curve is shown in **Figure 6**. Only one data point (11 % of tested specimens) falls outside the 5 % and 95 % confidence limits, which provides support to the reliability of the tests. Based on the SINTAP screening criterion of ASTM E1921, the material is judged to be homogeneous.

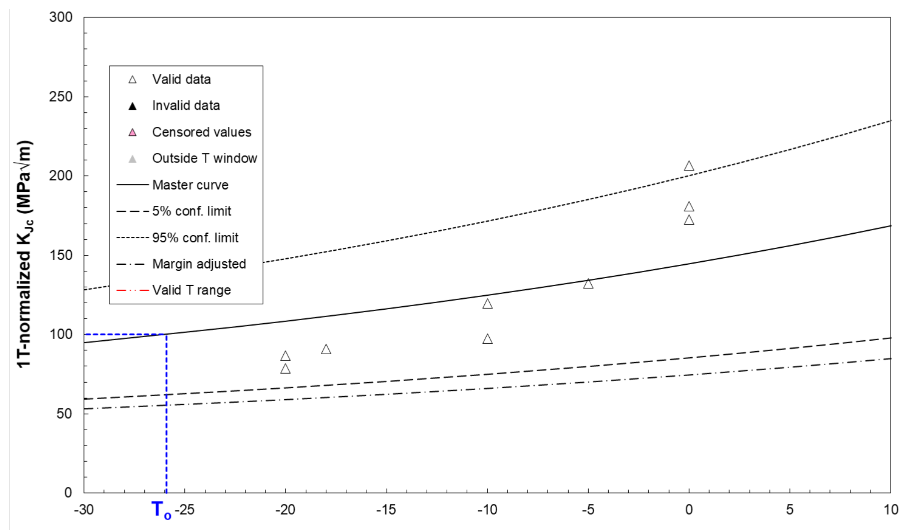


**Figure 6** – Homogeneous Master Curve analysis of the tests performed at lower rates on 1TC(T) specimens of Biblis C base material.

### 7.3.1.3. Combined Master Curve Analysis (Higher Rate and Lower Rate 1TC(T) Tests)

If all tests on 1TC(T) specimens are combined, the results of the Master Curve analysis summarized in **Table 7** and illustrated in **Figure 7** are obtained.

**Table 7** - Master Curve analysis results from all tests performed (higher and lower rates) on 1TC(T) specimens of Biblis C base material.

| Number of tests | Valid tests | $T_{0,X}$ (°C) | $\sum r_i n_i$ | $K_{med}$ (MPa√m) | $\sigma_{T0}$ (°C) |
|---|---|---|---|---|---|
| 19 | 19 | -18.1 | 3.1 | 107.3 | 5.7 |

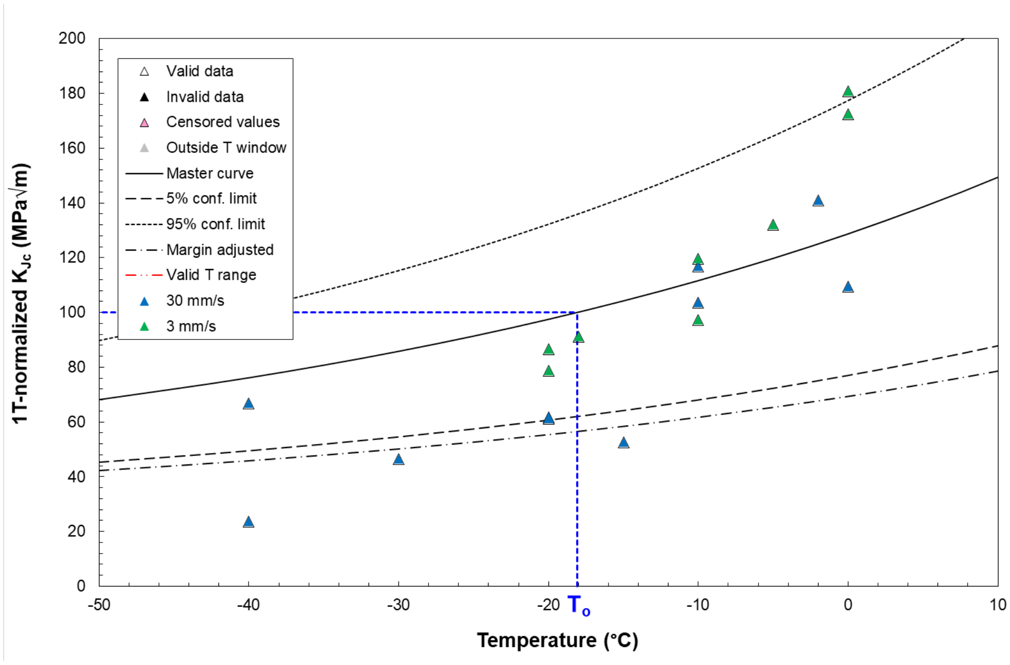


**Figure 7** – Homogeneous Master Curve analysis of all 1TC(T) tests performed on Biblis C base material.

As could be expected from the "hybrid" nature of this combined data set, the outcome of the SINTAP screening criterion classifies the material as inhomogeneous, with an adjusted reference temperature $T_{0IN}$ = -8.7 °C. Even after this conservative adjustment, 5 data points out of 19 (26 %) fall outside the 5 % and 95 % confidence limits (**Figure 8**).

It's also worth noting that the reference temperature obtained for the combined data set ($T_{0,X}$ = -18.1 °C) is much closer to that calculated for lower rate tests (-25.9 °C) than for higher rate tests (-1.9 °C). This shows that the lower rates tests have a larger weight in determining the overall reference temperature.

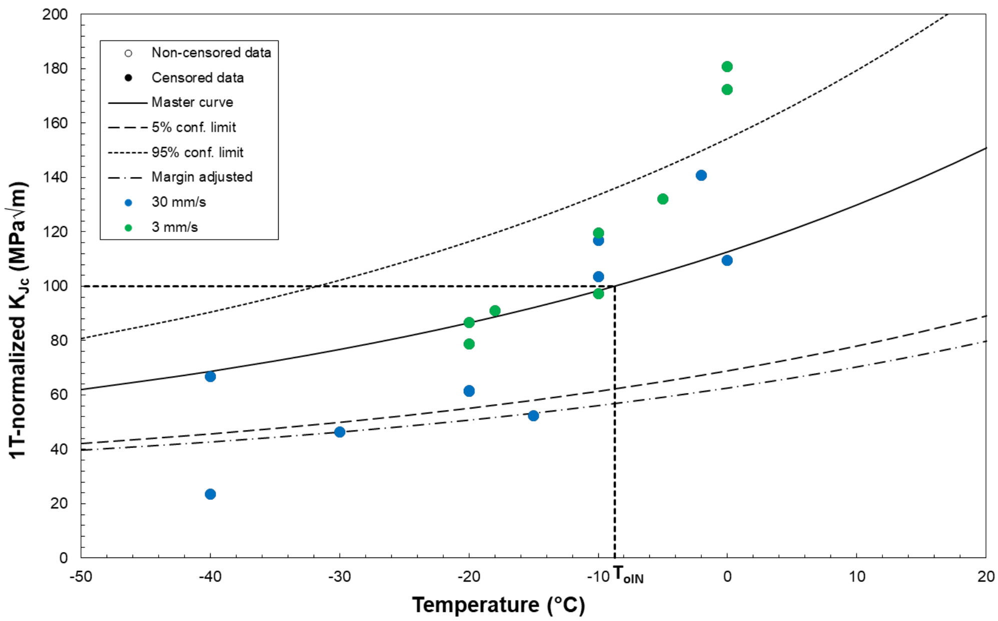


**Figure 8** – Inhomogeneous Master Curve analysis of all 1TC(T) tests performed on Biblis C base material.

### 7.3.2. Tests on SE(B) 20/40 Specimens

#### 7.3.2.1. Higher Rate Tests

Eight specimens were tested between -20 °C and 12 °C at actuator displacement rates between 31.21 mm/s – 44.86 mm/s, corresponding to crack-mouth opening displacement (CMOD) rates between 13.17 mm/s – 25.06 mm/s. A summary of individual test results is provided in **Table 8**.

**Table 8** - Results obtained from higher rate tests on SE(B) 20/40 specimens of Biblis C base material.

| Specimen id | $T$ (°C) | Actuator rate (mm/s) | CMOD rate (mm/s) | Loading rate, $dK/dt$ (MPa√m/s) | $K_{Jc}$ (MPa√m) | $EBW$ (Hz) | $BW_{min}$ (Hz) | $\frac{EBW}{BW_{min}}$ |
|---|---|---|---|---|---|---|---|---|
| 4.1 B11 | -20 | 44.86 | 13.17 | 1.52E+03 | 63.94 | 59.6 | 1260.1 | **5%** |
| 3.1 B01 | -10 | 43.70 | 17.66 | 2.17E+03 | 101.79 | 49.6 | 971.8 | **5%** |
| 4.1 A11 | -10 | 44.47 | 15.68 | 1.81E+03 | 77.61 | 57.4 | 1174.7 | **5%** |
| 3.1 B04 | 0 | 42.08 | 14.46 | 1.56E+03 | 60.88 | 54.3 | 1444.3 | **4%** |
| 4.1 A09 | 0 | 42.36 | 17.08 | 2.05E+03 | 94.27 | 34.8 | 1006.3 | **3%** |
| 4.1 B01 | 5 | 36.42 | 25.06 | 2.89E+03 | 192.14 | 41.6 | 393.2 | **11%** |
| 3.1 B10 | 10 | 33.35 | 24.87 | 2.95E+03 | 230.35 | 39.9 | 241.9 | **17%** |
| 4.1 B07 | 12 | 31.21 | 22.83 | 2.76E+03 | 265.48 | 39.4 | 147.6 | **27%** |

For all tests performed, the estimated bandwidth is less than 28 % of the minimum bandwidth required by ASTM E1820 Annex A14.

A valid reference temperature, $T_{0,3}$ = -26.9 °C, was obtained by analyzing test results. Details of the Master Curve analyses (summarized in **Table 9**) are given in Appendix F.

**Table 9** - Master Curve analysis results from higher rate tests on SE(B) 20/40 specimens of Biblis C base material.

| Number of tests | Valid tests | $T_{0,3}$ (°C) | $\sum r_i n_i$ | Average loading rate (MPa√m/s) | $K_{med}$ (MPa√m) | $\sigma_{T0}$ (°C) |
|---|---|---|---|---|---|---|
| 8 | 8 | -26.9 | 1.2 | 2.21E+3 | 140.9 | 7.9 |

Based on the SINTAP screening criterion of ASTM E1921, the material is judged to be potentially inhomogeneous, with a conservative estimated reference temperature $T_{0IN}$ = 12.9 °C. This value is almost identical to the adjusted reference temperature calculated for 1TC(T) specimens at higher loading rate (12.8 °C). The adjusted Master Curve is shown in **Figure 9**. Three data points (38 % of the tested specimens) fall outside the 5 % and 95 % confidence limits, again supporting the assumption that these higher rate tests provide unreliable results. All the "outlier" tests correspond to fracture toughness values much higher than the 95 % confidence limit.

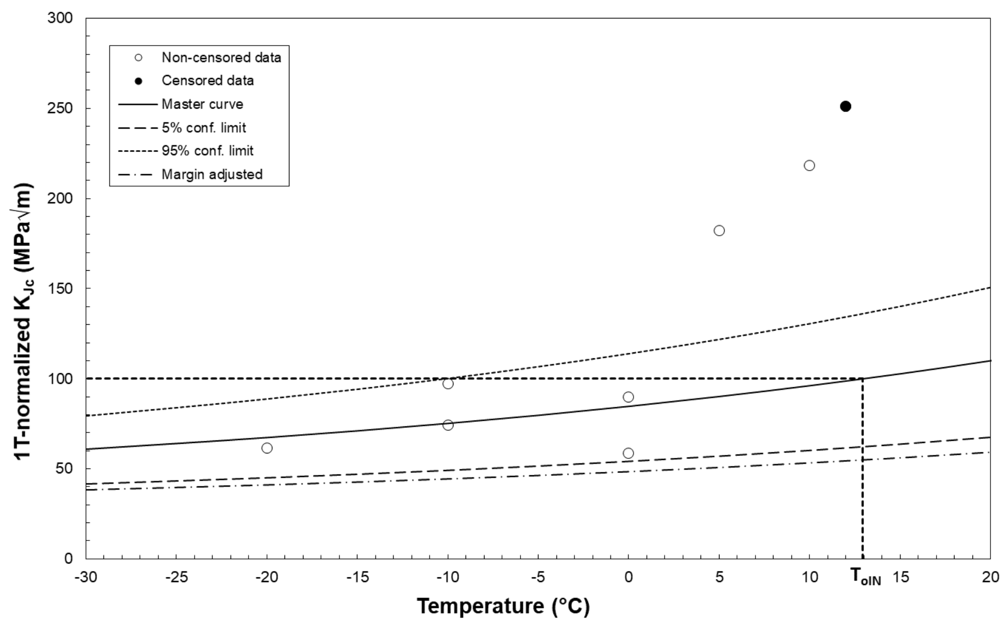


**Figure 9** – Inhomogeneous Master Curve analysis of the tests performed at higher rates on SE(B) 20/40 specimens of Biblis C base material.

#### 7.3.2.2. Lower Rate Tests

Twelve specimens were tested between -50 °C and 5 °C at actuator displacement rates between 3.00 mm/s – 3.04 mm/s, corresponding to CMOD rates between 1.25 mm/s – 2.22 mm/s. A summary of individual test results is provided in **Table 10**.

**Table 10** - Results obtained from lower rate tests on SE(B) 20/40 specimens of Biblis C base material.

| Specimen id | $T$ (°C) | Actuator rate (mm/s) | CMOD rate (mm/s) | Loading rate, $dK/dt$ (MPa√m/s) | $K_{Jc}$ (MPa√m) | $EBW$ (Hz) | $BW_{min}$ (Hz) | $\frac{EBW}{BW_{min}}$ |
|---|---|---|---|---|---|---|---|---|
| 4.1 A13 | -50 | 3.03 | 1.31 | 3.11E+02 | 70.87 | 43.9 | 96.3 | **46%** |
| 3.1 B02 | -45 | 3.03 | 1.25 | 2.82E+02 | 55.13 | 41.2 | 114.8 | **36%** |
| 3.1 B08 | -40 | 3.00 | 1.34 | 3.14E+02 | 84.67 | 42.7 | 85.7 | **50%** |
| 4.2 A08 | -40 | 3.01 | 2.22 | 3.14E+02 | 84.67 | 37.4 | 20.0 | **187%** |
| 4.2 B03 | -35 | 3.00 | 1.41 | 3.34E+02 | 104.37 | 46.1 | 79.0 | **58%** |
| 3.1 B05 | -30 | 3.04 | 1.59 | 3.56E+02 | 134.60 | 42.9 | 57.4 | **75%** |
| 4.1 B03 | -25 | 3.03 | 1.53 | 3.46E+02 | 130.09 | 43.5 | 60.4 | **72%** |
| 4.1 B09 | -20 | 3.02 | 1.57 | 3.59E+02 | 132.59 | 45.5 | 59.7 | **76%** |
| 3.1 B13 | -15 | 3.03 | 1.95 | 3.54E+02 | 142.35 | 43.9 | 53.4 | **82%** |
| 3.1 B11 | -10 | 3.03 | 1.59 | 3.49E+02 | 133.06 | 40.7 | 60.5 | **67%** |
| 3.1 B07 | -5 | 3.01 | 2.04 | 3.46E+02 | 196.09 | 39.1 | 29.6 | **132%** |
| 4.2 A01 | 5 | 3.01 | 2.15 | 3.44E+02 | 218.17 | 30.5 | 24.2 | **126%** |

The two tests performed at the lowest temperatures produced an estimated bandwidth somewhat lower than 50 % of the minimum required bandwidth.

A valid reference temperature, $T_0$ = -31.0 °C, was obtained by analyzing all 12 test results (only 4.1 °C lower than the value calculated for higher rate tests). If the 2 lowest temperature tests (for which $EBW$ < 50 % $BW_{min}$) are removed, the reference temperature decreases by 2.1 °C ($T_0$ = -33.1 °C). The average loading rate is one order of magnitude lower than for the tests conducted at higher rates. Details of both Master Curve analyses are summarized in **Table 11**, while details of the analysis on the 10 fully valid tests are provided in Appendix G.

**Table 11** - Master Curve analyses results from lower rate tests on 1TC(T) specimens of Biblis C base material.

| Number of tests | Valid tests | $T_{0,2}$ (°C) | $\sum r_i n_i$ | Average loading rate (MPa√m/s) | $K_{med}$ (MPa√m) | $\sigma_{T0}$ (°C) | Notes |
|---|---|---|---|---|---|---|---|
| 12 | 12 | -31.0 | 2.0 | 3.34E+02 | 111.2 | 6.6 | All tests |
| 10 | 10 | -33.1 | 1.7 | 3.42E+02 | 120.7 | 7.0 | Only tests with $EBW$ ≥ 50 % $BW_{min}$ |

The Master Curves for the full data set is shown in **Figure 10**. Only one data point falls marginally below the 5 % confidence limit. This data point corresponds to one of the two lowest test temperatures, hence if those two tests are removed all data points are enveloped by the 5 % - 95 % confidence limits.

Based on the SINTAP screening criterion of ASTM E1921, the material is screened to be homogeneous.

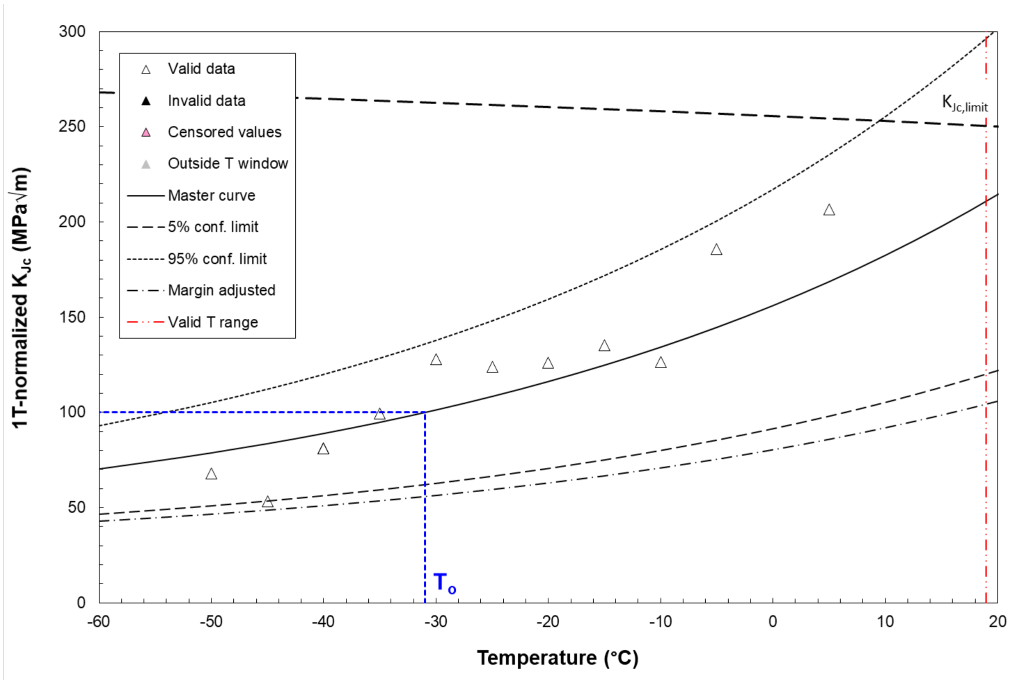


**Figure 10** – Homogeneous Master Curve analysis of the tests performed at lower rates on SE(B) 20/40 (all tests) specimens of Biblis C base material. For the two lowest temperatures, tests have estimated bandwidths lower than 50 % of the required minimum bandwidth.

### 7.3.2.3. Combined Master Curve Analysis (Higher Rate and Lower Rate SE(B) Tests)

If all tests on SE(B) 20/40 specimens are combined, the results of the Master Curve analysis summarized in **Table 12** and illustrated in **Figure 11** are obtained.

Considering all the Master Curve analyses conducted on SE(B) 20/40 tests (faster tests, slower tests, all tests), all calculated reference temperatures were within 6.2 °C (between -26.9 °C and -33.1 °C).

Consistent with this narrow reference temperature range, the SINTAP analysis screens the overall data set as homogeneous.

**Table 12** - Master Curve analysis results from all tests performed (higher and lower rates) on SE(B) 20/40 specimens of Biblis C base material.

| Number of tests | Valid tests | $T_{0,X}$ (°C) | $\sum r_i n_i$ | $K_{med}$ (MPa√m) | $\sigma_{T0}$ (°C) |
|---|---|---|---|---|---|
| 20 | 19 | -29.6 | 3.1 | 122.8 | 5.7 |

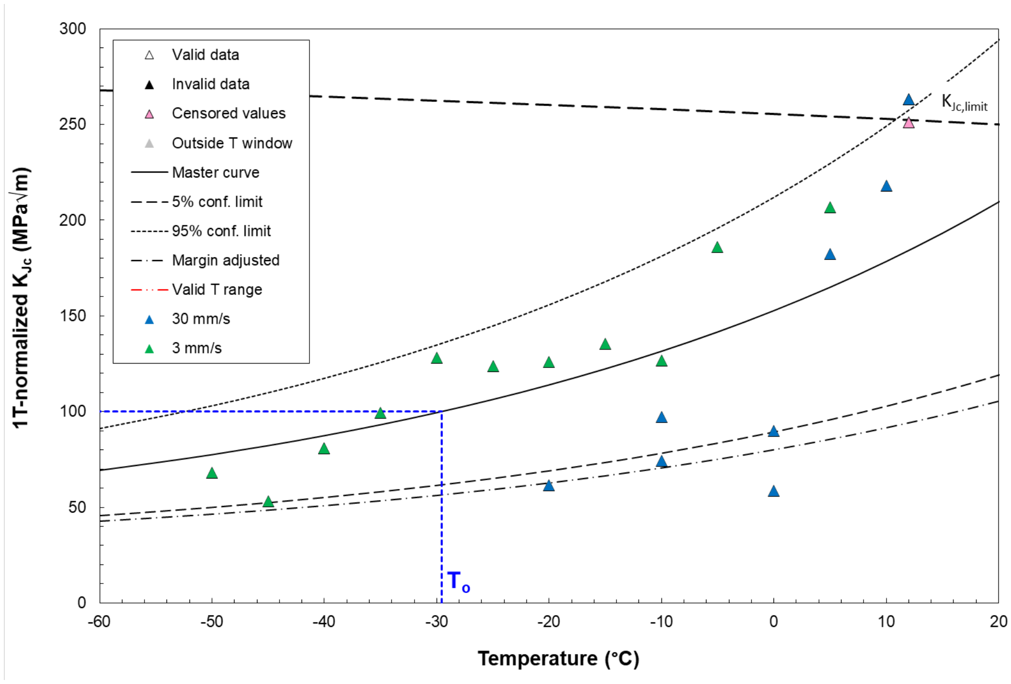


**Figure 11** – Homogeneous Master Curve analysis of all SE(B) 20/40 tests performed on Biblis C base material.

### 7.3.3. Additional Master Curve Analyses

#### 7.3.3.1. All Higher Rate Tests (1TC(T) and SE(B) 20/40)

It is well established [15,16] that median $K_{Jc}$ values tend to vary with the specimen type at a given test temperature, due to constraint differences between compact tension and single-edge bend specimens. This $K_{Jc}$ dependency ultimately leads to discrepancies in calculated $T_0$ values as a function of specimen type for the same material. Specifically, $T_0$ values obtained from C(T) specimens are expected to be higher than $T_0$ values obtained from SE(B) specimens, as stress triaxiality and crack-tip constraint are higher for mixed tension/bending loading (compact specimens) than for pure bending loading (single-edge bend specimens). Best estimate comparisons of several materials indicate that the average difference between C(T)- and SE(B)-derived $T_0$ values is approximately 10 °C [15], with differences up to 15 °C also reported [16].

For the tests performed at NIST on 1TC(T) and SE(B) 20/40 specimens at higher rates (between 30 mm/s and 45 mm/s), the calculated $T_{0,3}$ values followed the expected trend, with C(T) specimens yielding a higher reference temperature than SE(B) specimens. In this case, however, the difference (25 °C) is larger than what typically reported.

A combined Master Curve analysis of the 1TC(T) and SE(B) higher rate data sets provided the results summarized in **Table 13** and illustrated in **Figure 12**.

As expected, the large difference between $T,3_0$ calculated from 1TC(T) and SE(B) 20/40 specimens (25 °C) causes the overall data set to be screened as inhomogeneous by the SINTAP approach.

**Table 13** - Master Curve analysis results from all tests performed at higher rates on 1TC(T) and SE(B) 20/40 specimens of Biblis C base material.

| Number of tests | Valid tests | $T_{0,3}$ (°C) | $\sum r_i n_i$ | $K_{med}$ (MPa√m) | $\sigma_{T0}$ (°C) |
|---|---|---|---|---|---|
| 18 | 17 | -17.8 | 2.8 | 110.2 | 5.9 |

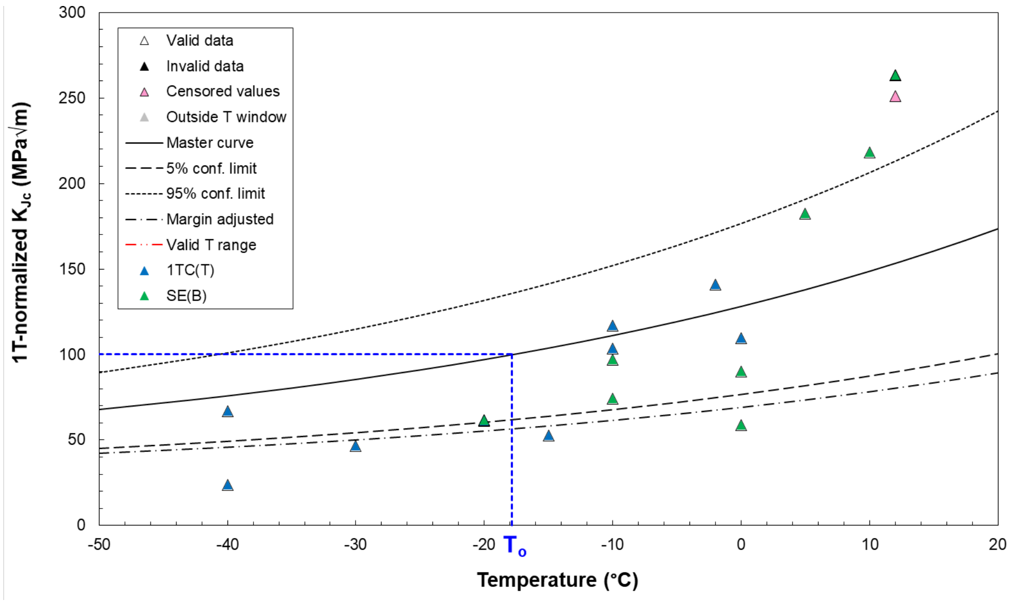


**Figure 12** – Homogeneous Master Curve analysis of all 1TC(T) and SE(B) 20/40 tests performed at higher rates on Biblis C base material.

### 7.3.3.2. All Lower Rate Tests (1TC(T) and SE(B) 20/40)

When only tests conducted at lower rates (3 mm/s) on 1TC(T) and SE(B) specimens are considered, the specimen geometry effect is confirmed, with compact tension specimens yielding a 7.2 °C higher reference temperature than single-edge bend specimens (-33.1 °C vs. -25.9 °C).

A combined Master Curve analysis of the 1TC(T) and SE(B) lower rate data sets provided the results summarized in **Table 14** and illustrated in **Figure 13**.

**Table 14** - Master Curve analysis results from all tests performed at lower rates on 1TC(T) and SE(B) 20/40 specimens of Biblis C base material.

| Number of tests | Valid tests | $T_{0,2}$ (°C) | $\sum r_i n_i$ | $K_{med}$ (MPa√m) | $\sigma_{T0}$ (°C) |
|---|---|---|---|---|---|
| 21 | 21 | -29.0 | 3.5 | 118.8 | 5.6 |

Interestingly, the data set screens homogeneous based on the SINTAP approach. Most likely, even though two separate subsets are present (1TC(T) and SE(B) 20/40), the difference in $T_0$ (7.2 °C) is not large enough to trigger a verdict of inhomogeneity from the SINTAP methodology.

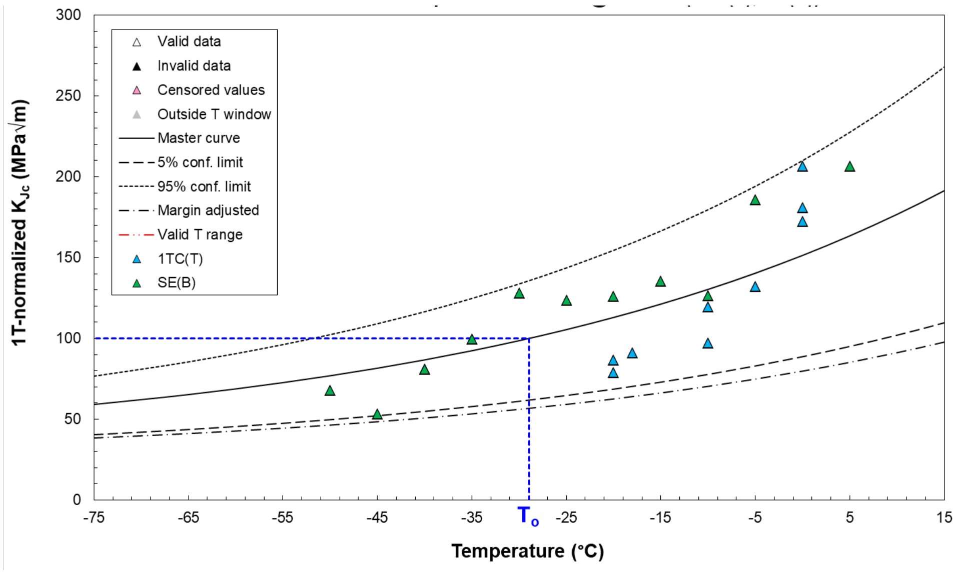


**Figure 13** – Homogeneous Master Curve analysis of all 1TC(T) and SE(B) 20/40 tests performed at lower rates on Biblis C base material.

## 7.4. Tests on Biblis C Weld Material (SE(B) 20/40 specimens)

Ten SE(B) 20/40 specimens were tested between -75 °C and -40 °C at actuator displacement rates between 3.00 mm/s – 3.04 mm/s, corresponding to CMOD rates between 1.30 mm/s – 2.20 mm/s. A summary of individual test results is provided in **Table 15**.

**Table 15** - Results obtained from tests on SE(B) 20/40 specimens of Biblis C weld material.

| Specimen id | $T$ (°C) | CMOD rate (mm/s) | Load-line rate (mm/s) | Loading rate, $dK/dt$ (MPa√m/s) | $K_{Jc}$ (MPa√m) | $EBW$ (Hz) | $BW_{\min}$ (Hz) | $\frac{EBW}{BW_{min}}$ |
|---|---|---|---|---|---|---|---|---|
| SV2D3 | -75 | 3.01 | 1.30 | 3.17E+02 | 94.42 | 49.2 | 73.4 | **67%** |
| SV2C6 | -65 | 3.00 | 1.65 | 3.47E+02 | 90.24 | 48.5 | 75.8 | **64%** |
| SV2C2 | -60 | 3.03 | 1.40 | 3.51E+02 | 123.28 | 46.2 | 65.2 | **71%** |
| SV2B9 | -55 | 3.02 | 2.14 | 3.83E+02 | 255.81 | 45.9 | 25.7 | **178%** |
| SV2D7 | -54 | 3.03 | 1.30 | 3.50E+02 | 127.67 | 46.9 | 60.0 | **78%** |
| SV2C10 | -50 | 3.04 | 1.42 | 3.31E+02 | 105.43 | 46.3 | 72.9 | **63%** |
| SV2A4 | -50 | 3.02 | 1.73 | 3.77E+02 | 197.23 | 44.9 | 38.7 | **116%** |
| SV2B5 | -45 | 3.02 | 1.74 | 3.69E+02 | 202.96 | 47.3 | 36.2 | **130%** |
| SV2B01 | -45 | 3.02 | 2.17 | 3.76E+02 | 243.30 | 47.4 | 27.6 | **172%** |
| SV2A08 | -40 | 3.01 | 2.20 | 3.54E+02 | 300.32 | 41.2 | 18.4 | **224%** |

For all tests performed, the estimated bandwidth is above 50 % of the minimum bandwidth required by ASTM E1820 Annex A14. Therefore, all tests can be considered reliable.

A valid reference temperature, $T_{0,2}$ = -89.6 °C, was obtained by analyzing test results. Details of the Master Curve analyses (summarized in **Table 16**) are given in Appendix H.

**Table 16** - Master Curve analysis results from tests performed on SE(B) 20/40 specimens of Biblis C weld material.

| Number of tests | Valid tests | $T_{0,2}$ (°C) | $\sum r_i n_i$ | Average loading rate (MPa√m/s) | $K_{med}$ (MPa√m) | $\sigma_{T0}$ (°C) |
|---|---|---|---|---|---|---|
| 10 | 10 | -89.6 | 1.7 | 3.55E+2 | 170.2 | 7.0 |

Based on the SINTAP screening criterion of ASTM E1921, the material is judged to be potentially inhomogeneous, with a conservative estimated reference temperature $T_{0IN}$ = -77.5 °C. The adjusted Master Curve is shown in **Figure 14**.

In the homogeneous analysis, two data points (20 % of the tested specimens) fall outside the 5 % and 95 % confidence limits. This number increases to 3 (30 %) for the inhomogeneous analysis shown in **Figure 14**.

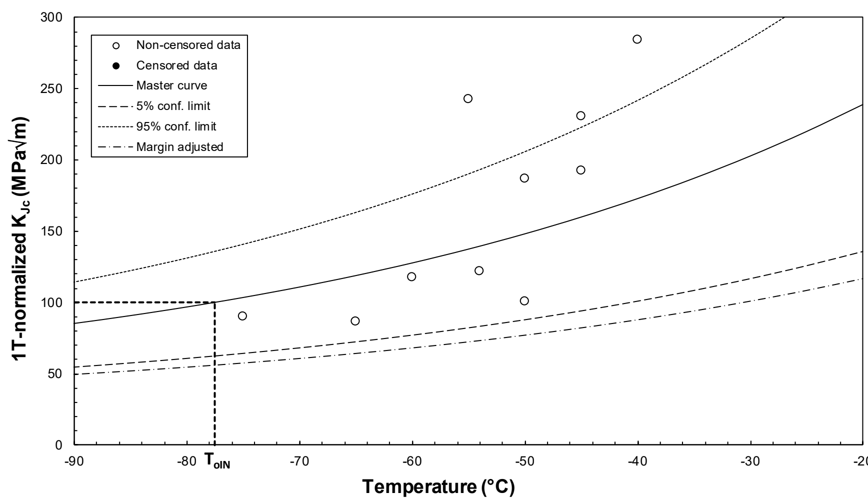


**Figure 14** – Inhomogeneous Master Curve analysis of the tests performed on SE(B) 20/40 specimens of Biblis C weld material.

## 7.5. Tests on S690QL steel (1TC(T) specimens)

Two series of 8 1TC(T) specimens, for a total of 16 specimens, were tested between -75 °C and -10 °C. The first two specimens of the first series (AA09 and BE09) were tested at “fast” actuator displacement rates, 41.73 mm/s and 32.50 mm/s, respectively. The remaining 14 specimens were tested at lower actuator displacement rates, in the range 2.36 mm/s – 2.55 mm/s. A summary of individual test results for all tests performed is given in **Table 17**.

**Table 17** - Results obtained from tests on 1TC(T) specimens of S690QL steel.

| **Specimen id** | ***T* (°C)** | **Actuator rate (mm/s)** | **Load-line rate (mm/s)** | **Loading rate, *dK/dt* (MPa√m/s)** | **$K_{Jc}$ (MPa√m)** | ***EBW* (Hz)** | **$BW_{min}$ (Hz)** | **$\frac{EBW}{BW_{min}}$** |
|---|---|---|---|---|---|---|---|---|
| AA09 | -60 | 41.73 | 13.72 | 1.64E+03 | 78.57 | 54.7 | 622.3 | **9%** |
| BE09 | -50 | 32.50 | 10.98 | 1.95E+03 | 150.23 | 51.2 | 256.0 | **20%** |
| BB09 | -50 | 2.51 | 0.92 | 1.68E+02 | 146.17 | 47.0 | 28.6 | **164%** |
| AB12 | -45 | 2.50 | 0.81 | 1.86E+02 | 107.47 | 48.0 | 34.7 | **138%** |
| AA03 | -45 | 2.43 | 0.71 | 1.30E+02 | 63.20 | 49.2 | 56.2 | **88%** |
| BD06 | -40 | 2.50 | 0.78 | 1.82E+02 | 93.32 | 41.1 | 37.4 | **110%** |
| AC09 | -40 | 2.51 | 0.87 | 1.48E+02 | 112.97 | 48.4 | 34.8 | **139%** |
| AB06 | -35 | 2.36 | 0.71 | 1.26E+02 | 63.83 | 49.5 | 51.9 | **95%** |
| BC03 | -32 | 2.50 | 0.83 | 1.69E+02 | 114.82 | 43.8 | 33.0 | **133%** |
| BE03 | -30 | 2.42 | 0.79 | 1.40E+02 | 84.55 | 40.1 | 43.0 | **93%** |
| BC12 | -27 | 2.53 | 0.87 | 1.68E+02 | 137.27 | 47.5 | 29.2 | **163%** |
| AE06 | -25 | 2.54 | 0.85 | 1.80E+02 | 116.05 | 49.8 | 32.8 | **152%** |
| AD03 | -20 | 2.51 | 0.88 | 2.08E+02 | 178.07 | 48.4 | 24.6 | **197%** |
| BA06 | -20 | 2.55 | 0.98 | 1.78E+02 | 191.32 | 47.6 | 22.9 | **208%** |
| BA12 | -15 | 2.51 | 0.94 | 2.05E+02 | 187.75 | 45.2 | 22.7 | **199%** |
| AD12 | -10 | 2.41 | 0.76 | 1.22E+02 | 68.57 | 49.3 | 52.7 | **94%** |

For the first two tests, the estimated bandwidth was much lower than half of the required minimum bandwidth. The remaining 14 tests, for which the ratio was higher than 88 %, can be considered reliable.

Upon excluding the first two tests, a valid reference temperature, $T_{0,2}$ = -46.9 °C, was obtained by analyzing the 14 tests conducted at 2.5 mm/s actuator rate. Details of these Master Curve analyses (summarized in **Table 16**) are given in Appendix I.

**Table 18** - Master Curve analysis results from tests performed at 2.5 mm/s on 1TC(T) specimens of S690QL steel.

| **Number of tests** | **Valid tests** | **$T_{0,2}$ (°C)** | $\sum r_i n_i$ | **Average loading rate (MPa√m/s)** | **$K_{med}$ (MPa√m)** | **$\sigma_{T0}$ (°C)** |
|---|---|---|---|---|---|---|
| 14 | 14 | -46.9 | 2.3 | 1.19E+2 | 127.1 | 6.3 |

Based on the SINTAP screening criterion of ASTM E1921, the material is judged to be homogeneous. The obtained Master Curve is illustrated in **Figure 15**, which shows that 4 data points (29 %) fall outside the 5 % - 95 % confidence limits.

If the two higher rate tests are included in the analysis, the reference temperature decreases by 3.4 °C ($T_{0,2}$ = -50.3 °C), and one additional data point falls outside the 5 % - 95 % confidence limits. The overall data set remains homogeneous.

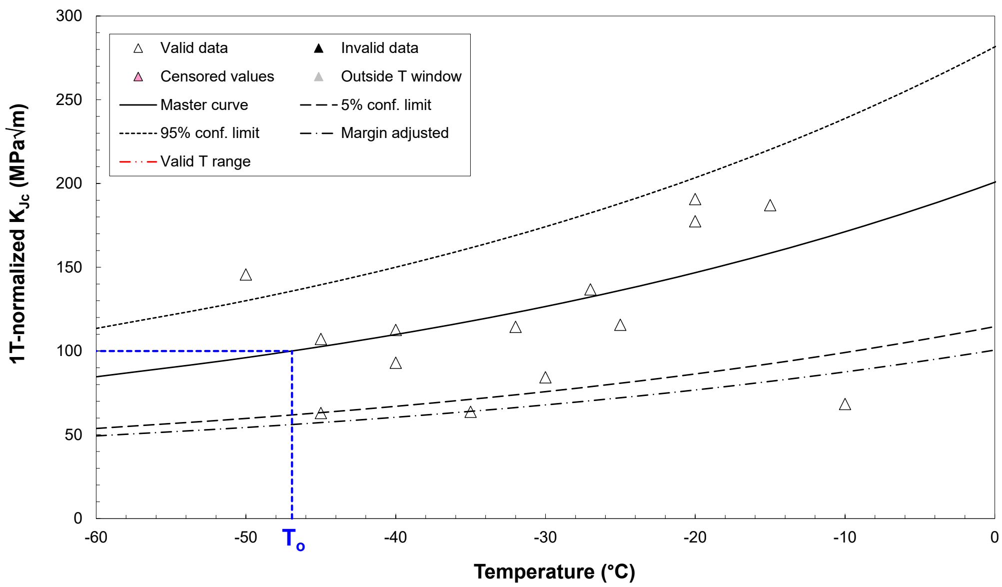


**Figure 15** – Homogeneous Master Curve analysis of the 14 tests performed at 2.5 mm/s on 1TC(T) specimens of S690QL steel.

## 8. Technical Discussion

### 8.1. Influence of Loading Rate on Reference Temperatures

It has long been established [18-21] that, in the brittle and ductile-to-brittle fracture regimes, an increase in loading rate causes a decrease in the fracture toughness of steels, and therefore an increase in transition (reference) temperature. The opposite occurs under fully ductile conditions, where increasing loading rate enhances the fracture toughness of steels [20,22].

Within this investigation, fracture toughness tests at NIST were initially performed at actuator displacement rates in the order of 30 mm/s – 40 mm/s, following recommendations from the round-robin coordinator, U. Mayer of MPA Stuttgart [3]. Analysis of these 20 early tests (10 tests on 1TC(T) specimens and 8 tests on SE(B) 20/40 specimens of Biblis C base material, and 2 tests on 1TC(T) specimens of S690QL steel) revealed that the frequency bandwidth of the force measuring system, estimated from the fracture time, was one or two orders of magnitude lower than the minimum bandwidth required by ASTM E1820 ($10/t_Q$).

For the 65 tests performed at NIST on 1TC(T) and SE(B) 20/40 specimens, *EBW* values ranged between 30.5 Hz and 73.7 Hz, and exhibited a moderate decreasing trend with increasing 1T-normalized toughness (**Figure 16**) and a slight increasing trend with increasing actuator displacement rate (**Figure 17**).

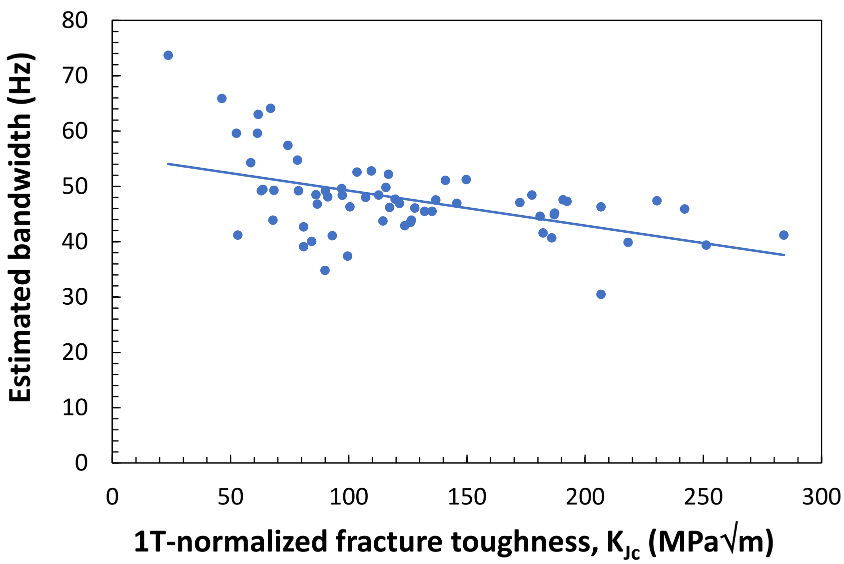


**Figure 16** - Estimated bandwidth for the NIST tests as a function of measured 1T-normalized fracture toughness. NOTE: the regression line shown does not represent an analytical relationship, but is just a guide for the eye showing the overall trend.

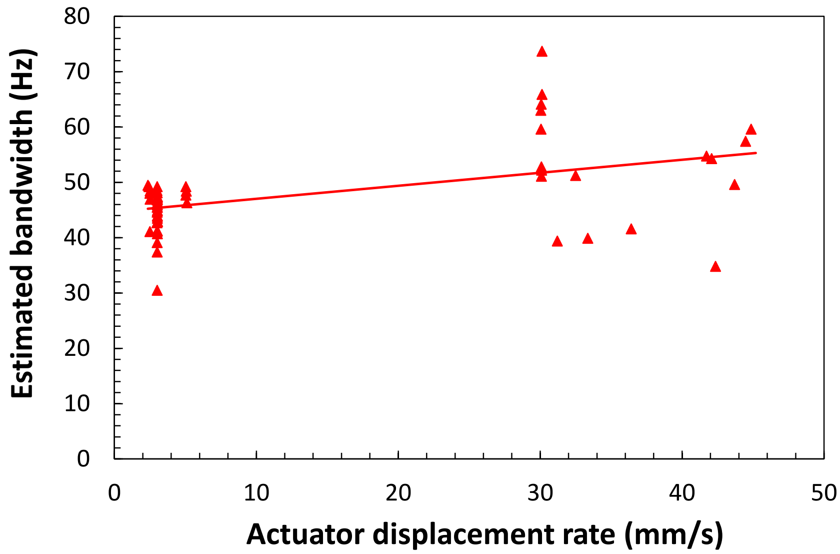


**Figure 17** - Estimated bandwidth for the NIST tests as a function of actuator displacement rate. NOTE: the regression line shown does not represent an analytical relationship, but is just a guide for the eye showing the overall trend.

Conversely, the minimum required bandwidth decreases with 1T-normalized fracture toughness (**Figure 18**) and increases with actuator displacement rate (**Figure 19**).

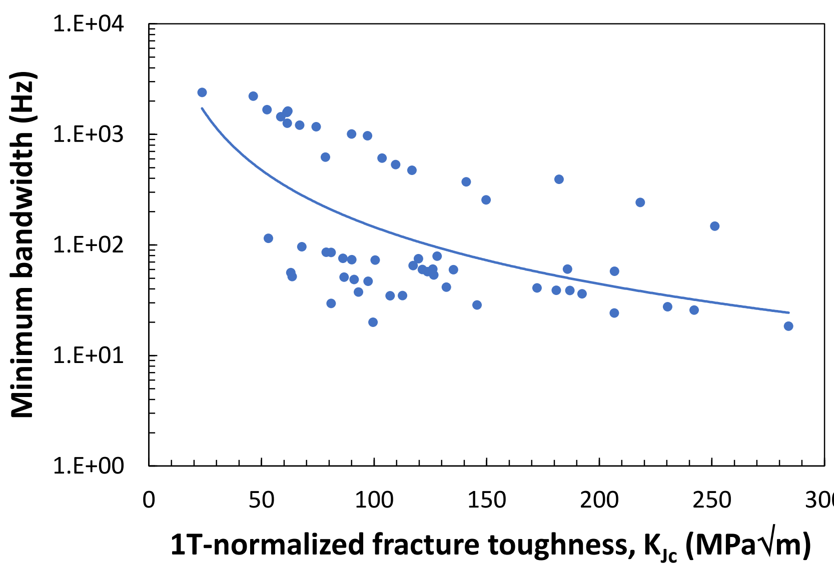


**Figure 18** – Log-linear plot of minimum required bandwidth as a function of measured 1T-normalized fracture toughness for the NIST tests. NOTE: the regression line shown does not represent an analytical relationship, but is just a guide for the eye showing the overall trend.

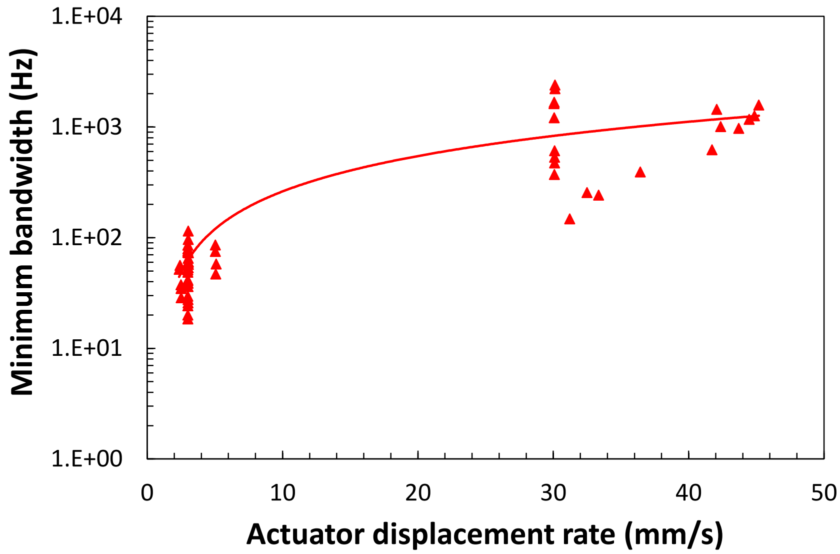


**Figure 19** – Log-linear plot of minimum required bandwidth as a function of actuator displacement rate for the NIST tests. NOTE: the regression curve shown does not represent an analytical relationship, but is just a guide for the eye showing the overall trend.

According to Annex A14 of ASTM E1820, the signal conditioner of the force signal must have a frequency bandwidth in excess of $10/t_Q$ to obtain "*an accurate measurement of the elastic component of the J-integral near crack initiation.*" Namely, the elastic component of $J$ is calculated from the force at unstable fracture, and a too low frequency bandwidth might cause an overestimation of the fracture force (overshoot), and therefore an overestimation of the fracture toughness. For tests conducted with $EBW \ll BW_{\min}$, one would therefore expect to calculate a Master Curve reference temperature which is lower than the "true" value.

However, based on technical discussions with the round-robin coordinator, U. Mayer, tests performed in this study were considered reliable whenever the estimated frequency bandwidth of the force measuring system exceeded $5/t_Q$, or $EBW > 50\ \%\ BW_{\min}$ if the ASTM definition of $BW_{\min}$ is retained.

The remaining 45 NIST tests were therefore performed at actuator displacement rates in the range 2.5 mm/s – 3 mm/s, which corresponded to minimum required bandwidths between 18.4 Hz and 114.8 Hz, depending on the level of toughness. For 43 of these tests, the estimated bandwidth was at least 50 % of the minimum required bandwidth. For the two remaining tests, the ratio $EBW/BW_{\min}$ was relatively close to 50 % (36 % and 46 %, respectively).

The effect of varying the loading rate on the calculated Master Curve reference temperature is shown in **Table 19** for Biblis C base material and S690QL.

**Table 19** - Effect of loading rate on $T_{0,X}$ for Biblis C base material and S690QL.

| Steel | Specimen type | Number of tests | Average loading rate (MPa√m/s) | Average $EBW/BW_{min}$ | $T_{0,X}$ (°C) | $T_{0,X}$ valid?[5] | $\Delta T_{0,X}$ (°C) |
|---|---|---|---|---|---|---|---|
| Biblis C base | 1TC(T) | 10 | 1.70E+3 | 7 % | -1.9 | YES | 24.0 |
| | | 9 | 2.80E+2 | 93 % | -25.9 | YES | |
| | SE(B) 20/40 | 8 | 2.21E+3 | 9 % | -26.9 | YES | 6.2 |
| | | 10 | 3.42E+3 | 93 % | -33.1 | YES | |
| S690QL | 1TC(T) | 2 | 1.79E+3 | 14 % | -64.4 | NO | 17.5 |
| | | 14 | 1.19E+2 | 141 % | -46.9 | YES | |

In all cases, increasing the loading rate yields an increase of $T_0$ (apparent material embrittlement), which is in line with expectations. However, the magnitude of this increase ($\Delta T_{0,X}$ in **Table 19**) is very different between the two 1TC(T) data sets (Biblis C base and S690QL) and the SE(B) data set (Biblis C base). Furthermore, no clear evidence is observed for the potential toughness overestimation caused by having an estimated bandwidth much lower than the required minimum.

The contrasting effects of increasing loading rate and insufficient bandwidth are impossible to deconvolute in this case, and most likely contribute to the spread of $\Delta T_{0,X}$ for the different steels and specimen types.

Annex A1 of ASTM E1921 (*Special Requirements for Determining the Reference Temperature, $T_{0,X}$, at Elevated Loading Rates*) provides the following relationship for deriving an estimate of $T_{0,X}$ (with $X$ being the logarithm of the average loading rate d$K$/d$t$) [18]:

$$T_{0,X}^{est} = \frac{(T_0+273.15)\cdot\Gamma}{\Gamma - ln\left(\frac{dK}{dt}\right)} - 273.15\,, \tag{1}$$

where the function Γ is given by:

$$\Gamma = 9.9 \cdot exp\left[\left(\frac{T_0+273.15}{190}\right)^{1.66} + \left(\frac{\sigma_{ys}}{722}\right)^{1.09}\right] \quad , \tag{2}$$

with $T_0$ = reference temperature at quasi-static loading rates ($dK/dt \leq 2$ MPa√m/s) and $\sigma_{ys}$ = room temperature yield strength measured at quasi-static strain rates.

**Figure 20** compares calculated reference temperatures from NIST tests on all materials with estimations obtained from Eq. (1). It can be observed that estimates are generally lower (*i.e.*, less conservative) then measured values by about 15 °C, with discrepancies as large as 38 °C. It's interesting to note that the effect of loading rate (expressed by the slope of the linear fits in **Figure 20**) is reasonably similar (0.0097 vs. 0.0079).

[5] Valid if $\sum_i r_i n_i \geq 1$.

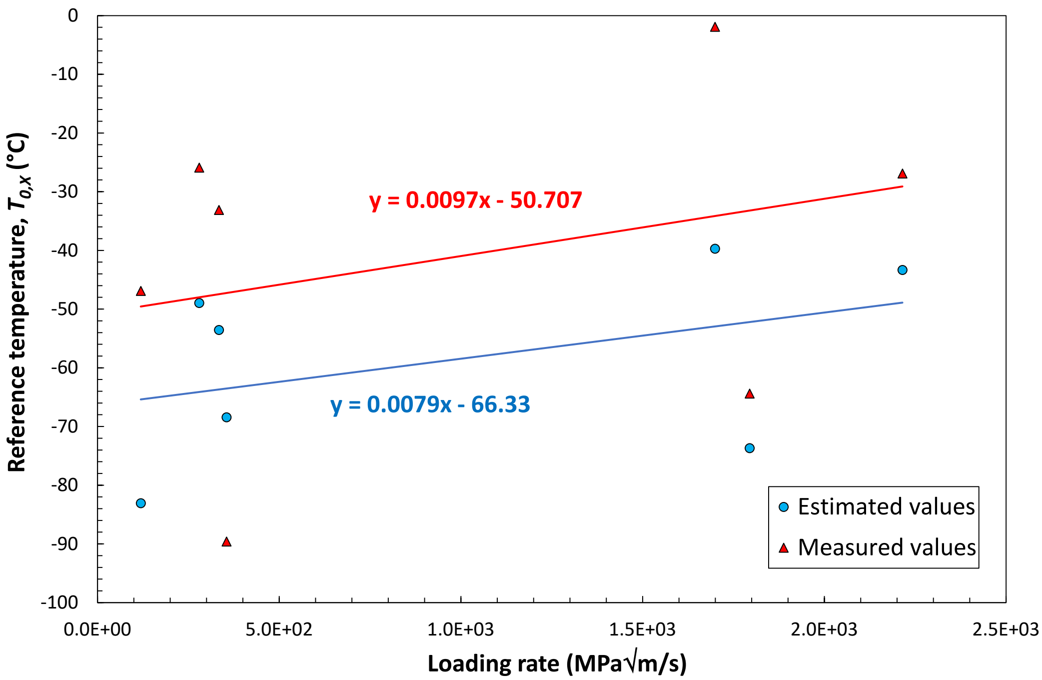


**Figure 20** – Estimated and measured reference temperatures $T_{0,X}$ for all materials.

## 8.2. Influence of Specimen Type/Loading Mode on Reference Temperatures

As already mentioned, the mixed bending/tension loading mode of a compact tension specimen produces a higher stress triaxiality state and higher crack-tip constraint with respect to the pure bending mode of a single-edge bend specimen. Consequently, Master Curve reference temperatures measured on C(T) specimens tend to be 10 °C - 15 °C higher than those calculated from tests on SE(B) specimens [16,17].

Four data sets from this investigation, all from Biblis C base material, allow assessing the effect of specimen type/loading mode on $T_{0,X}$ (for similar loading rates). $T_{0,X}$ values obtained from 1TC(T) and SE(B) 20/40 specimens for both $X = 2$ and $X = 3$ are compared in **Table 20**.

**Table 20** - Effect of specimen type/loading mode on $T_{0,X}$ for Biblis C base material.

| Loading rate range (MPa√m) | $X$ | Specimen type | $T_{0,X}$ (°C) | $\Delta T_{0,X}$ (°C) |
|---|---|---|---|---|
| 2.80E+2 to 3.34E+2 | 2 | SE(B) 20/40<br>1TC(T) | -33.1<br>-25.9 | 7.2 |
| 1.70E+3 to 2.21E+3 | 3 | SE(B) 20/40<br>1TC(T) | -26.9<br>-1.9 | 25.0 |

While the difference in $T_{0,X}$ for $X = 2$ is relatively close to the typical range, the increase for $X = 3$ (25 °C) appears quite large, which is likely due to the unreliability of the fast test results for which the estimated frequency bandwidth was clearly insufficient.

### 8.3. Possible Modification of the Master Curve for Higher Loading Rates

It has been contended [23] that some modifications to the standard Master Curve equation:

$$K_{Jc(med)} = 30 + 70 \cdot e^{0.019(T-T_0)} \tag{3}$$

are needed when the loading rate $dK/dt$ exceeds the higher limit for quasi-static testing (2 MPa√m/s), particularly when loading rates in excess of $10^5$ MPa√m/s are used in impact tests on precracked Charpy specimens. Specifically, Schindler and Kalkhof [7] advocated a change in the shape of the Master Curve, by replacing the exponent $p = 0.019$ in Eq. (3) with $p = 0.03$, which renders the Master Curve steeper for $T > T_0$.

For the four data sets developed at NIST at "slower" loading rates (*i.e.*, tests with $EBW < 0.5\ BW_{min}$), the Master Curve analyses were repeated with $p = 0.03$ and compared with the results obtained from the standard analyses with $p = 0.019$ (**Table 21**).

**Table 21** - Master Curve analyses on valid data sets using $p = 0.019$ (ASTM E1921-20) and $p = 0.03$ (proposed modification). $N_{out}$ is the percentage of data points falling outside the 5 % - 95 % confidence limits (green if $N_{out} \leq 10$ %, red if $N_{out} > 10$ %).

| Material | Specimen type | Average loading rate (MPa√m/s) | $p = 0.019$ | | | $p = 0.03$ | | |
|---|---|---|---|---|---|---|---|---|
| | | | $T_{0,2}$ (°C) | $K_{med}$ (MPa√m) | $N_{out}$ (%) | $T_{0,2}$ (°C) | $K_{med}$ (MPa√m) | $N_{out}$ (%) |
| Biblis C base | 1TC(T) | 2.80E+2 | -25.9 | 127.2 | 11 | -17.6 | 122.6 | 0 |
| | SE(B) 20/40 | 3.42E+2 | -33.1 | 120.7 | 0 | -28.5 | 125.2 | 0 |
| Biblis C weld | SE(B) 20/40 | 3.55E+2 | -89.6 | 170.2 | 30 | -75.1 | 167.6 | 10 |
| S690QL | 1TC(T) | 1.19E+2 | -46.9 | 127.1 | 29 | -43.3 | 137.7 | 21 |

Changing the Master Curve exponent causes an increase in reference temperature and decreases the number of data points falling outside the 5 % - 95 % confidence limits. For three of the four data sets, $N_{out}$ decreases from above to below the theoretical value of 10 %.

Our results therefore provide some support to the proposed modification of the Master Curve shape for loading rates beyond the quasi-static regime.

Master Curves and confidence limits for $p = 0.019$ and p = 0.03 are compared in **Figure 21** (Biblis C base, 1TC(T)), **Figure 22** (Biblis C base, SE(B) 20/40), **Figure 23** (Biblis C weld), and **Figure 24** (S690QL).

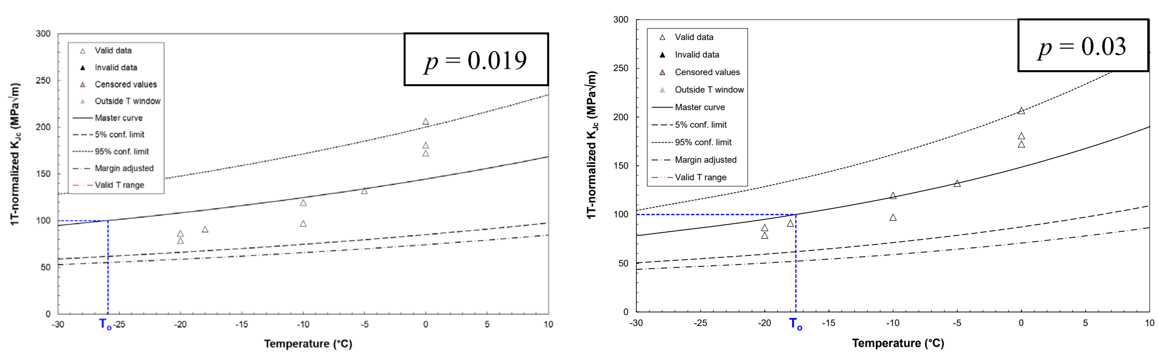


**Figure 21** - Master Curve analysis from Biblis C base 1TC(T) specimen tests with $p = 0.019$ (left) and $p = 0.03$ (right).

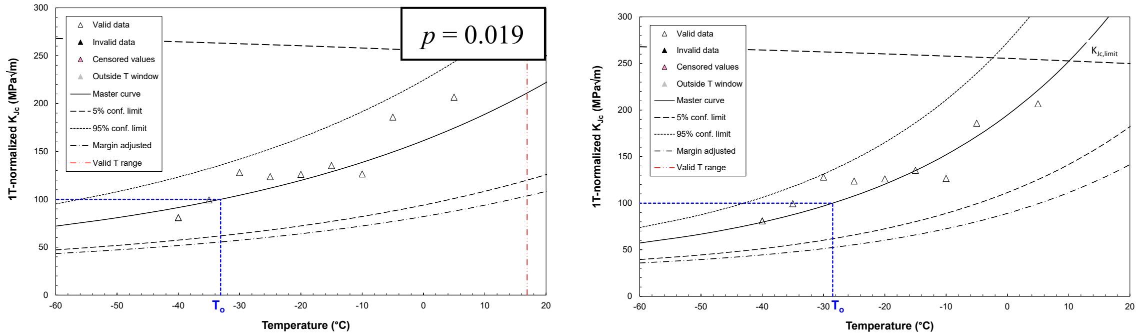


**Figure 22** - Master Curve analysis from Biblis C base SE(B) 20/40 specimen tests with $p$ = 0.019 (left) and $p$ = 0.03 (right).

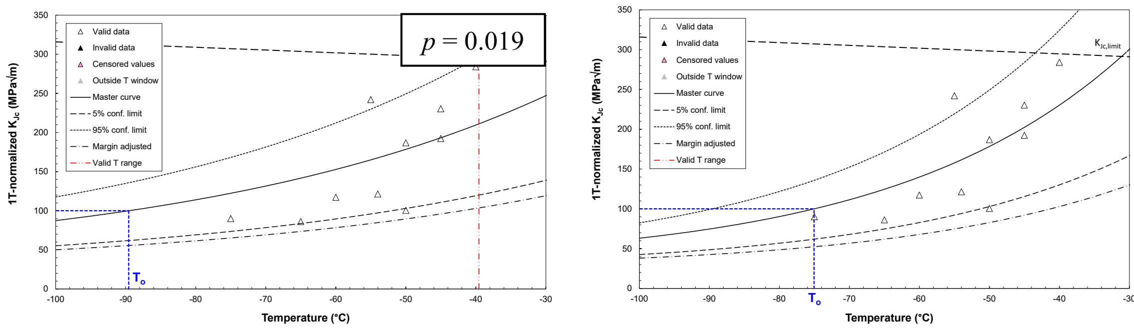


**Figure 23** - Master Curve analysis from Biblis C weld SE(B) 20/40 specimen tests with $p$ = 0.019 (left) and $p$ = 0.03 (right).

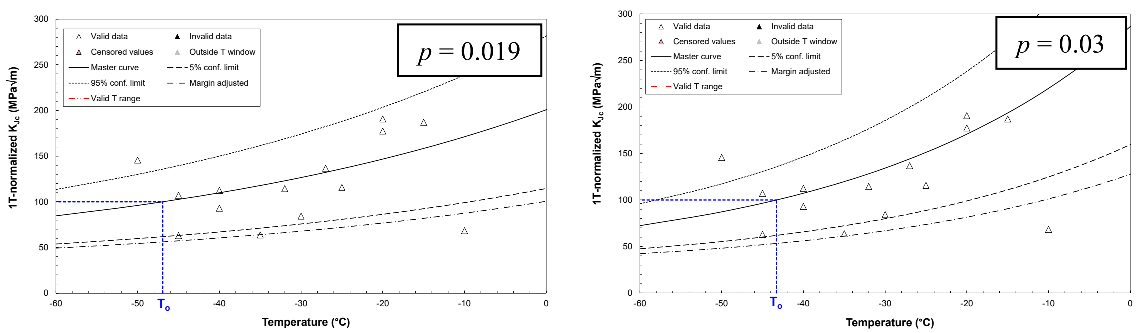


**Figure 24** - Master Curve analysis from S690QL 1TC(T) specimen tests with $p$ = 0.019 (left) and $p$ = 0.03 (right).

# 9. Ancillary Investigation: Rapid Tests on Precracked Charpy (PCCv) Specimens

## 9.1. Background

In April 2020, J. Tlatlik from Fraunhöfer Institut (Freiburg, Germany) proposed an additional study, consisting in high-rate Master Curve testing of precracked Charpy V-notch (PCCv) specimens, extracted from tested SE(B) 20/40 samples from two of the investigated materials (Biblis C base and weld materials). NIST agreed to take part in this ancillary activity.

Ten PCCv specimens were extracted from two SE(B) samples from each material, for a total of 20 specimens, as shown in **Figure 25** (base) and **Figure 26** (weld).

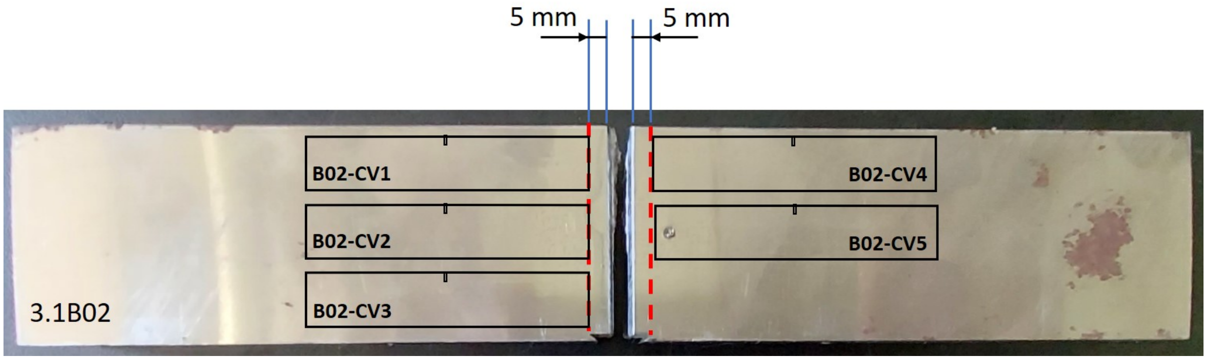


**Figure 25** – Extraction of five PCCv specimens from SE(B) sample 3.1B02 (base material).

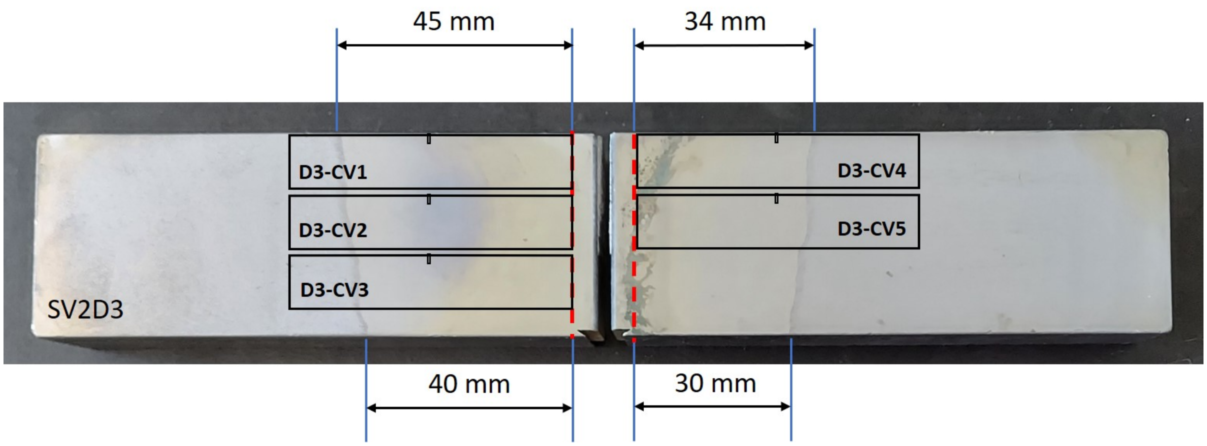


**Figure 26** – Extraction of five PCCv specimens from SE(B) sample SV2D3 (weld material). The SE(B) sample halves were previously etched to expose the weld seam.

The specimens, after being machined in Colorado, were sent to Fraunhöfer Institut for fatigue precracking, and were finally shipped back to Boulder.

## 9.2. Test Results

### 9.2.1. Biblis C Base Material

Ten PCCv specimens were tested between -65 °C and -25 °C at actuator displacement rates between 2.94 mm/s – 3.02 mm/s, corresponding to CMOD rates between 0.21 mm/s – 1.51 mm/s. A summary of individual test results is provided in **Table 22**.

For all tests performed, the estimated bandwidth was higher than 54 % of the minimum bandwidth required by ASTM E1820 Annex A14; therefore, all tests can be considered reliable. The average loading rate (1.05E+2 MPa√m/s) is approximately three times lower than the value for lower rate SE(B) 20/40 tests (**Table 11**).

A valid reference temperature, $T_{0,2}$ = -15.0 °C, was obtained by analyzing test results. This is 18.1 °C higher than $T_{0,2}$ obtained from lower rate SE(B) 20/40 tests. Details of the Master Curve analyses (summarized in **Table 23**) are given in Appendix J.

**Table 22** - Results obtained from tests on PCCv specimens of Biblis C base material.

| Specimen id | $T$ (°C) | Actuator rate (mm/s) | CMOD rate (mm/s) | Loading rate, $dK/dt$ (MPa√m/s) | $K_{Jc}$ (MPa√m) | $EBW$ (Hz) | $BW_{\min}$ (Hz) | $\frac{EBW}{BW_{min}}$ |
|---|---|---|---|---|---|---|---|---|
| B02-CV5 | -65 | 2.96 | 0.21 | 7.28E+01 | 62.53 | 44.1 | 37.5 | **118%** |
| B02-CV2 | -55 | 2.99 | 0.38 | 7.88E+01 | 61.80 | 46.2 | 47.9 | **97%** |
| A08-CV5 | -50 | 2.96 | 0.71 | 9.83E+01 | 81.61 | 42.6 | 54.3 | **78%** |
| B02-CV4 | -46 | 2.99 | 0.69 | 8.93E+01 | 74.38 | 44.0 | 56.1 | **78%** |
| A08-CV3 | -45 | 2.94 | 0.54 | 9.22E+01 | 68.31 | 45.8 | 57.7 | **79%** |
| A08-CV1 | -40 | 3.02 | 1.06 | 1.20E+02 | 109.82 | 44.1 | 67.4 | **65%** |
| A08-CV4 | -40 | 2.97 | 1.31 | 1.17E+02 | 102.09 | 41.3 | 73.4 | **56%** |
| A08-CV2 | -35 | 2.97 | 1.12 | 1.08E+02 | 94.10 | 42.4 | 78.3 | **54%** |
| B02-CV1 | -30 | 2.97 | 1.51 | 1.33E+02 | 120.88 | 42.2 | 75.2 | **56%** |
| B02-CV3 | -25 | 2.99 | 1.30 | 1.36E+02 | 123.79 | 41.4 | 56.4 | **73%** |

**Table 23** - Master Curve analysis results from tests on PCCv specimens of Biblis C base material.

| Number of tests | Valid tests | $T_{0,2}$ (°C) | $\sum r_i n_i$ | Average loading rate (MPa√m/s) | $K_{med}$ (MPa√m) | $\sigma_{T0}$ (°C) |
|---|---|---|---|---|---|---|
| 10 | 10 | -15.0 | 1.4 | 1.05E+2 | 72.0 | 7.2 |

The Master Curve is shown in **Figure 27**. All data points fall within the 5 % and 95 % confidence limits. Based on the SINTAP screening criterion of ASTM E1921, the material is screened to be homogeneous.

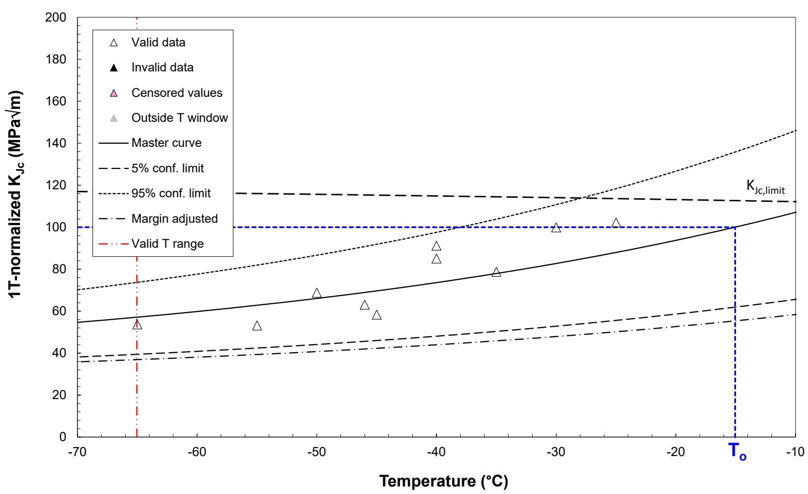


**Figure 27** – Homogeneous Master Curve analysis of the tests performed on PCCv specimens of Biblis C base material.

#### 9.2.1.1. Combined Master Curve Analysis of PCCv and SE(B) 20/40 Tests

As mentioned above, the reference temperature measured from PCCv specimens was 18.1 °C higher (more conservative) than the value obtained from reliable SE(B) 20/40 tests. Although this difference is close to the variability normally associated to $T_0$ determinations (20 °C), its magnitude is somewhat surprising. Several aspects can be considered when comparing the two data sets.

(a) Considering that PCCv tests were conducted at lower loading rates *dK/dt*, one would expect the reference temperature to be lower than for SE(B).

(b) The loading mode (pure bending) is identical between PCCv and SE(B) specimens, so loading mode cannot be a factor.

(c) PCCv tests were generally performed at lower temperatures (-65 °C to -25 °C) than SE(B) tests (-40 °C to 5 °C), since the maximum $K_{Jc}$ capacity, $K_{Jclimit}$, is inversely proportional to the specimen size, and therefore PCCv specimens tested at higher temperatures run a greater risk of exceeding $K_{Jclimit}$. The influence of test temperature on $T_0$ is generally considered to be negligible [24-26] within the valid temperature window (-50 °C ≤ $T_0$ ≤ 50 °C), where all PCCv and SE(B) tests in this investigation were performed. In particular, Viehrig *et al* [10] came to this same conclusion when analyzing fracture toughness tests performed at quasi-static loading rates on specimens of various type and size from Biblis C base material.

(d) The standard deviation of the measured $T_0$, according to ASTM E1921, can be estimated as:

$$\sigma_{T0} = \sqrt{\frac{\beta^2}{r} + \sigma_{exp}^2} \quad , \tag{4}$$

where $\beta$ is a sample size uncertainty factor ($\beta$ = 18.8 °C for PCCv tests and $\beta$ = 18 °C for SE(B) tests), $r$ is the number of valid test results, and $\sigma_{exp}$ = 4 °C is the contribution of experimental uncertainties when standard calibration practices are followed. Based on Eq. (4), $\sigma_{T0}$ is 7.2 °C for PCCv specimens (**Table 23**) and 7.0 °C for SE(B) 20/40 specimens (**Table 11**). The ± $2\sigma_{T0}$ intervals, corresponding to approximately 95 % confidence, are found to overlap ($T_0 - 2\sigma_{T0}$ = -29.4 °C for PCCv specimens, $T_0 + 2\sigma_{T0}$ = -19.1 °C for SE(B) specimens), and therefore the two reference temperatures cannot be considered statistically different.

The combined PCCv/SE(B) Master Curve analysis yielded $T_0$ = -27.1 °C (**Table 24** and **Figure 28**). From **Figure 28**, the difference in test temperature range is apparent. All the data points are encompassed by the 5 % - 95 % Master Curve confidence limits.

**Table 24** – Combined Master Curve analysis results from valid PCCv and SE(B) 20/40 specimens of Biblis C base material.

| **Number of tests** | **Valid tests** | **$T_{0,2}$ (°C)** | **$\sum r_i n_i$** | **Average loading rate (MPa√m/s)** | **$K_{med}$ (MPa√m)** | **$\sigma_{T0}$ (°C)** |
|---|---|---|---|---|---|---|
| 20 | 20 | -27.1 | 3.2 | 2.23E+2 | 96.9 | 5.7 |

The SINTAP procedure screened the combined data set as homogeneous, which supports the previous conclusion that the difference between specimen types is not significant.

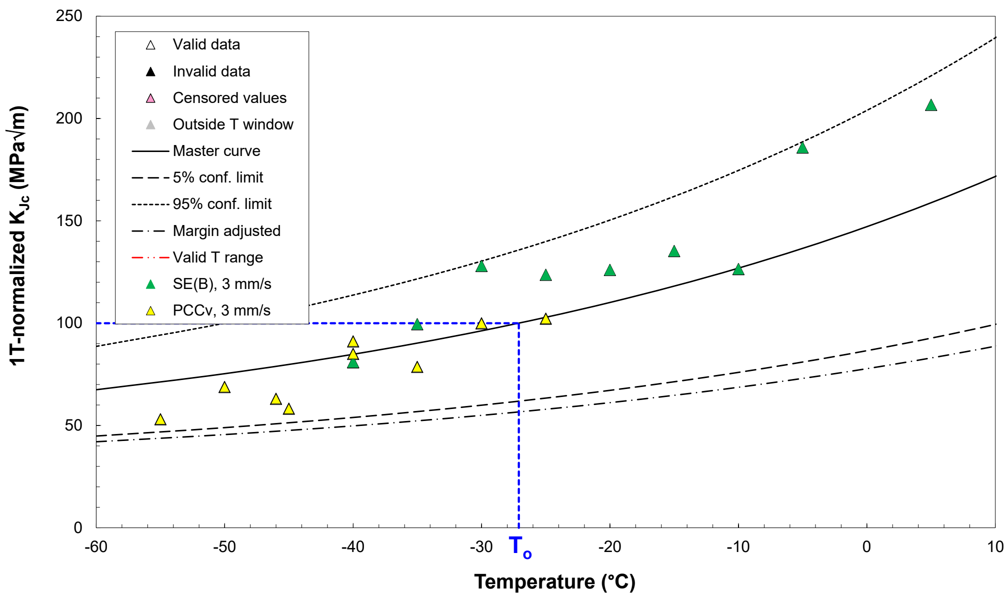


**Figure 28** – Combined Master Curve analysis of the tests performed on PCCv and SE(B) 20/40 specimens of Biblis C base material.

A modified Master Curve analysis, conducted using $p = 0.03$ instead of $p = 0.019$, yielded a marginally different $T_0 = -26.7$ °C, and causes one data point (5 %) to fall slightly above the 95 % confidence limits (**Figure 29**).

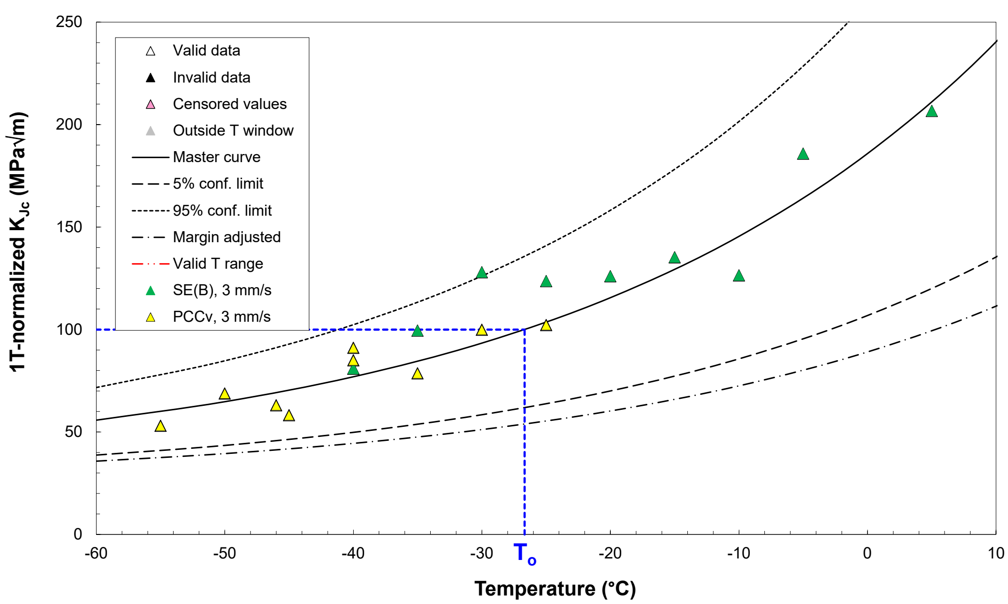


**Figure 29** – Modified combined Master Curve analysis ($p = 0.03$) of the tests performed on PCCv and SE(B) 20/40 specimens of Biblis C base material.

### 9.2.2. Biblis C Weld Material

Of the ten available PCCv specimens, only five were actually tested between -60 °C and -25 °C at actuator displacement rates between 2.99 mm/s – 3.01 mm/s, corresponding to CMOD rates between 0.53 mm/s – 2.60 mm/s. For the remaining five specimens, various experimental problems prevented obtaining valid results. Moreover, three of the tested specimens yielded invalid results, which had to be censored in the Master Curve analysis. A summary of individual test results is provided in **Table 25**.

For all tests performed, the estimated bandwidth is at least 72 % of the minimum bandwidth required by ASTM E1820 Annex A14. The average loading rate (1.05E+2 MPa√m/s) is approximately three times lower than the value for lower rate SE(B) 20/40 tests (**Table 11**).

**Table 25** - Results obtained from tests on PCCv specimens of Biblis C base material.

| Specimen id | $T$ (°C) | Actuator rate (mm/s) | Load-line rate (mm/s) | Loading rate, $dK/dt$ (MPa√m/s) | $K_{Jc}$ (MPa√m) | $EBW$ (Hz) | $BW_{min}$ (Hz) | $\frac{EBW}{BW_{min}}$ | Valid? |
|---|---|---|---|---|---|---|---|---|---|
| D7-CV5 | -60 | 2.99 | 0.53 | 1.05E+02 | 103.12 | 45.0 | 54.6 | 82% | YES |
| D3-CV4 | -50 | 2.99 | 1.01 | 1.04E+02 | 108.45 | 42.9 | 59.3 | 72% | YES |
| D3-CV1 | -45 | 2.97 | 1.64 | 1.75E+02 | **170.48** | 42.8 | 53.1 | 81% | **NO***  |
| D7-CV4 | -30 | 3.00 | 1.87 | 1.79E+02 | **248.08** | 40.0 | 29.7 | 135% | **NO§** |
| D7-CV2 | -25 | 3.01 | 2.60 | 2.29E+02 | **353.33** | 26.8 | 18.6 | 144% | **NO§** |

INVALIDITY CAUSES: * $K_{Jc} > K_{Jclimit}$ .
§ $K_{Jc} > K_{Jclimit}$ and $\Delta a_p > 0.05(W - a_0)$ .

Because of the limited number of valid test results available, the calculated reference temperature, $T_{0,2}$ = -55.2 °C, was invalid according to ASTM E1921, which requires a minimum of 6 valid data. This is significantly higher (by 34.4 °C) than $T_0$ obtained from lower rate SE(B) 20/40 tests (-89.6 °C). Details of the Master Curve analyses (summarized in **Table 26**) are given in Appendix K. The Master Curve obtained is shown in **Figure 30**.

**Table 26** - Master Curve analysis results from tests on PCCv specimens of Biblis C weld material.

| Number of tests | Valid tests | $T_{0,2}$ (°C) | $\sum r_i n_i$ | Average loading rate (MPa√m/s) | $K_{med}$ (MPa√m) | $\sigma_{T0}$ (°C) |
|---|---|---|---|---|---|---|
| 5 | 2 | -55.2 | 0.3 | 1.58E+2 | 100.6 | 13.3 |

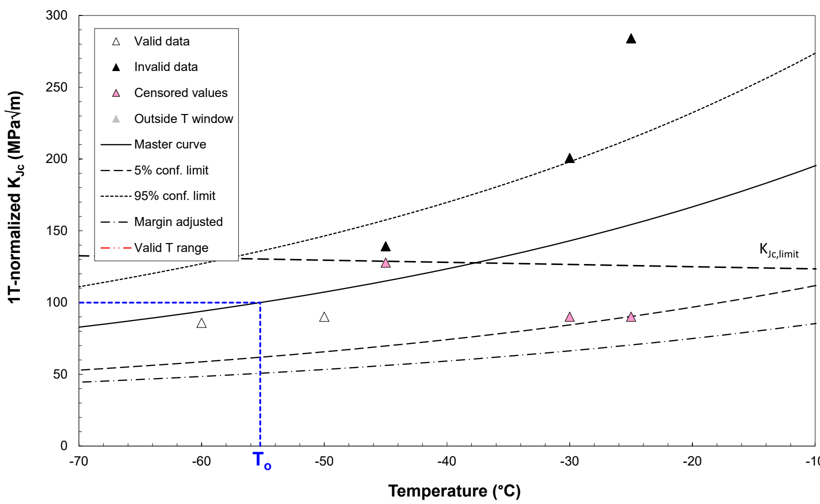


**Figure 30** – Homogeneous Master Curve analysis of the tests performed on PCCv specimens of Biblis C weld material.

### 9.2.2.1. Combined Master Curve Analysis of PCCv and SE(B) 20/40 Tests

The five data points from PCCv tests were combined with the ten test results (all valid) obtained from SE(B) 20/40 specimens (**Table 16**). The overall Master Curve analysis yielded $T_0$ = -87.3 °C (**Table 24** and **Figure 31**), which is only 2.3 °C higher than the value obtained for SE(B) specimens only. Only one data point (7 %) falls outside the 5 % - 95 % Master Curve confidence limits.

**Table 27** – Combined Master Curve analysis results from valid PCCv and SE(B) 20/40 specimens of Biblis C weld material.

| **Number of tests** | **Valid tests** | $T_{0,2}$ **(°C)** | $\sum r_i n_i$ | **Average loading rate (MPa√m/s)** | $K_{med}$ **(MPa√m)** | $\sigma_{T0}$ **(°C)** |
|---|---|---|---|---|---|---|
| 15 | 12 | -87.3 | 2.0 | 2.90E+2 | 163.5 | 6.6 |

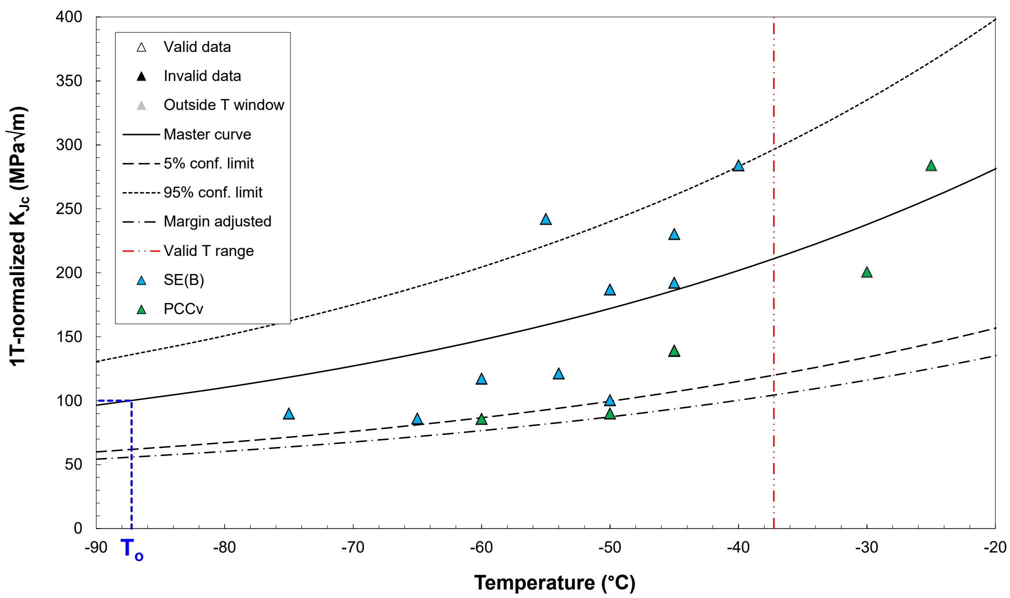


**Figure 31** – Combined homogeneous Master Curve analysis of the tests performed on PCCv and SE(B) 20/40 specimens of Biblis C weld material.

The SINTAP procedure screened the combined data set as inhomogeneous, with an adjusted conservative reference temperature $T_{0IN}$ = -71.4 °C (**Figure 32**).

If the Master Curve slope is modified ($p$ = 0.03 instead of 0.019), the reference temperature obtained is $T_0$ = -73.6 °C, which is comparatively close to the value of $T_{0IN}$ above. The number of data points falling outside the 5 % - 95 % confidence limits remains the same (**Figure 33**).

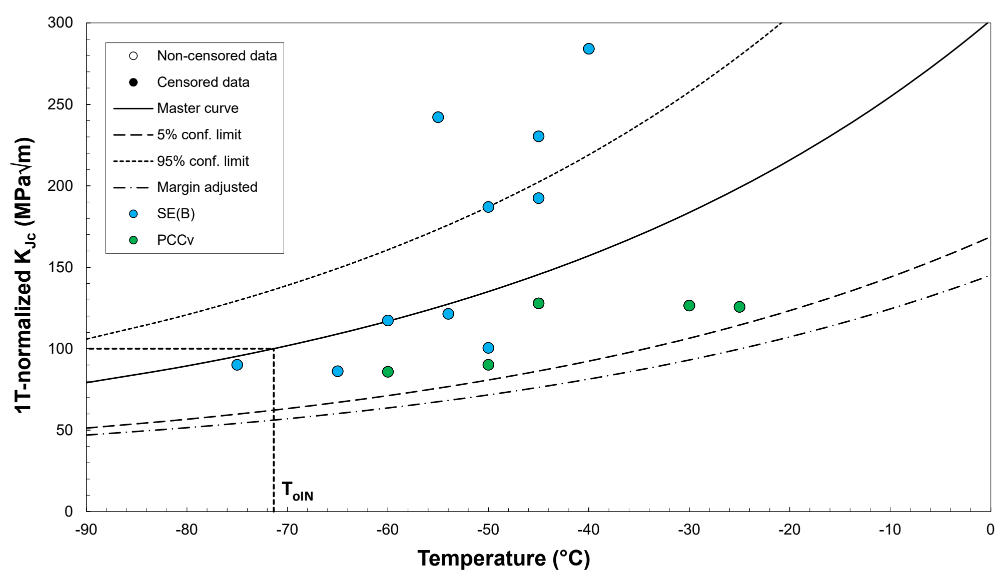


**Figure 32** – Combined inhomogeneous Master Curve analysis of the tests performed on PCCv and SE(B) 20/40 specimens of Biblis C weld material.

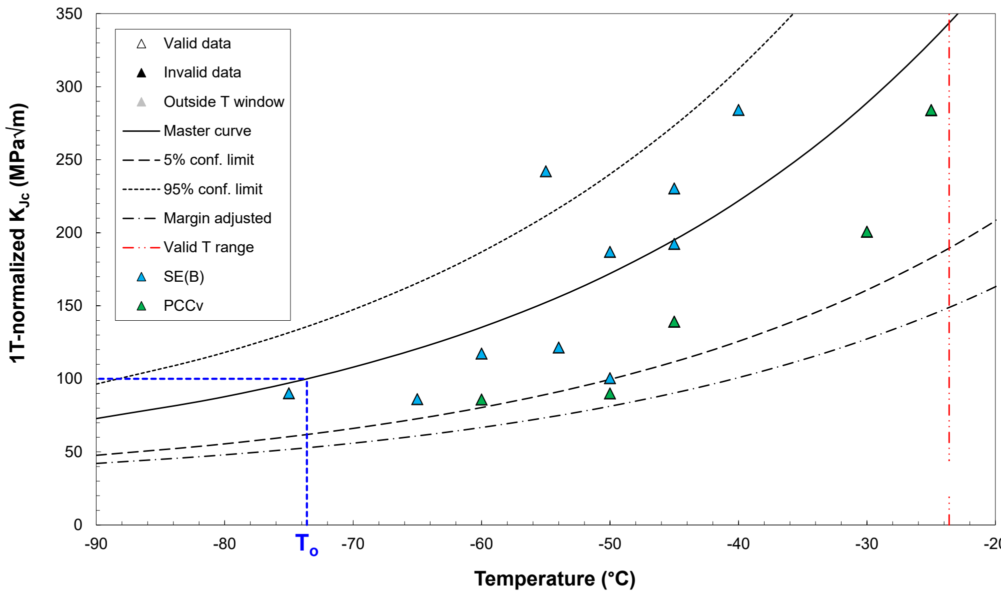


**Figure 33** – Modified combined Master Curve analysis ($p$ = 0.03) of the tests performed on PCCv and SE(B) 20/40 specimens of Biblis C weld material.

## 10. Summary of Master Curve Analysis Results

**Table 28** summarizes the results of the Master Curve analyses conducted on the most significant data sets generated within this investigation. The following types of data sets are not included in the summary:

- data sets including a small number of tests for which $EBW < BW_{min}$ (e.g., all SE(B) 20/40 tests conducted at 3 mm/s on Biblis C base (**Table 11**));
- data sets combining specimens with different loading modes, e.g., C(T) and SE(B);
- data sets combining tests at lower (~$10^2$ MPa√m/s) and higher (~$10^3$ MPa√m/s) loading rates.

**Table 28** also includes information about the potential macroscopic inhomogeneity of the different data sets. It's interesting to note that, for Biblis C base material, only the higher rate data sets (which are unreliable because of the too low frequency bandwidth) are screened as inhomogeneous. This outcome of the SINTAP procedure should also be considered dubious. Conversely, for the Biblis C weld material, both the SE(B) and combined SE(B)+PCCv data sets appear to be macroscopically inhomogeneous, which is a rather common occurrence for weld materials.

**Table 28** – Summary of the most relevant Master Curve analysis results obtained in this investigation.

| Material | Specimen type | Loading rate (MPa√m/s) | $T_{0,X}$ (°C) | $\sum r_i n_i$ | Bandwidth valid? | HOM? | $T_{0IN}$ (°C) | NOTES |
|---|---|---|---|---|---|---|---|---|
| Biblis C base material | 1TC(T) | 1.70E+03 | -1.9 | 1.5 | NO | NO | 12.8 | |
| | | 2.80E+02 | -25.9 | 1.5 | YES | YES | - | |
| | SE(B) 20/40 | 2.21E+03 | -26.9 | 1.2 | NO | NO | 12.9 | |
| | | 3.42E+02 | -31.0 | 2.0 | YES | YES | - | |
| | PCCv | 1.05E+02 | -15.0 | 1.4 | YES | YES | - | |
| | SE(B)+PCCv | 2.23E+02 | -27.1 | 3.2 | YES | YES | - | |
| Biblis C weld material | SE(B) 20/40 | 3.55E+02 | -89.6 | 1.7 | YES | NO | -77.5 | |
| | PCCv | 1.58E+02 | -55.2 | 0.3 | YES | YES | - | $T_0$ invalid ($\sum r_i n_i < 1$) |
| | SE(B)+PCCv | 0.00E+00 | -87.3 | 2.0 | YES | NO | -71.4 | |
| S690QL | 1TC(T) | 1.88E+02 | -44.4 | 1.0 | YES | YES | - | |

## 11. Conclusions

NIST contributed to the ASTM E08.07.05 Round Robin on Determining the Master Curve Reference Temperature, $T_{0,X}$, at Elevated Loading Rates (ILS #1547), by performing 80 low-temperature fracture toughness tests at elevated loading rates. Three specimen types were used: 1TC(T), SE(B) 20/40, and PCCv.

Some the tests performed cannot be considered reliable, as the relevant actuator displacement rate (in the range 30 mm/s - 40 mm/s) corresponded to a minimum required frequency bandwidth that was significantly higher than the estimated value for the NIST force measuring system. The remaining tests, however, were conducted at actuator rates in the range 2.5 mm/s - 3 mm/s, which produced estimated bandwidths greater than half the required minimum, and should therefore be considered reliable.

Various Master Curve analyses were performed in accordance with ASTM E1921, including potential inhomogeneity screening of the various data sets by means of the simplified SINTAP procedure.

A few observations can be derived from the tests performed at NIST and the analyses conducted on the results.

(a) It appears possible, and actually desirable, to relax the minimum required frequency bandwidth in a future revision of the ASTM E1820 standard to approximately half of its current value. This will hopefully be supported by a favorable comparison between the lower rate NIST results and data obtained by other round-robin participants, that disposed of measuring systems with large enough bandwidth.

(b) For one of the test materials (Biblis C base material), 1TC(T) provided higher reference temperatures than SE(B) specimens, in agreement with the published literature. However, while for lower rate tests the difference (7.2 °C) is in line with expected values, for higher rate tests the difference (25 °C) is too large, which confirms the unreliability of the higher loading rate tests.

(c) Despite the uncertainties of the higher rate test results, the observed loading rate effects are congruent with expectations (higher loading rates correspond to higher reference temperatures, or lower fracture toughness).

(d) Our results support the proposal to modify the slope of the Master Curve by changing its exponent from 0.019 to 0.03. Doing so decreased the number of points falling outside the Master Curve 5 % and 95 % confidence limits for several data sets. Another consequence is also the increase of the calculated reference temperature.
(e) The additional tests on precracked Charpy (PCCv) specimens of Biblis C base and weld materials provided higher reference temperatures than those calculated from bigger SE(B) 20/40 specimens, although considering $\pm 2\sigma_{T0}$ confidence intervals, the $T_0$ values for the base material cannot be considered statistically different.

For a comprehensive assessment of NIST test results, it will be necessary to wait until the final Report of the Interlaboratory Study, comparing all the participants' results, is released. This is expected to happen by the end of 2021.

**Acknowledgments**

I would like to express my sincere gratitude to Jake Benzing for his help in setting up and performing the tests described in this report, as well as reviewing this report, along with Dash Weeks..

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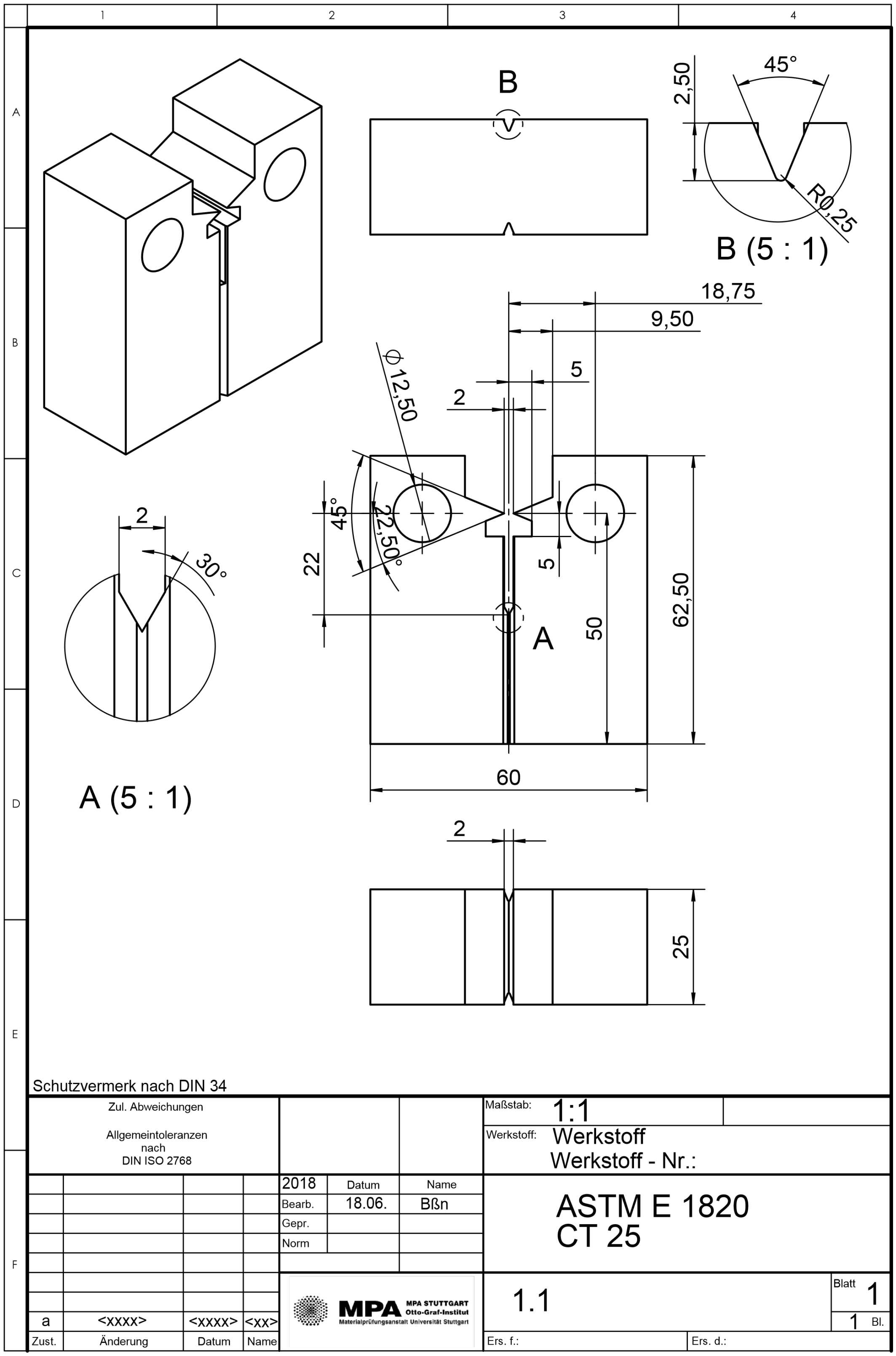
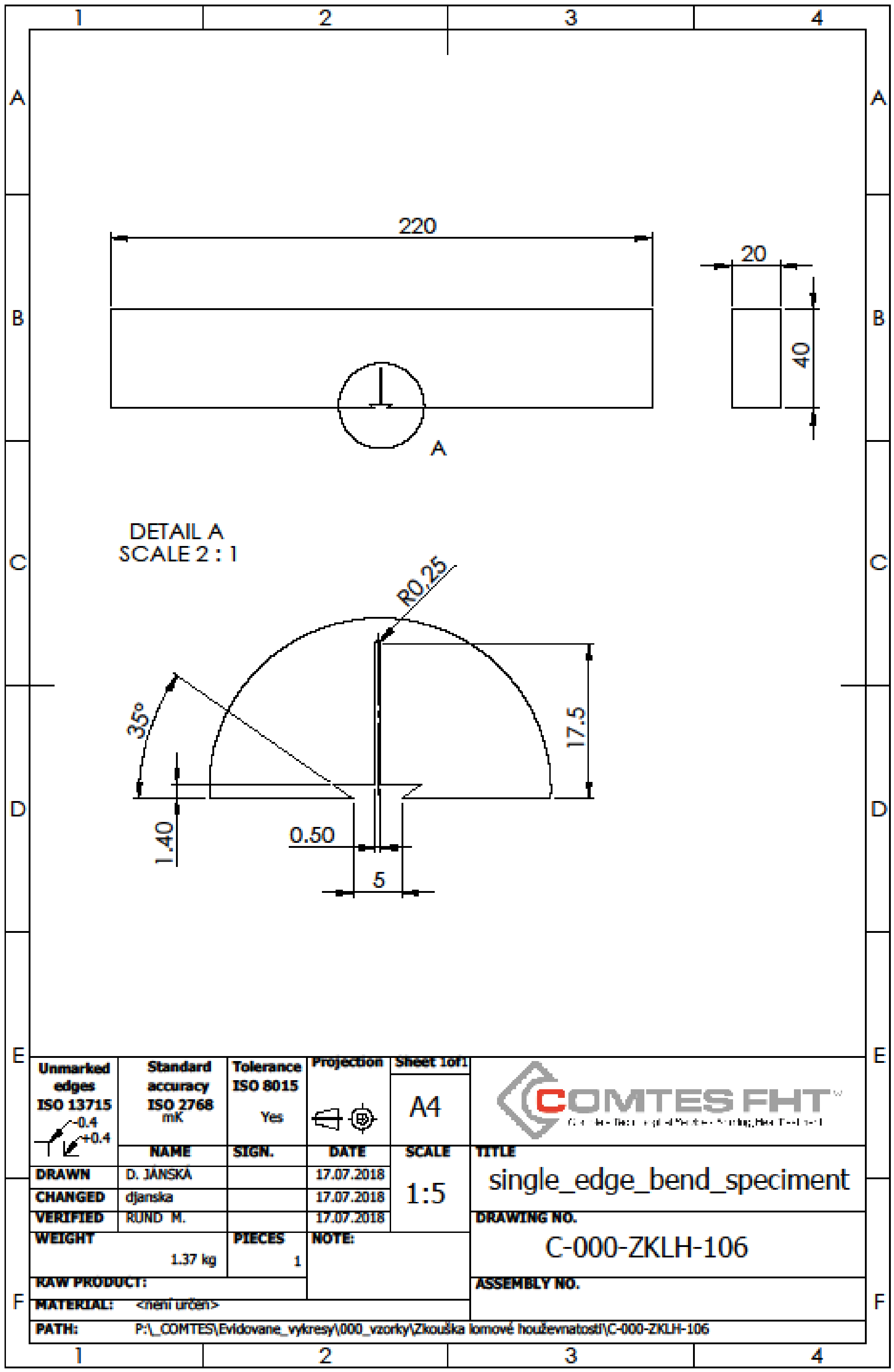
## Appendix A: Drawing of the Compact Tension specimen with B = 25 mm thickness and integral knife edges



## Appendix B: Drawing of the Single-Edge Bend specimen with B = 20 mm, W = 40 mm, and integral knife edges



## Appendix C: Use of the Macro-Enabled Spreadsheet *Rapid-Load KJc Fracture Toughness Test.xlsm*

### C.1 Worksheet "Data"

This is where the raw data from the test (time, actuator displacement command signal[6], actuator displacement, clip-gage displacement, force) are entered in columns A-E (highlighted in yellow[7]). Clicking the button "CLEAR DATA" erases all existing data.

Test information can be entered in cells B4:B9. Most importantly, the user needs to select from a drop-down menu in cell I1 the test loading mode, or specimen type (bend – SE(B) or PCCv – or compact tension). This will determine what formulas are used for several test parameters, such as $K$, $J$, compliance, etc. Additional user's input is required in cells I23:I24 (time range for calculating the actuator displacement rate), I28:I29 (time range for calculating the CMOD/LLD[8] rate), and Q5 (index, or row number, of data record corresponding to unstable fracture – unless this coincides with maximum force, which is automatically entered in cell Q4 of the spreadsheet).

Various calculations are performed in columns O-AL. Currently, the spreadsheet can accommodate data files containing up to 2000 data points. It could be easily modified to increase the number of data points in the raw data file.

### C.2 Chart "Test record"

The force vs. CMOD/LLD graph of the test is plotted, for quick review. Axis scales and titles can be freely edited by the user (for example, replacing "CMOD" with "LLD", or *vice versa*).

### C.3 Chart "Displacement vs time"

The actuator displacement vs. time graph of the test is plotted. This can be reviewed by the user to determine the time range to be used for calculating the actuator displacement rate, see A.1 ("Data"). If the command signal is available, the corresponding data are plotted for comparison with the actual displacement signal (example in **Figure C.1**). Axis scales and titles can be freely edited by the user.

### C.4 Chart "CMOD vs time" [9]

The CMOD/LLD vs. time graph of the test is plotted. This can be reviewed by the user to determine the time range to be used for calculating the CMOD/LLD rate, see C.1 ("Data"). Axis scales and titles can be freely edited by the user.

[6] If the command signal is not available, column B can be simply left blank.
[7] In this spreadsheet, any cell that requires input from the user is highlighted in yellow.
[8] The user can replace "CMOD" with "LLD" (or *vice versa*) in cells H27 and H30, depending upon which specimen type (compact tension or single-edge bend) has been tested.
[9] The title of the worksheet can also be edited to replace "CMOD" with "LLD".

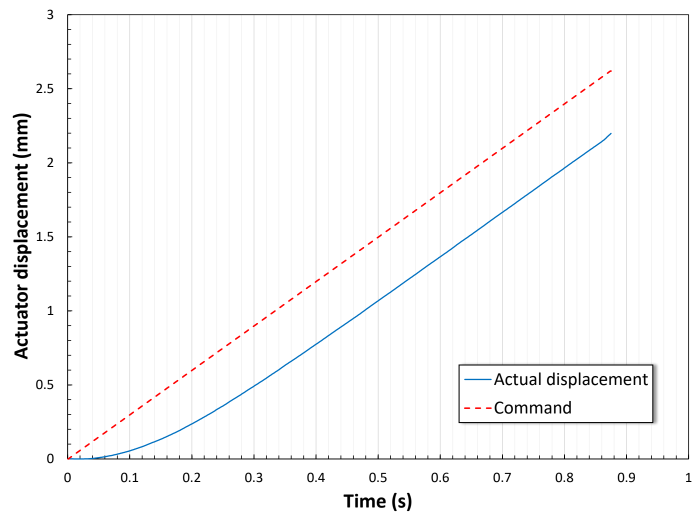



**Figure C.1** – Example of actuator displacement vs. time plot, with command signal.

## C.5 Chart "Force vs time"

The force vs. time graph of the test is plotted. The data points automatically selected by the spreadsheet to calculate the linear regression of the force drop following the onset of cleavage (see worksheet "Data", cells J4:L6) are plotted as red square symbols. These data points are automatically selected starting 5 rows after specimen failure until 2 rows before the force/time plot crosses the x-axis, or the first negative force value. An example is shown in **Figure C.2**.

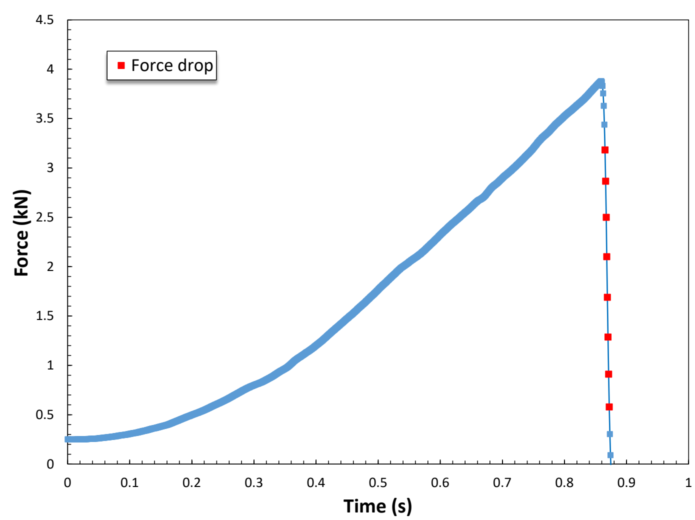



**Figure C.2** – Example of force vs. time plot, highlighting data corresponding to the force drop.

## C.6 Chart "J vs time"

The *J*-integral vs. time graph of the test is plotted. The linear regression of the data in the range between 0.5 $J_c$ and $J_c$ ($J_c$ being the value of *J*-integral at cleavage), highlighted by red square symbols, is also plotted and back-extrapolated to the x-axis to calculate the test time

$t_Q$, as seen in the example of **Figure C.3**. Note: the red arrow indicating $t_Q$ must be adjusted by the user on the plot. The value of $t_Q$ is shown in cell Z17 of worksheet "Data".

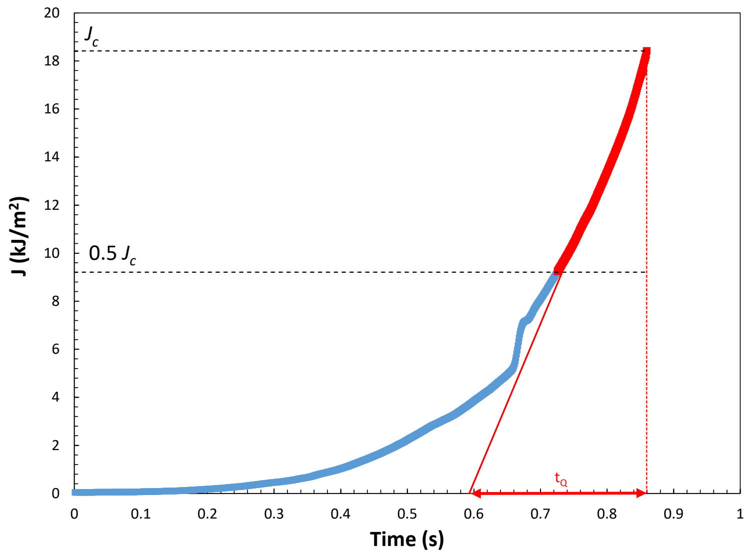



**Figure C.3** – Example of $J$ vs. time plot, with the linear regression of data between 0.5 $J_c$ and $J_c$, which allows calculating $t_Q$.

## C.7 Chart "dF_dt vs CMOD"[10]

Values of force application rate (*dF/dt*), calculated in column O of worksheet "Data", are plotted vs. CMOD/LLD. The onset of cleavage is indicated by a large drop in *dF/dt* (**Figure C.4)**. Axis scales and titles can be freely edited by the user, as well as the position of the label "CLEAVAGE".

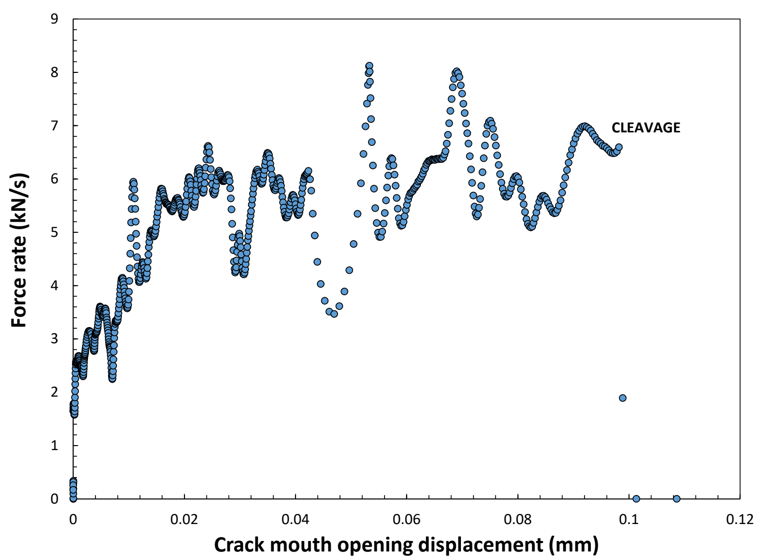



**Figure C.4** – Example of *dF/dt* vs. CMOD plot.

[10] The title of the worksheet can be edited to replace "CMOD" with "LLD".

## C.8 Chart "dCMOD_dt vs time"[11]

Values of CMOD/LLD rate, calculated in column P of worksheet "Data", are plotted vs. time. The onset of cleavage is indicated by a large upward jump in dCMOD/dt (or dLLD/dt) (**Figure C.5**). Axis scales and titles can be freely edited by the user, as well as the position of the label "CLEAVAGE".

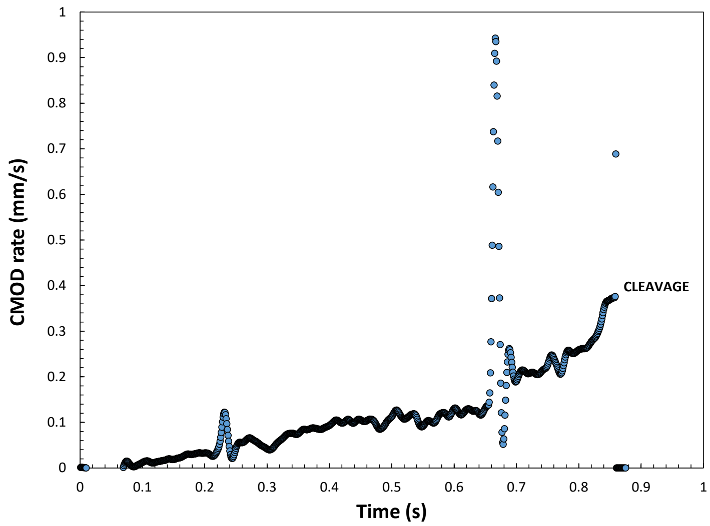



**Figure C.5** – Example of dCMOD/dt vs. time plot.

## C.9 Chart "Force vs CMOD"[11]

Force and CMOD or LLD data points up to the onset of cleavage are plotted, as well as the linear fit of the elastic portion of the test record (**Figure C.6**). This chart can be checked by the user to confirm the reliability of the linear regression. Axis scales and titles can be freely edited by the user.

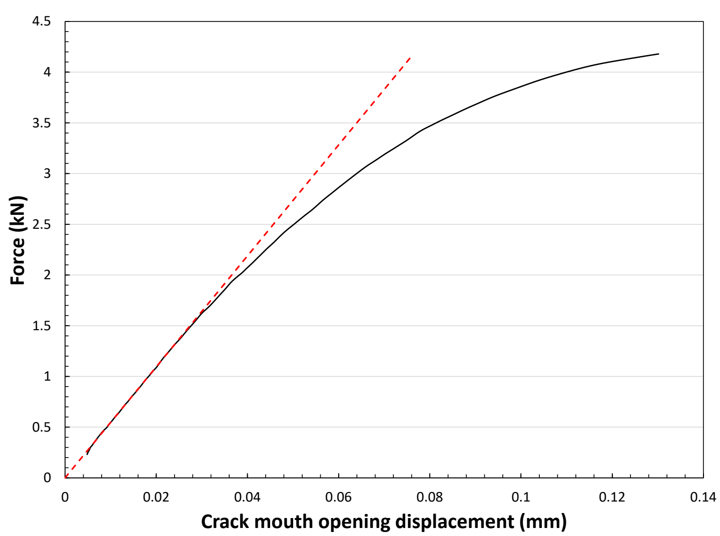



**Figure C.6** – Example of force vs. CMOD plot up to the onset of cleavage.

[11] The title of the worksheet can be edited to replace "CMOD" with "LLD".

C.10 Worksheet "Calculations"

All the main test results are calculated in this worksheet, namely:

- cell M10: $J$-integral at the onset of cleavage, $J_c$;
- cell M11: corresponding value of stress intensity factor, $K_{Jc}$ (to be used in the Master Curve analysis);
- cell M23: loading rate, $dK/dt$ (obtained by dividing $K_{Jc}$ by the time to cleavage, $t_f$).

The following input is required from the user:

- cell H1: specimen id;
- cells H4-H6: specimen thickness $B$, net thickness $B_N$, and width $W$;
- cell H12: ductile crack growth preceding cleavage, $\Delta a_p$;
- cell H14: Young's modulus, $E$.
- cells H24-H25: slope and intercept of the linear fit[12] of the elastic portion of the test record, respectively;
- cell H27: effective mass of the specimen, $M_{eff}$ (given by half of its weight, in kg).

The following validity checks are also performed in this worksheet.

- The test time, $t_Q$, must be higher than the minimum test time, $t_w$, given by:

$$t_w = \frac{2\pi}{\sqrt{\frac{k_S}{M_{eff}}}} \tag{C.1}$$

where $k_s$, the initial specimen stiffness, is given by a linear regression analysis of data over the range from 20 % to 50 % of the maximum force.

- The estimated frequency bandwidth of the test, $EBW$, must be greater than the minimum required bandwidth[13], $BW_{min}$. The formulae used for the calculations are:

$$EBW = \frac{0.35}{t_f} \tag{C.2}$$

$$BW_{min} = \frac{10}{t_Q} \tag{C.3}$$

- The experimental specimen compliance $C_{exp}$, given by the inverse of the initial stiffness $k_s$, must be within ±10 % of the theoretical specimen compliance, given for a single-edge bend specimen by:

$$C_{exp} = \frac{6S}{EWB_e}\left(\frac{a_0}{W}\right)\left[0.76 - 2.28\left(\frac{a_0}{W}\right) + 3.87\left(\frac{a_0}{W}\right)^2 - 2.04\left(\frac{a_0}{W}\right)^3 + \frac{0.66}{\left(1-\frac{a_0}{W}\right)^2}\right], \tag{C.4}$$

where $S$ is the test setup span, or the distance between the bottom rollers in the 3-point bend fixture, $B_e = B - \frac{(B-B_N)^2}{B}$ is the specimen effective thickness, and $a_0$ is the measured initial crack size. If the specimen tested is of the compact tension type, the formula in cell O32 becomes:

[12] The linear fit must be determined by means of a separate spreadsheet/application.
[13] In this investigation, however, tests have been considered reliable if $EBW$ was at least 50 % of $BW_{min}$.

$$C_{exp} = \frac{1}{EB_e}\left(\frac{W+a_0}{W-a_0}\right)^2 \left[2.163 + 12.219\left(\frac{a_0}{W}\right) - 20.065\left(\frac{a_0}{W}\right)^2 - 0.9925\left(\frac{a_0}{W}\right)^3 + 20.609\left(\frac{a_0}{W}\right)^4 - 9.9314\left(\frac{a_0}{W}\right)^5\right] \quad \text{(A.5)}$$

The correct equation (C.4 or C.5) is automatically selected in cell O32 based on the option selected by the user in cell I1 of the "Data" worksheet.

- Finally, the "Calculations" worksheet also provides a force smoothness verification in graphical form (example in **Figure C.7**). Two lines parallel to the elastic portion of the test record constitute a "force smoothness band". Test data must be contained inside such band, up to the point ("Final crossover") where the test record visibly departs from linearity (represented by a red circle, which must be manually placed on the graph).

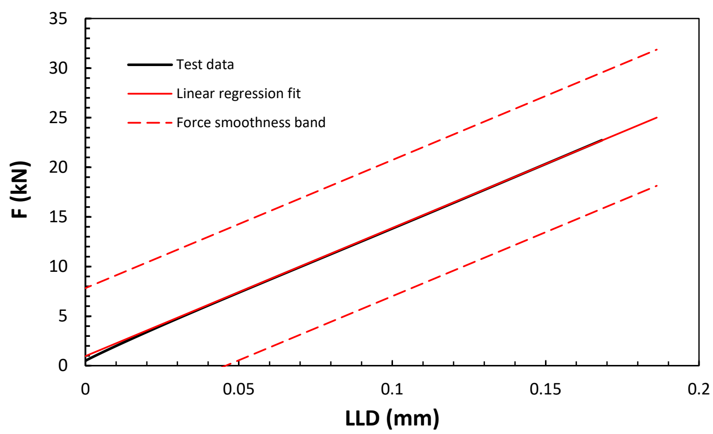



**Figure C.7** – Example of force smoothness verification.

## C.11 Worksheet "Fracture surface"

The last worksheet of this spreadsheet allows users to paste a digital picture of the specimen's fracture surface, and to input the measured value of initial crack size, $a_0$, in cell N2. If this worksheet is not needed, it can be easily removed – but in this case, the measured $a_0$ must be input by the user in cell H7 of worksheet "Calculations".

# Appendix D: Master Curve Analysis for Higher Rate Tests on 1TC(T) Specimens of Biblis C Base Material

**Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range**
**ASTM E1921-20**

Analysis for Homogeneous Materials - Multi-Temperature Approach

1. Material characteristics

*Material specifications:* **Biblis C base - 1TC(T) specimens, 1st series - 30 mm/s**

| Fit coefficients | $\sigma_{YS}$ (MPa) | E (GPa) | $\sigma_{YS,RT}$ (MPa) |
|---|---|---|---|
| (2nd order term) A = | -3.13E-03 | 0 | |
| (1st order term) B = | -0.8875 | -0.051 | |
| (Intercept) C = | 494.24992 | 210.5 | |

2. Dimensional and crack growth requirements

Poisson's Ratio = 0.3

| Specimen code | T (°C) | $a_o$ (mm) | W (mm) | B (mm) | $b_o$ (mm) | $\Delta a$ (mm) {Excessive crack growth} | $K_{Jc}$ (MPa√m) {Above $K_{limit}$} | $\sigma_{ys}$ (MPa) | E (GPa) | $K_{lim}$ (MPa√m) | Censored? (YES/NO) | $K_{Jcanalysis}$ (MPa√m) | Test Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| L1.1 AA2 | -40 | 26.040 | 50 | 25 | 23.96 | 0.000 | 67.09 | 524.75 | 212.54 | 312.87 | NO | 67.09 | 1.76E+03 |
| L1.1 AA1 | -40 | 26.069 | 50 | 25 | 23.93 | 0.000 | 23.63 | 524.75 | 212.54 | 312.68 | NO | 23.63 | 8.34E+02 |
| L1.1 BB2 | -30 | 26.044 | 50 | 25 | 23.96 | 0.000 | 46.46 | 518.06 | 212.03 | 310.47 | NO | 46.46 | 1.36E+03 |
| L1.1 AC3 | -20 | 26.048 | 50 | 25 | 23.95 | 0.000 | 61.41 | 510.75 | 211.52 | 307.87 | NO | 61.41 | 1.50E+03 |
| L1.1 AA3 | -20 | 26.027 | 50 | 25 | 23.97 | 0.000 | 61.87 | 510.75 | 211.52 | 308.01 | NO | 61.87 | 1.58E+03 |
| L1.1 BB6 | -15 | 25.998 | 50 | 25 | 24.00 | 0.000 | 52.60 | 506.86 | 211.27 | 306.83 | NO | 52.60 | 1.42E+03 |
| L1.1 BB5 | -10 | 26.080 | 50 | 25 | 23.92 | 0.000 | 103.83 | 502.81 | 211.01 | 304.90 | NO | 103.83 | 2.08E+03 |
| L1.1 BB1 | -10 | 26.184 | 50 | 25 | 23.82 | 0.000 | 117.20 | 502.81 | 211.01 | 304.23 | NO | 117.20 | 2.07E+03 |
| L1.1 AC1 | -2 | 26.134 | 50 | 25 | 23.87 | 0.000 | 141.40 | 496.01 | 210.60 | 302.19 | NO | 141.40 | 2.26E+03 |
| L1.1 AC2 | 0 | 26.074 | 50 | 25 | 23.93 | 0.000 | 109.92 | 494.25 | 210.50 | 301.96 | NO | 109.92 | 2.12E+03 |

3. Application of the multi-temperature approach for the calculation of the reference temperature

| Specimen code | T (°C) | $K_{Jc(anal)}$ (MPa√m) | $K_{Jc,1T}$ (MPa√m) | $\delta_i$ | $r_i$ | $n_i$ | 1st member | 2st member |
|---|---|---|---|---|---|---|---|---|
| L1.1 AA2 | -40 | 67.1 | 66.9 | 1 | 1 | 0.125 | 0.0100 | 0.0089 |
| L1.1 AA1 | -40 | 23.6 | 23.6 | 1 | 1 | 0.125 | 0.0100 | 0.0000 |
| L1.1 BB2 | -30 | 46.5 | 46.4 | 1 | 1 | 0.143 | 0.0104 | 0.0005 |
| L1.1 AC3 | -20 | 61.4 | 61.2 | 1 | 1 | 0.143 | 0.0108 | 0.0017 |
| L1.1 AA3 | -20 | 61.9 | 61.7 | 1 | 1 | 0.143 | 0.0108 | 0.0018 |
| L1.1 BB6 | -15 | 52.6 | 52.5 | 1 | 1 | 0.167 | 0.0110 | 0.0005 |
| L1.1 BB5 | -10 | 103.8 | 103.5 | 1 | 1 | 0.167 | 0.0111 | 0.0154 |
| L1.1 BB1 | -10 | 117.2 | 116.8 | 1 | 1 | 0.167 | 0.0111 | 0.0278 |
| L1.1 AC1 | -2 | 141.4 | 140.9 | 1 | 1 | 0.167 | 0.0114 | 0.0408 |
| L1.1 AC2 | 0 | 109.9 | 109.6 | 1 | 1 | 0.167 | 0.0114 | 0.0108 |

**USE TEMPERATURE LIMITS?** YES
**($T_o$ - 50 °C ≤ T ≤ $T_o$ + 50 °C)**

T limits (°C)
-51.9
48.1

Sum of 1° member: 0.108

Sum of 2° member: 0.108

Difference: 0.000

**$T_o$ = -1.9 °C**
**(valid per ASTM E1921)**

$\sum_i r_i n_i$ = 1.5

# tests = 10
N = 10
r = 10

$K_{min}$ = 20 MPa√m

**$K_{o,eq}$ = 88.5 MPa√m**

**$K_{med,eq}$ = 82.5 MPa√m**

**dK/dt = 1.70E+03 MPa√m/s**

4. Master curve fit to data

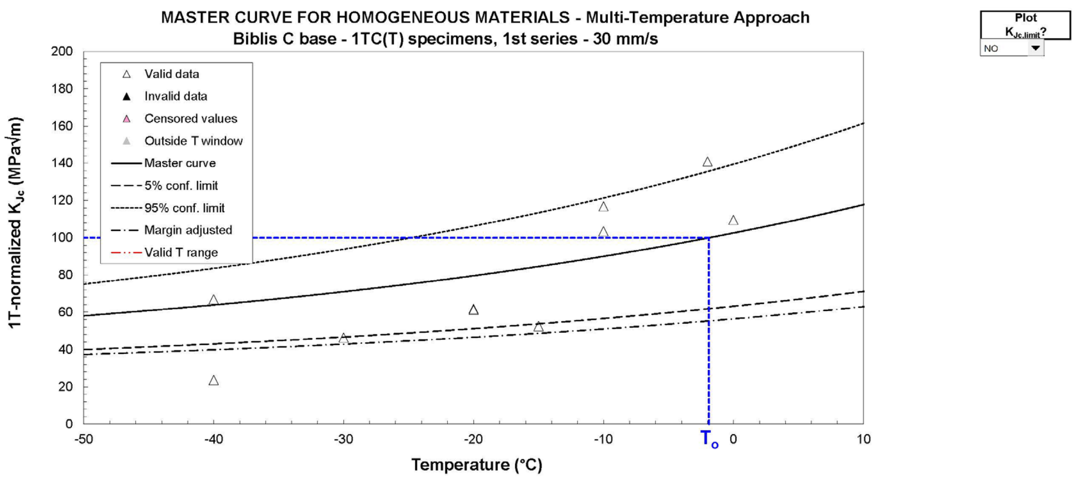
Margin adj. (85 % conf.): 10.3 °C (est.)

Stand. dev. on $T_o$ = 7.2 °C (est.)

| T (°C) | $K_{Jc(exp)}$ (MPa√m) | $K_{Jc,1T}$ (MPa√m) | $K_{MC(1T)}$ (MPa√m) | 5% conf. (MPa√m) | 95% conf. (MPa√m) | 5% L.B. (MPa√m) |
|---|---|---|---|---|---|---|
| -40 | 67.1 | 66.9 | | | | |
| -40 | 23.6 | 23.6 | | | | |
| -30 | 46.5 | 46.4 | | | | |
| -20 | 61.4 | 61.2 | | | | |
| -20 | 61.9 | 61.7 | | | | |
| -15 | 52.6 | 52.5 | | | | |
| -10 | 103.8 | 103.5 | | | | |
| -10 | 117.2 | 116.8 | | | | |
| -2 | 141.4 | 140.9 | | | | |
| 0 | 109.9 | 109.6 | | | | |
| -50 | | | 58.1 | 39.9 | 75.1 | 37.3 |
| -40 | | | 63.9 | 43.0 | 83.6 | 39.8 |
| -30 | | | 71.0 | 46.7 | 93.9 | 42.9 |
| -20 | | | 79.6 | 51.2 | 106.3 | 46.6 |
| -10 | | | 90.0 | 56.6 | 121.3 | 51.1 |
| 0 | | | 102.6 | 63.2 | 139.5 | 56.5 |
| 10 | | | 117.7 | 71.2 | 161.5 | 63.0 |
| 20 | | | 136.1 | 80.8 | 188.0 | 70.9 |
| 30 | | | 158.3 | 92.4 | 220.2 | 80.4 |
| 40 | | | 185.2 | 106.5 | 259.0 | 92.0 |

| | |
|---|---|
| Start T (°C) | -50 |
| Step T (°C) | 10 |
| Min test T (°C) | -40 |
| Max test T (°C) | 0 |
| Max $K_{Jc,1T}$ | 140.9 |

| | | | | |
|---|---|---|---|---|
| 50 | 217.6 | 123.5 | 306.0 | 106.0 |
| 60 | 256.9 | 144.0 | 362.8 | 122.9 |
| 70 | 304.4 | 168.9 | 431.5 | 143.3 |
| 80 | 361.8 | 198.9 | 514.6 | 168.0 |
| 90 | 431.2 | 235.3 | 615.1 | 197.9 |
| 100 | 515.1 | 279.2 | 736.5 | 234.0 |
| 110 | 616.7 | 332.3 | 883.5 | 277.7 |



**Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range**
**ASTM E1921-20**

Homogeneity Screening Procedure based on SINTAP method

1. Data censoring

| *Specimen code* | *T (°C)* | *$K_{Jc(exp)}$ (MPa √m)* | *$K_{Jci,1T}$ (MPa √m)* | *$K_{CENS}$ (MPa √m)* | $\delta_i$ | *$K_{analysis}$ (MPa √m)* |
|---|---|---|---|---|---|---|
| L1.1 AA2 | -40 | 67.1 | 66.9 | 55.9 | 0 | 55.9 |
| L1.1 AA1 | -40 | 23.6 | 23.6 | 55.9 | 1 | 23.6 |
| L1.1 BB2 | -30 | 46.5 | 46.4 | 61.3 | 1 | 46.4 |
| L1.1 AC3 | -20 | 61.4 | 61.2 | 67.8 | 1 | 61.2 |
| L1.1 AA3 | -20 | 61.9 | 61.7 | 67.8 | 1 | 61.7 |
| L1.1 BB6 | -15 | 52.6 | 52.5 | 71.6 | 1 | 52.5 |
| L1.1 BB5 | -10 | 103.8 | 103.5 | 75.8 | 0 | 75.8 |
| L1.1 BB1 | -10 | 117.2 | 116.8 | 75.8 | 0 | 75.8 |
| L1.1 AC1 | -2 | 141.4 | 140.9 | 83.3 | 0 | 83.3 |
| L1.1 AC2 | 0 | 109.9 | 109.6 | 85.3 | 0 | 85.3 |

Benchmark $T_o$ = 12.4 °C

2. Analysis of the censored data and obtainment of a new estimate of $T_o$

**USE LIMITS :** YES

T limits (°C)
-37.2
62.8

| *Specimen code* | *T (°C)* | *$K_{analysis}$ (MPa √m)* | $\delta_i$ | *1° member* | *2° member* |
|---|---|---|---|---|---|
| L1.1 AA2 | -40 | 55.9 | 0 | 0.0000 | 0.0000 |
| L1.1 AA1 | -40 | 23.6 | 1 | 0.0000 | 0.0000 |
| L1.1 BB2 | -30 | 46.4 | 1 | 0.0098 | 0.0011 |
| L1.1 AC3 | -20 | 61.2 | 1 | 0.0103 | 0.0040 |
| L1.1 AA3 | -20 | 61.7 | 1 | 0.0103 | 0.0042 |
| L1.1 BB6 | -15 | 52.5 | 1 | 0.0105 | 0.0012 |
| L1.1 BB5 | -10 | 75.8 | 0 | 0.0000 | 0.0075 |
| L1.1 BB1 | -10 | 75.8 | 0 | 0.0000 | 0.0075 |
| L1.1 AC1 | -2 | 83.3 | 0 | 0.0000 | 0.0077 |
| L1.1 AC2 | 0 | 85.3 | 0 | 0.0000 | 0.0077 |

*Sum of 1° member:* 0.041

*Sum of 2° member:* 0.041

*Difference :* 0.000

**$T_o$ = 12.8 °C**

**$T_{0(stepn)} \geq T_{0(stepn-1)}$ + 0.5 °C ?**
**NO - Analysis Completed**

**$T_{0scrn}$ = 12.8 °C**

**Screening Criterion**
THE MATERIAL IS
INHOMOGENEOUS

**Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range**
**ASTM E1921-20**

Determination of the Reference Temperature for Inhomogeneous Materials (Simplified Method)

1. Calculation of the maximum value of $T_o$ (based on a single data point) and establishment of $T_o$ for the data set

| Specimen id | T (°C) | $K_{Jc[exp]}$ (MPa√m) | $K_{Jci}$ (MPa√m) | $\delta_i$ | $T_{oi}$ (°C) |
|---|---|---|---|---|---|
| L1.1 AA2 | -40 | 67.1 | 66.9 | 1 | -42.5 |
| L1.1 AA1 | -40 | 23.6 | 23.6 | 1 | |
| L1.1 BB2 | -30 | 46.5 | 46.4 | 1 | 3.8 |
| L1.1 AC3 | -20 | 61.4 | 61.2 | 1 | -14.7 |
| L1.1 AA3 | -20 | 61.9 | 61.7 | 1 | -15.4 |
| L1.1 BB6 | -15 | 52.6 | 52.5 | 1 | 5.1 |
| L1.1 BB5 | -10 | 103.8 | 103.5 | 1 | -45.9 |
| L1.1 BB1 | -10 | 117.2 | 116.8 | 1 | -54.2 |
| L1.1 AC1 | -2 | 141.4 | 140.9 | 1 | -58.6 |
| L1.1 AC2 | 0 | 109.9 | 109.6 | 1 | -39.9 |

$T_{omax}$ = 5.1 °C

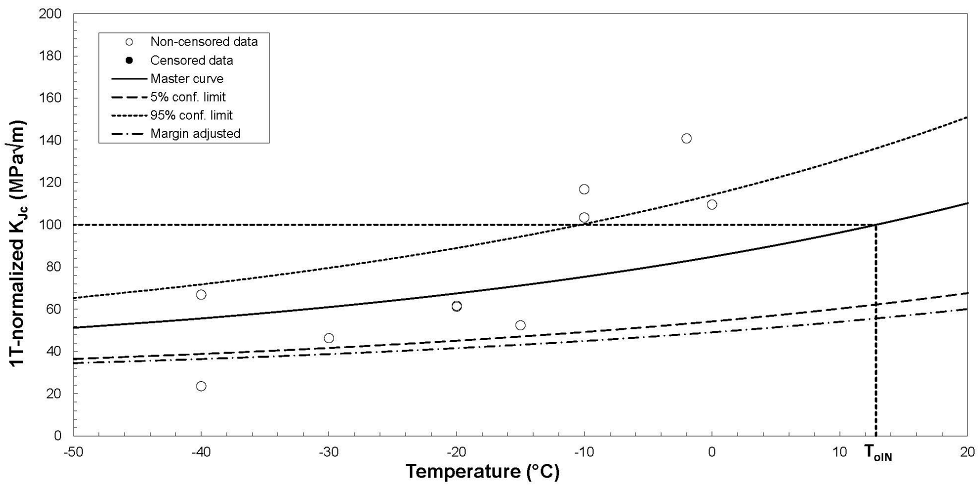
$T_{oscrn}$ = 12.8 °C

$T_{omax}$ - $T_{oscrn}$ > 8 °C : **NO**
Number of tests N = 10
**$T_{oIN}$ = 12.8 °C**

2. Final Master Curve fit to data

Margin adj. (85 % conf.): 10.3 °C (est.) Stand. dev. on $T_o$ = 7.2 °C (est.)

| | T (°C) | $K_{Jc(exp)}$ (MPa √m) | $K_{Jc(1T)}$ (MPa √m) | $K_{MC(1T)}$ (MPa √m) | 5% conf. (MPa √m) | 95% conf. (MPa √m) | 5% L.B. (MPa √m) |
|---|---|---|---|---|---|---|---|
| | -40.0 | 67.1 | 66.9 | | | | |
| | -40.0 | 23.6 | 23.6 | | | | |
| | -30.0 | 46.5 | 46.4 | | | | |
| | -20.0 | 61.4 | 61.2 | | | | |
| | -20.0 | 61.9 | 61.7 | | | | |
| | -15.0 | 52.6 | 52.5 | | | | |
| | -10.0 | 103.8 | 103.5 | | | | |
| | -10.0 | 117.2 | 116.8 | | | | |
| | -2.0 | 141.4 | 140.9 | | | | |
| | 0.0 | 109.9 | 109.6 | | | | |
| Start T (°C) | -50 | | | 51.2 | 36.5 | 65.3 | 34.5 |
| -50 | -40 | | | 55.7 | 38.8 | 71.8 | 36.4 |
| Step T (°C) | -30 | | | 61.0 | 41.7 | 79.6 | 38.7 |
| 10 | -20 | | | 67.5 | 45.1 | 89.0 | 41.6 |
| Min test T (°C) | -10 | | | 75.4 | 49.2 | 100.4 | 45.0 |
| -40 | 0 | | | 84.9 | 54.2 | 114.2 | 49.1 |
| Max test T (°C) | 10 | | | 96.3 | 60.3 | 130.8 | 54.1 |
| 0 | 20 | | | 110.2 | 67.6 | 151.0 | 60.1 |
| Max $K_{Jci}$ (MPa√m) | 30 | | | 127.0 | 76.5 | 175.4 | 67.4 |
| 141.3984201 | 40 | | | 147.3 | 87.2 | 204.8 | 76.2 |
| | 50 | | | 171.8 | 100.2 | 240.5 | 86.8 |
| | 60 | | | 201.5 | 115.8 | 283.6 | 99.7 |
| | 70 | | | 237.4 | 134.8 | 335.7 | 115.3 |
| | 80 | | | 280.8 | 157.7 | 398.7 | 134.1 |
| | 90 | | | 333.3 | 185.4 | 474.9 | 156.9 |
| | 100 | | | 396.7 | 218.9 | 567.0 | 184.4 |
| | 110 | | | 473.5 | 259.4 | 678.5 | 217.8 |


Non-censored data
Censored data
Master curve
5% conf. limit
95% conf. limit
Margin adjusted
1T-normalized $K_{Jc}$ (MPa√m)
Temperature (°C)
$T_{oIN}$
200
180
160
140
120
100
80
60
40
20
0
-50
-40
-30
-20
-10
0
10
20

# Appendix E: Master Curve Analysis for Lower Rate Tests on 1TC(T) Specimens of Biblis C Base Material

## Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range
## ASTM E1921-20

Analysis for Homogeneous Materials - Multi-Temperature Approach

1. Material characteristics

*Material specifications:* **Biblis C base - 1TC(T) specimens, 2nd series - 3 mm/s**

| Fit coefficients | $\sigma_{YS}$ (MPa) | E (GPa) | $\sigma_{YS,RT}$ (MPa) |
|---|---|---|---|
| ($2^{nd}$ order term) A = | -3.13E-03 | 0 | |
| ($1^{st}$ order term) B = | -0.8875 | -0.051 | |
| (Intercept) C = | 494.24992 | 210.5 | |

2. Dimensional and crack growth requirements

Poisson's Ratio = 0.3

| Specimen code | T (°C) | $a_o$ (mm) | W (mm) | B (mm) | $b_o$ (mm) | $\Delta a$ (mm) {Excessive crack growth} | $K_{Jc}$ (MPa√m) {Above $K_{limit}$} | $\sigma_{ys}$ (MPa) | E (GPa) | $K_{lim}$ (MPa√m) | Censored? (YES/NO) | $K_{Jcanalysis}$ (MPa√m) | Test Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| L1.3-AA5 | -20 | 25.864 | 50 | 25 | 24.14 | 0.000 | 78.94 | 510.75 | 211.52 | 309.05 | NO | 78.94 | 3.21E+02 |
| L1.3-BB7 | -20 | 25.646 | 50 | 25 | 24.35 | 0.000 | 86.94 | 510.75 | 211.52 | 310.44 | NO | 86.94 | 2.00E+02 |
| L1.2-BB7 | -18 | 25.943 | 50 | 25 | 24.06 | 0.000 | 91.38 | 509.21 | 211.42 | 308.00 | NO | 91.38 | 2.02E+02 |
| L1.3-BB1 | -10 | 25.910 | 50 | 25 | 24.09 | 0.000 | 120.03 | 502.81 | 211.01 | 305.98 | NO | 120.03 | 3.49E+02 |
| L1.2-BB3 | -10 | 25.466 | 50 | 25 | 24.53 | 0.000 | 97.59 | 502.81 | 211.01 | 308.78 | NO | 97.59 | 2.11E+02 |
| L1.3-AC4 | -5 | 25.865 | 50 | 25 | 24.14 | 0.000 | 132.55 | 498.61 | 210.76 | 304.80 | NO | 132.55 | 2.31E+02 |
| L1.3-AA2 | 0 | 26.068 | 50 | 25 | 23.93 | 0.000 | 207.47 | 494.25 | 210.50 | 302.00 | NO | 207.47 | 4.59E+02 |
| L1.2-AC2 | 0 | 25.767 | 50 | 25 | 24.23 | 0.000 | 172.95 | 494.25 | 210.50 | 303.89 | NO | 172.95 | 2.77E+02 |
| L1.3-AC6 | 0 | 26.325 | 50 | 25 | 23.67 | 0.000 | 181.55 | 494.25 | 210.50 | 300.37 | NO | 181.55 | 2.71E+02 |

3. Application of the multi-temperature approach for the calculation of the reference temperature

| Specimen code | T (°C) | $K_{Jc(anal)}$ (MPa√m) | $K_{Jc,1T}$ (MPa√m) | $\delta_i$ | $r_i$ | $n_i$ | $1^{st}$ member | $2^{st}$ member |
|---|---|---|---|---|---|---|---|---|
| L1.3-AA5 | -20 | 78.9 | 78.7 | 1 | 1 | 0.167 | 0.0115 | 0.0015 |
| L1.3-BB7 | -20 | 86.9 | 86.7 | 1 | 1 | 0.167 | 0.0115 | 0.0026 |
| L1.2-BB7 | -18 | 91.4 | 91.1 | 1 | 1 | 0.167 | 0.0116 | 0.0029 |
| L1.3-BB1 | -10 | 120.0 | 119.6 | 1 | 1 | 0.167 | 0.0117 | 0.0066 |
| L1.2-BB3 | -10 | 97.6 | 97.3 | 1 | 1 | 0.167 | 0.0117 | 0.0024 |
| L1.3-AC4 | -5 | 132.5 | 132.1 | 1 | 1 | 0.167 | 0.0118 | 0.0075 |
| L1.3-AA2 | 0 | 207.5 | 206.7 | 1 | 1 | 0.167 | 0.0119 | 0.0413 |
| L1.2-AC2 | 0 | 172.9 | 172.3 | 1 | 1 | 0.167 | 0.0119 | 0.0183 |
| L1.3-AC6 | 0 | 181.6 | 180.9 | 1 | 1 | 0.167 | 0.0119 | 0.0228 |

**USE TEMPERATURE LIMITS?** YES
**($T_o$ - 50 °C ≤ T ≤ $T_o$ + 50 °C)**

T limits (°C)
-75.9
24.1

Sum of 1° member: 0.106

Sum of 2° member: 0.106

Difference: 0.000

**$T_o$ = -25.9 °C**
**(valid per ASTM E1921)**

$\sum_i r_i n_i$ = 1.5

# tests = 9
N = 9
r = 9

$K_{min}$ = 20 MPa√m

**$K_{o,eq}$ = 137.5 MPa√m**

**$K_{med,eq}$ = 127.2 MPa√m**

**dK/dt = 2.80E+02 MPa√m/s**

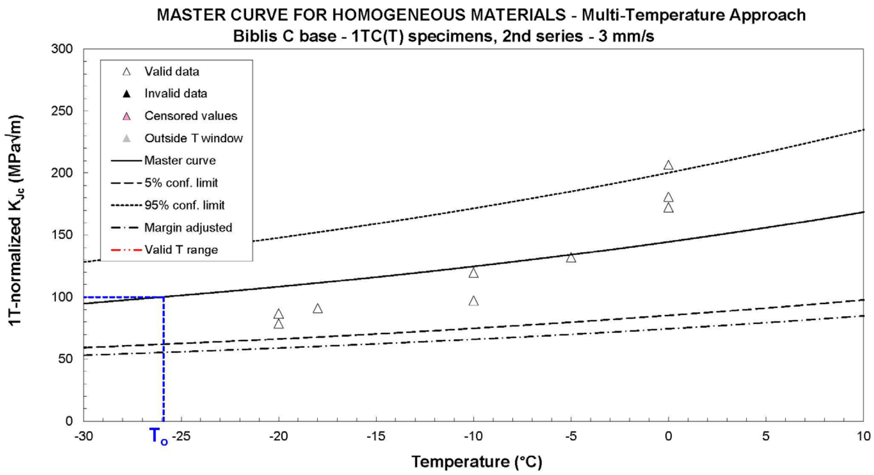
4. Master curve fit to data

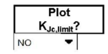
Margin adj. (85 % conf.): 10.4 °C (est.)    Stand. dev. on $T_o$ = 7.2 °C (est.)

| | |
|---|---|
| Start T (°C) | -30 |
| Step T (°C) | 10 |
| Min test T (°C) | -20 |
| Max test T (°C) | 0 |
| Max $K_{Jc,1T}$ | 206.7 |

| T (°C) | $K_{Jc(exp)}$ (MPa√m) | $K_{Jc,1T}$ (MPa√m) | $K_{MC(1T)}$ (MPa√m) | 5% conf. (MPa√m) | 95% conf. (MPa√m) | 5% L.B. (MPa√m) |
|---|---|---|---|---|---|---|
| -20 | 78.9 | 78.7 | | | | |
| -20 | 86.9 | 86.7 | | | | |
| -18 | 91.4 | 91.1 | | | | |
| -10 | 120.0 | 119.6 | | | | |
| -10 | 97.6 | 97.3 | | | | |
| -5 | 132.5 | 132.1 | | | | |
| 0 | 207.5 | 206.7 | | | | |
| 0 | 172.9 | 172.3 | | | | |
| 0 | 181.6 | 180.9 | | | | |
| -30 | | | 94.8 | 59.1 | 128.2 | 53.1 |
| -20 | | | 108.3 | 66.2 | 147.8 | 58.9 |
| -10 | | | 124.7 | 74.8 | 171.5 | 65.9 |
| 0 | | | 144.5 | 85.2 | 200.2 | 74.4 |
| 10 | | | 168.5 | 97.7 | 234.9 | 84.7 |
| 20 | | | 197.5 | 112.9 | 276.8 | 97.2 |
| 30 | | | 232.5 | 131.2 | 327.5 | 112.3 |
| 40 | | | 274.9 | 153.4 | 388.8 | 130.5 |
| 50 | | | 326.1 | 180.2 | 463.0 | 152.5 |
| 60 | | | 388.1 | 212.7 | 552.7 | 179.1 |
| 70 | | | 463.0 | 251.9 | 661.1 | 211.3 |
| 80 | | | 553.6 | 299.3 | 792.2 | 250.3 |

| | | | | |
|---|---|---|---|---|
| 90 | 663.2 | 356.7 | 950.8 | 297.3 |
| 100 | 795.6 | 426.0 | 1142.5 | 354.3 |
| 110 | 955.9 | 509.9 | 1374.3 | 423.1 |
| 120 | 1149.6 | 611.3 | 1654.7 | 506.4 |
| 130 | 1383.9 | 734.0 | 1993.7 | 607.1 |



**Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range**
**ASTM E1921-20**

Homogeneity Screening Procedure based on SINTAP method

1. Data censoring

| Specimen code | T (°C) | $K_{Jc(exp)}$ (MPa √m) | $K_{Jci,1T}$ (MPa √m) | $K_{CENS}$ (MPa √m) | $\delta_i$ | $K_{analysis}$ (MPa √m) |
|---|---|---|---|---|---|---|
| L1.3-AA5 | -20 | 78.9 | 78.7 | 100.6 | 1 | 78.7 |
| L1.3-BB7 | -20 | 86.9 | 86.7 | 100.6 | 1 | 86.7 |
| L1.2-BB7 | -18 | 91.4 | 91.1 | 103.3 | 1 | 91.1 |
| L1.3-BB1 | -10 | 120.0 | 119.6 | 115.4 | 0 | 115.4 |
| L1.2-BB3 | -10 | 97.6 | 97.3 | 115.4 | 1 | 97.3 |
| L1.3-AC4 | -5 | 132.5 | 132.1 | 123.9 | 0 | 123.9 |
| L1.3-AA2 | 0 | 207.5 | 206.7 | 133.2 | 0 | 133.2 |
| L1.2-AC2 | 0 | 172.9 | 172.3 | 133.2 | 0 | 133.2 |
| L1.3-AC6 | 0 | 181.6 | 180.9 | 133.2 | 0 | 133.2 |

Benchmark $T_o$ = -20.5 °C

2. Analysis of the censored data and obtainment of a new estimate of $T_o$

USE LIMITS : YES

T limits (°C)
-72.7
27.3

| Specimen code | T (°C) | $K_{analysis}$ (MPa √m) | $\delta_i$ | 1° member | 2° member |
|---|---|---|---|---|---|
| L1.3-AA5 | -20 | 78.7 | 1 | 0.0114 | 0.0019 |
| L1.3-BB7 | -20 | 86.7 | 1 | 0.0114 | 0.0031 |
| L1.2-BB7 | -18 | 91.1 | 1 | 0.0115 | 0.0036 |
| L1.3-BB1 | -10 | 115.4 | 0 | 0.0000 | 0.0068 |
| L1.2-BB3 | -10 | 97.3 | 1 | 0.0117 | 0.0029 |
| L1.3-AC4 | -5 | 123.9 | 0 | 0.0000 | 0.0069 |
| L1.3-AA2 | 0 | 133.2 | 0 | 0.0000 | 0.0069 |
| L1.2-AC2 | 0 | 133.2 | 0 | 0.0000 | 0.0069 |
| L1.3-AC6 | 0 | 133.2 | 0 | 0.0000 | 0.0069 |

*Sum of 1° member:* 0.046

*Sum of 2° member:* 0.046

*Difference :* 0.000

**$T_o$ = -22.7 °C**

**$T_{0(stepn)} \geq T_{0(stepn-1)}$ + 0.5 °C ?**
**NO - Analysis Completed**

**$T_{0scrn}$ = -20.5 °C**

**Screening Criterion**
THE MATERIAL IS
HOMOGENEOUS

# Appendix F: Master Curve Analysis for Higher Rate Tests on SE(B) 20/40 Specimens of Biblis C Base Material

**Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range**
**ASTM E1921-20**

Analysis for Homogeneous Materials - Multi-Temperature Approach

1. Material characteristics

*Material specifications:* **Biblis C base - SE(B) specimens - 30 mm/s**

| Fit coefficients | $\sigma_{YS}$ (MPa) | E (GPa) | $\sigma_{YS,RT}$ (MPa) |
|---|---|---|---|
| (2nd order term) A = | -3.13E-03 | 0 | |
| (1st order term) B = | -0.8875 | -0.051 | |
| (Intercept) C = | 494.24992 | 210.5 | |

2. Dimensional and crack growth requirements

Poisson's Ratio = 0.3

| Specimen code | T (°C) | $a_o$ (mm) | W (mm) | B (mm) | $b_o$ (mm) | $\Delta a$ (mm) {Excessive crack growth} | $K_{Jc}$ (MPa√m) {Above $K_{limit}$} | $\sigma_{ys}$ (MPa) | E (GPa) | $K_{lim}$ (MPa√m) | Censored? (YES/NO) | $K_{Jc analysis}$ (MPa√m) | Test Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 4.1 B11 | -20 | 20.986 | 40 | 20 | 19.01 | 0.000 | 63.94 | 510.75 | 211.52 | 274.31 | NO | 63.94 | 1.52E+03 |
| 3.1 B01 | -10 | 20.647 | 40 | 20 | 19.35 | 0.000 | 101.79 | 502.81 | 211.01 | 274.25 | NO | 101.79 | 2.17E+03 |
| 4.1 A11 | -10 | 20.752 | 40 | 20 | 19.25 | 0.000 | 77.61 | 502.81 | 211.01 | 273.51 | NO | 77.61 | 1.81E+03 |
| 3.1 B04 | 0 | 20.697 | 40 | 20 | 19.30 | 0.000 | 60.88 | 494.25 | 210.50 | 271.23 | NO | 60.88 | 1.56E+03 |
| 4.1 A09 | 0 | 21.176 | 40 | 20 | 18.82 | 0.000 | 94.27 | 494.25 | 210.50 | 267.84 | NO | 94.27 | 2.05E+03 |
| 4.1 B01 | 5 | 21.293 | 40 | 20 | 18.71 | 0.000 | 192.14 | 489.73 | 210.25 | 265.62 | NO | 192.14 | 2.89E+03 |
| 3.1 B10 | 10 | 20.686 | 40 | 20 | 19.31 | 0.000 | 230.35 | 485.06 | 209.99 | 268.44 | NO | 230.35 | 2.95E+03 |
| 4.1 B07 | 12 | 21.026 | 40 | 20 | 18.97 | 0.000 | 278.08 | 483.15 | 209.89 | 265.48 | YES | 265.48 | 2.76E+03 |

3. Application of the multi-temperature approach for the calculation of the reference temperature

| Specimen code | T (°C) | $K_{Jc(anal)}$ (MPa√m) | $K_{Jc,1T}$ (MPa√m) | $\delta_i$ | $r_i$ | $n_i$ | 1st member | 2nd member |
|---|---|---|---|---|---|---|---|---|
| 4.1 B11 | -20 | 63.9 | 61.4 | 1 | 1 | 0.167 | 0.0115 | 0.0004 |
| 3.1 B01 | -10 | 101.8 | 97.0 | 1 | 1 | 0.167 | 0.0118 | 0.0022 |
| 4.1 A11 | -10 | 77.6 | 74.3 | 1 | 1 | 0.167 | 0.0118 | 0.0005 |
| 3.1 B04 | 0 | 60.9 | 58.5 | 1 | 1 | 0.167 | 0.0120 | 0.0001 |
| 4.1 A09 | 0 | 94.3 | 90.0 | 1 | 1 | 0.167 | 0.0120 | 0.0008 |
| 4.1 B01 | 5 | 192.1 | 182.2 | 1 | 1 | 0.167 | 0.0120 | 0.0155 |
| 3.1 B10 | 10 | 230.3 | 218.1 | 1 | 1 | 0.167 | 0.0121 | 0.0244 |
| 4.1 B07 | 12 | 265.5 | 251.2 | 0 | 0 | 0.000 | 0.0000 | 0.0394 |

**USE TEMPERATURE LIMITS?** YES
**($T_o$ - 50 °C ≤ T ≤ $T_o$ + 50 °C)**

T limits (°C)
-76.9
23.1

Sum of 1° member: 0.083
Sum of 2° member: 0.083
Difference: 0.000

**$T_o$ = -26.9 °C**
**(valid per ASTM E1921)**

$\sum_i r_i n_i$ = 1.2

\# tests = 8
N = 8
r = 7

$K_{min}$ = 20 MPa√m

**$K_{o,eq}$ = 152.5 MPa√m**

**$K_{med,eq}$ = 140.9 MPa√m**

**dK/dt = 2.21E+03 MPa√m/s**

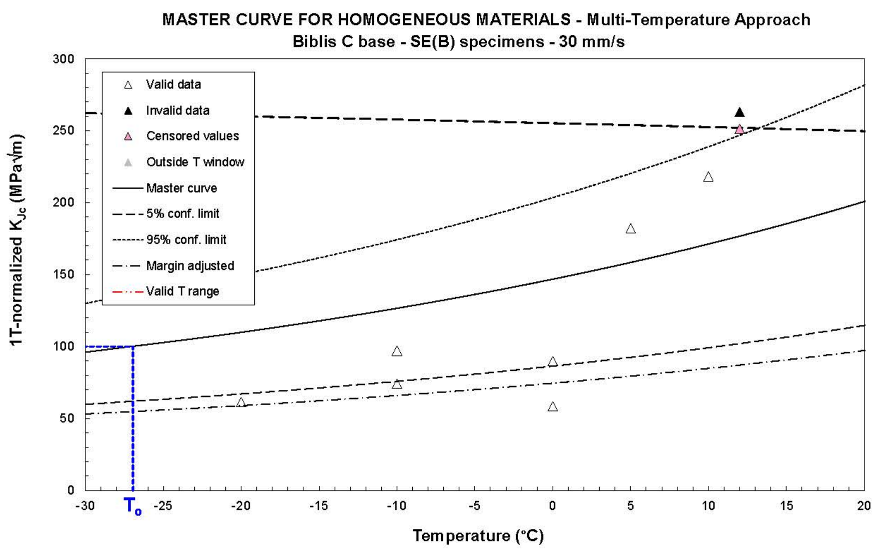
4. Master curve fit to data

Margin adj. (85 % conf.): 11.4 °C (est.)

$\sigma_{T_o}$ = 7.9 °C (est.)

| | T (°C) | $K_{Jc(exp)}$ (MPa√m) | $K_{Jc,1T}$ (MPa√m) | $K_{MC(1T)}$ (MPa√m) | *5% conf.* (MPa√m) | *95% conf.* (MPa√m) | *5% L.B.* (MPa√m) |
|---|---|---|---|---|---|---|---|
| | -20 | 63.9 | 61.4 | | | | |
| | -10 | 101.8 | 97.0 | | | | |
| | -10 | 77.6 | 74.3 | | | | |
| | 0 | 60.9 | 58.5 | | | | |
| | 0 | 94.3 | 90.0 | | | | |
| | 5 | 192.1 | 182.2 | | | | |
| | 10 | 230.3 | 218.1 | | | | |
| | 12 | 278.1 | 263.1 | | | | |
| Start T (°C) | -30 | | | 96.1 | 59.8 | 130.1 | 53.1 |
| -30 | -20 | | | 109.9 | 67.0 | 150.1 | 58.9 |

| | | | | | |
|---|---|---|---|---|---|
| Step T (°C) | -10 | 126.6 | 75.8 | 174.3 | 66.0 |
| 10 | 0 | 146.8 | 86.4 | 203.5 | 74.5 |
| Min test T (°C) | 10 | 171.2 | 99.2 | 238.9 | 84.8 |
| -20 | 20 | 200.8 | 114.6 | 281.6 | 97.3 |
| Max test T (°C) | 30 | 236.5 | 133.4 | 333.4 | 112.4 |
| 12 | 40 | 279.7 | 156.0 | 395.9 | 130.6 |
| Max $K_{Jc,1T}$ | 50 | 332.0 | 183.3 | 471.5 | 152.6 |
| 263.1 | 60 | 395.2 | 216.4 | 563.0 | 179.3 |
| | 70 | 471.6 | 256.4 | 673.6 | 211.5 |
| | 80 | 564.0 | 304.8 | 807.3 | 250.5 |
| | 90 | 675.8 | 363.3 | 969.0 | 297.6 |
| | 100 | 810.9 | 434.0 | 1164.6 | 354.6 |
| | 110 | 974.3 | 519.6 | 1401.1 | 423.6 |
| | 120 | 1171.9 | 623.0 | 1687.0 | 506.9 |
| | 130 | 1410.9 | 748.1 | 2032.8 | 607.7 |




Plot $K_{Jc,limit}$?

YES

**Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range**
**ASTM E1921-20**

Homogeneity Screening Procedure based on SINTAP method

1. Data censoring

| *Specimen code* | *T (°C)* | *$K_{Jc(exp)}$ (MPa √m)* | *$K_{Jcl,1T}$ (MPa √m)* | *$K_{CENS}$ (MPa √m)* | *$\delta_i$* | *$K_{analysis}$ (MPa √m)* |
|---|---|---|---|---|---|---|
| 4.1 B11 | -20 | 63.9 | 61.4 | 75.3 | 1 | 61.4 |
| 3.1 B01 | -10 | 101.8 | 97.0 | 84.7 | 0 | 84.7 |
| 4.1 A11 | -10 | 77.6 | 74.3 | 84.7 | 1 | 74.3 |
| 3.1 B04 | 0 | 60.9 | 58.5 | 96.2 | 1 | 58.5 |
| 4.1 A09 | 0 | 94.3 | 90.0 | 96.2 | 1 | 90.0 |
| 4.1 B01 | 5 | 192.1 | 182.2 | 102.8 | 0 | 102.8 |
| 3.1 B10 | 10 | 230.3 | 218.1 | 110.1 | 0 | 110.1 |
| 4.1 B07 | 12 | 278.1 | 263.1 | 113.2 | 0 | 113.2 |

Benchmark $T_o$ = 2.9 °C

2. Analysis of the censored data and obtainment of a new estimate of $T_o$

USE LIMITS : YES

T limits (°C)
-46.7
53.3

| *Specimen code* | *T (°C)* | *$K_{analysis}$ (MPa √m)* | *$\delta_i$* | *1° member* | *2° member* |
|---|---|---|---|---|---|
| 4.1 B11 | -20 | 61.4 | 1 | 0.0106 | 0.0023 |
| 3.1 B01 | -10 | 84.7 | 0 | 0.0000 | 0.0077 |
| 4.1 A11 | -10 | 74.3 | 1 | 0.0110 | 0.0038 |
| 3.1 B04 | 0 | 58.5 | 1 | 0.0113 | 0.0005 |
| 4.1 A09 | 0 | 90.0 | 1 | 0.0113 | 0.0056 |
| 4.1 B01 | 5 | 102.8 | 0 | 0.0000 | 0.0080 |
| 3.1 B10 | 10 | 110.1 | 0 | 0.0000 | 0.0081 |
| 4.1 B07 | 12 | 113.2 | 0 | 0.0000 | 0.0081 |

*Sum of 1° member:* 0.044

*Sum of 2° member:* 0.044

*Difference :* 0.000

**$T_o$ = 3.3 °C**

**$T_{0(stepn)} \geq T_{0(stepn-1)}$ + 0.5 °C ?**
**NO - Analysis Completed**

**$T_{0scrn}$ = 3.3 °C**

**Screening Criterion**
THE MATERIAL IS
INHOMOGENEOUS

## Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range
## ASTM E1921-20

Determination of the Reference Temperature for Inhomogeneous Materials (Simplified Method)

1. Calculation of the maximum value of $T_o$ (based on a single data point) and establishment of $T_o$ for the data set

| Specimen id | T (°C) | $K_{Jc(exp)}$ (MPa√m) | $K_{Jci}$ (MPa√m) | $\delta_i$ | $T_{oi}$ (°C) |
|---|---|---|---|---|---|
| 4.1 B11 | -20 | 63.9 | 61.4 | 1 | -11.5 |
| 3.1 B01 | -10 | 101.8 | 97.0 | 1 | -38.2 |
| 4.1 A11 | -10 | 77.6 | 74.3 | 1 | -17.9 |
| 3.1 B04 | 0 | 60.9 | 58.5 | 1 | 12.9 |
| 4.1 A09 | 0 | 94.3 | 90.0 | 1 | -22.7 |
| 4.1 B01 | 5 | 192.1 | 182.2 | 1 | -64.6 |
| 3.1 B10 | 10 | 230.3 | 218.1 | 1 | -70.5 |
| 4.1 B07 | 12 | 278.1 | 251.2 | 0 | |

$T_{omax}$ = 12.9 °C

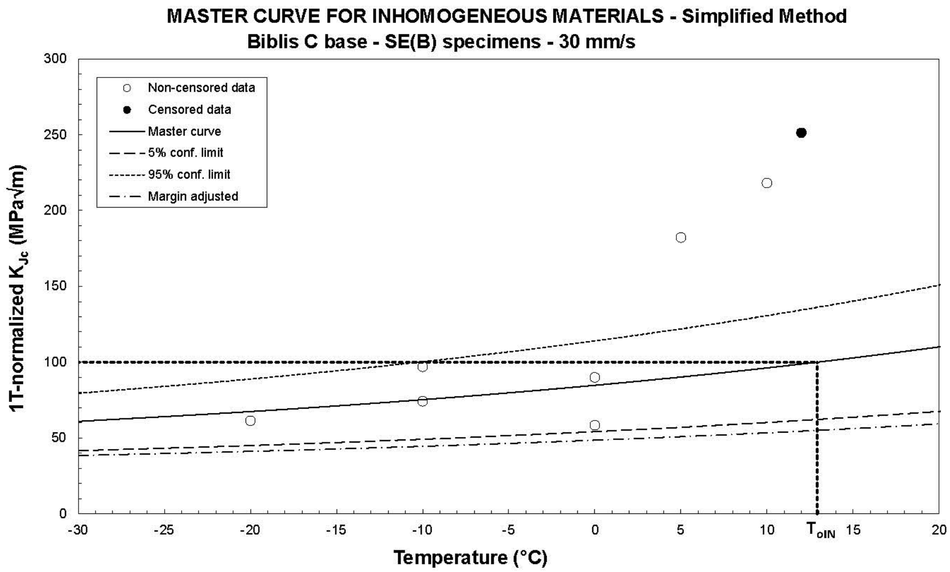
$T_{oscm}$ = 3.3 °C

$T_{omax} - T_{oscm} > 8$ °C : **YES**
Number of tests N = 8
**$T_{oIN}$ = 12.9 °C**

2. Final Master Curve fit to data

Margin adj. (85 % conf.): 11.4 °C (est.) Stand. dev. on $T_o$ = 7.9 °C (est.)

| | *T* (°C) | *$K_{Jc(exp)}$* (*MPa √m*) | *$K_{Jc(1T)}$* (*MPa √m*) | *$K_{MC(1T)}$* (*MPa √m*) | *5% conf.* (*MPa √m*) | *95% conf.* (*MPa √m*) | *5% L.B.* (*MPa √m*) |
|---|---|---|---|---|---|---|---|
| | -20.0 | 63.9 | 61.4 | | | | |
| | -10.0 | 101.8 | 97.0 | | | | |
| | -10.0 | 77.6 | 74.3 | | | | |
| | 0.0 | 60.9 | 58.5 | | | | |
| | 0.0 | 94.3 | 90.0 | | | | |
| | 5.0 | 192.1 | 182.2 | | | | |
| | 10.0 | 230.3 | 218.1 | | | | |
| | 12.0 | 265.5 | 251.2 | | | | |
| | #N/A | #N/A | #N/A | | | | |
| | #N/A | #N/A | #N/A | | | | |
| Start T (°C) | -30 | | | 61.0 | 41.6 | 79.5 | 38.5 |
| -30 | -20 | | | 67.5 | 45.1 | 88.9 | 41.2 |
| Step T (°C) | -10 | | | 75.3 | 49.2 | 100.3 | 44.5 |
| 10 | 0 | | | 84.8 | 54.2 | 114.0 | 48.6 |
| Min test T (°C) | 10 | | | 96.2 | 60.2 | 130.7 | 53.5 |
| -20 | 20 | | | 110.1 | 67.6 | 150.8 | 59.4 |
| Max test T (°C) | 30 | | | 126.8 | 76.4 | 175.1 | 66.5 |
| 12 | 40 | | | 147.1 | 87.1 | 204.6 | 75.1 |
| Max $K_{Jci}$ (MPa√m) | 50 | | | 171.6 | 100.1 | 240.1 | 85.5 |
| 278.0800802 | 60 | | | 201.2 | 115.7 | 283.2 | 98.1 |
| | 70 | | | 237.1 | 134.6 | 335.2 | 113.4 |
| | 80 | | | 280.4 | 157.5 | 398.1 | 131.8 |
| | 90 | | | 332.8 | 185.2 | 474.2 | 154.1 |
| | 100 | | | 396.2 | 218.6 | 566.2 | 181.1 |
| | 110 | | | 472.8 | 259.1 | 677.4 | 213.7 |
| | 120 | | | 565.4 | 308.0 | 812.0 | 253.1 |
| | 130 | | | 677.5 | 367.1 | 974.7 | 300.8 |


MASTER CURVE FOR INHOMOGENEOUS MATERIALS - Simplified Method
Biblis C base - SE(B) specimens - 30 mm/s
Non-censored data
Censored data
Master curve
5% conf. limit
95% conf. limit
Margin adjusted
1T-normalized $K_{Jc}$ (MPa√m)
Temperature (°C)
$T_{oIN}$
300
250
200
150
100
50
0
-30
-25
-20
-15
-10
-5
0
5
10
15
20

# Appendix G: Master Curve Analysis for Lower Rate Tests on SE(B) 20/40 Specimens of Biblis C Base Material

**Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range**
**ASTM E1921-20**

Analysis for Homogeneous Materials - Multi-Temperature Approach

1. Material characteristics

*Material specifications:* **Biblis C base - SE(B) specimens - 3 mm/s**

| | Fit coefficients $\sigma_{YS}$ (MPa) | Fit coefficients E (GPa) | $\sigma_{YS,RT}$ (MPa) |
|---|---|---|---|
| ($2^{nd}$ order term) A = | -3.13E-03 | 0 | |
| ($1^{st}$ order term) B = | -0.8875 | -0.051 | |
| (Intercept) C = | 494.24992 | 210.5 | |

2. Dimensional and crack growth requirements

Poisson's Ratio = 0.3

| Specimen code | T (°C) | $a_o$ (mm) | W (mm) | B (mm) | $b_o$ (mm) | $\Delta a$ (mm) {Excessive crack growth} | $K_{Jc}$ (MPa√m) {Above $K_{limit}$} | $\sigma_{ys}$ (MPa) | E (GPa) | $K_{Jlim}$ (MPa√m) | Censored? (YES/NO) | $K_{Jc\,analysis}$ (MPa√m) | Test Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 4.1 A13 | -50 | 20.703 | 40 | 20 | 19.30 | 0.000 | 70.87 | 530.81 | 213.05 | 282.74 | NO | 70.87 | 3.11E+02 |
| 3.1 B02 | -45 | 20.809 | 40 | 20 | 19.19 | 0.000 | 55.13 | 527.86 | 212.80 | 281.00 | NO | 55.13 | 2.82E+02 |
| 3.1 B08 | -40 | 20.544 | 40 | 20 | 19.46 | 0.000 | 84.67 | 524.75 | 212.54 | 281.93 | NO | 84.67 | 3.14E+02 |
| 4.2 B03 | -35 | 21.283 | 40 | 20 | 18.72 | 0.000 | 104.37 | 521.48 | 212.29 | 275.50 | NO | 104.37 | 3.34E+02 |
| 3.1 B05 | -30 | 20.663 | 40 | 20 | 19.34 | 0.000 | 134.60 | 518.06 | 212.03 | 278.93 | NO | 134.60 | 3.56E+02 |
| 4.1 B03 | -25 | 21.155 | 40 | 20 | 18.84 | 0.000 | 130.09 | 514.48 | 211.78 | 274.24 | NO | 130.09 | 3.46E+02 |
| 4.1 B09 | -20 | 21.078 | 40 | 20 | 18.92 | 0.000 | 132.59 | 510.75 | 211.52 | 273.64 | NO | 132.59 | 3.59E+02 |
| 3.1 B13 | -15 | 20.482 | 40 | 20 | 19.52 | 0.000 | 142.35 | 506.86 | 211.27 | 276.69 | NO | 142.35 | 3.54E+02 |
| 3.1 B11 | -10 | 20.356 | 40 | 20 | 19.64 | 0.000 | 133.06 | 502.81 | 211.01 | 276.31 | NO | 133.06 | 3.49E+02 |
| 3.1 B07 | -5 | 20.642 | 40 | 20 | 19.36 | 0.000 | 196.09 | 498.61 | 210.76 | 272.97 | NO | 196.09 | 3.46E+02 |
| 4.2 A08 | -40 | 21.197 | 40 | 20 | 18.80 | 0.000 | 84.67 | 524.75 | 212.54 | 277.16 | NO | 84.67 | 3.14E+02 |
| 4.2 A01 | 5 | 21.354 | 40 | 20 | 18.65 | 0.000 | 218.17 | 489.73 | 210.25 | 265.19 | NO | 218.17 | 3.44E+02 |

3. Application of the multi-temperature approach for the calculation of the reference temperature

**USE TEMPERATURE LIMITS?** YES
**($T_o$ - 50 °C ≤ T ≤ $T_o$ + 50 °C)**

T limits (°C)
-81.0
19.0

| Specimen code | T (°C) | $K_{Jc(anal)}$ (MPa√m) | $K_{Jc,1T}$ (MPa√m) | $\delta_i$ | $r_i$ | $n_i$ | $1^{st}$ member | $2^{st}$ member |
|---|---|---|---|---|---|---|---|---|
| 4.1 A13 | -50 | 70.9 | 67.9 | 1 | 1 | 0.143 | 0.0108 | 0.0033 |
| 3.1 B02 | -45 | 55.1 | 53.1 | 1 | 1 | 0.143 | 0.0109 | 0.0005 |
| 3.1 B08 | -40 | 84.7 | 80.9 | 1 | 1 | 0.167 | 0.0111 | 0.0046 |
| 4.2 B03 | -35 | 104.4 | 99.5 | 1 | 1 | 0.167 | 0.0113 | 0.0098 |
| 3.1 B05 | -30 | 134.6 | 127.9 | 1 | 1 | 0.167 | 0.0114 | 0.0241 |
| 4.1 B03 | -25 | 130.1 | 123.7 | 1 | 1 | 0.167 | 0.0115 | 0.0149 |
| 4.1 B09 | -20 | 132.6 | 126.1 | 1 | 1 | 0.167 | 0.0116 | 0.0117 |
| 3.1 B13 | -15 | 142.4 | 135.3 | 1 | 1 | 0.167 | 0.0117 | 0.0117 |
| 3.1 B11 | -10 | 133.1 | 126.5 | 1 | 1 | 0.167 | 0.0119 | 0.0061 |
| 3.1 B07 | -5 | 196.1 | 185.9 | 1 | 1 | 0.167 | 0.0119 | 0.0255 |
| 4.2 A08 | -40 | 84.7 | 80.9 | 1 | 1 | 0.167 | 0.0111 | 0.0046 |
| 4.2 A01 | 5 | 218.2 | 206.7 | 1 | 1 | 0.167 | 0.0121 | 0.0205 |

Sum of 1° member: 0.137

Sum of 2° member: 0.137

Difference: 0.000

**$T_o$ = -31.0 °C**
**(valid per ASTM E1921)**

$\Sigma_i$ $r_i n_i$ = 2.0

# tests = 12
N = 12
r = 12

$K_{min}$ = 20 MPa√m

**$K_{o,eq}$ = 119.9 MPa√m**

**$K_{med,eq}$ = 111.2 MPa√m**

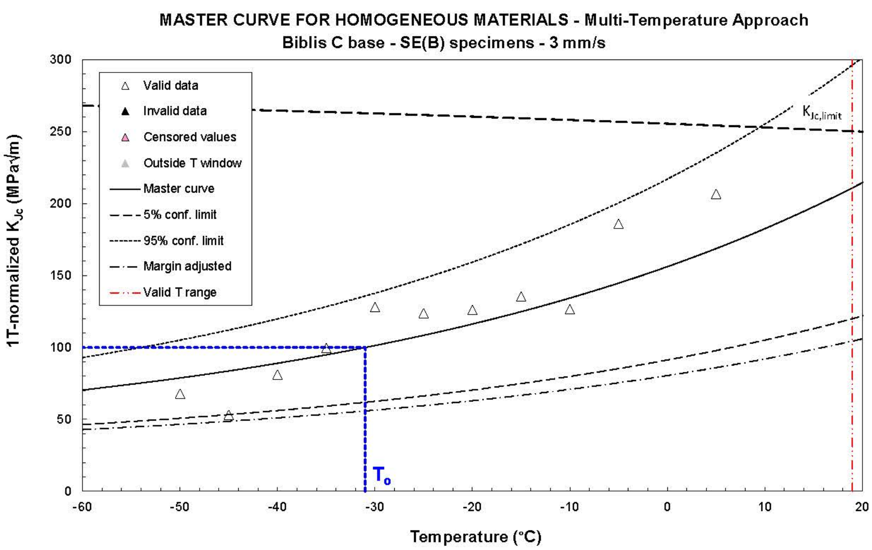
**dK/dt = 3.34E+02 MPa√m/s**

4. Master curve fit to data

Margin adj. (85 % conf.): 9.4 °C (est.)    Stand. dev. on $T_o$ = 6.6 °C (est.)

| T (°C) | $K_{Jc(exp)}$ (MPa√m) | $K_{Jc,1T}$ (MPa√m) | $K_{MC(1T)}$ (MPa√m) | *5% conf.* (MPa√m) | *95% conf.* (MPa√m) | *5% L.B.* (MPa√m) |
|---|---|---|---|---|---|---|
| -50 | 70.9 | 67.9 | | | | |
| -45 | 55.1 | 53.1 | | | | |
| -40 | 84.7 | 80.9 | | | | |
| -35 | 104.4 | 99.5 | | | | |
| -30 | 134.6 | 127.9 | | | | |
| -25 | 130.1 | 123.7 | | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | -20 | 132.6 | 126.1 | | | | |
| | -15 | 142.4 | 135.3 | | | | |
| | -10 | 133.1 | 126.5 | | | | |
| | -5 | 196.1 | 185.9 | | | | |
| | -40 | 84.7 | 80.9 | | | | |
| | 5 | 218.2 | 206.7 | | | | |
| Start T (°C) | -60 | | | 70.3 | 46.4 | 92.9 | 42.9 |
| -60 | -50 | | | 78.8 | 50.8 | 105.1 | 46.6 |
| Step T (°C) | -40 | | | 89.0 | 56.1 | 119.8 | 51.0 |
| 10 | -30 | | | 101.3 | 62.6 | 137.7 | 56.4 |
| Min test T (°C) | -20 | | | 116.3 | 70.4 | 159.3 | 63.0 |
| -50 | -10 | | | 134.3 | 79.8 | 185.4 | 70.9 |
| Max test T (°C) | 0 | | | 156.1 | 91.3 | 217.0 | 80.4 |
| 5 | 10 | | | 182.5 | 105.1 | 255.2 | 92.0 |
| Max $K_{Jc,1T}$ | 20 | | | 214.5 | 121.8 | 301.4 | 105.9 |
| 206.7 | 30 | | | 253.0 | 142.0 | 357.3 | 122.8 |
| | 40 | | | 299.7 | 166.4 | 424.8 | 143.2 |
| | 50 | | | 356.2 | 196.0 | 506.5 | 167.9 |
| | 60 | | | 424.4 | 231.7 | 605.2 | 197.8 |
| | 70 | | | 506.9 | 274.9 | 724.7 | 233.9 |
| | 80 | | | 606.7 | 327.2 | 869.1 | 277.6 |
| | 90 | | | 727.4 | 390.3 | 1043.8 | 330.4 |
| | 100 | | | 873.4 | 466.7 | 1254.9 | 394.2 |




Plot $K_{Jc,limit}$?
YES

**Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range**
**ASTM E1921-20**

Homogeneity Screening Procedure based on SINTAP method

1. Data censoring

| Specimen code | T (°C) | $K_{Jc(exp)}$ (MPa √m) | $K_{Jcl,1T}$ (MPa √m) | $K_{CENS}$ (MPa √m) | $\delta_i$ | $K_{analysis}$ (MPa √m) |
|---|---|---|---|---|---|---|
| 4.1 A13 | -50 | 70.9 | 67.9 | 78.8 | 1 | 67.9 |
| 3.1 B02 | -45 | 55.1 | 53.1 | 83.6 | 1 | 53.1 |
| 3.1 B08 | -40 | 84.7 | 80.9 | 89.0 | 1 | 80.9 |
| 4.2 B03 | -35 | 104.4 | 99.5 | 94.9 | 0 | 94.9 |
| 3.1 B05 | -30 | 134.6 | 127.9 | 101.3 | 0 | 101.3 |
| 4.1 B03 | -25 | 130.1 | 123.7 | 108.4 | 0 | 108.4 |
| 4.1 B09 | -20 | 132.6 | 126.1 | 116.3 | 0 | 116.3 |
| 3.1 B13 | -15 | 142.4 | 135.3 | 124.9 | 0 | 124.9 |
| 3.1 B11 | -10 | 133.1 | 126.5 | 134.3 | 1 | 126.5 |
| 3.1 B07 | -5 | 196.1 | 185.9 | 144.7 | 0 | 144.7 |
| 4.2 A08 | -40 | 84.7 | 80.9 | 89.0 | 1 | 80.9 |
| 4.2 A01 | 5 | 218.2 | 206.7 | 168.7 | 0 | 168.7 |

Benchmark $T_0$ = -31.0 °C

2. Analysis of the censored data and obtainment of a new estimate of $T_0$

USE LIMITS : YES

T limits (°C)
-85.3
14.7

| Specimen code | T (°C) | $K_{analysis}$ (MPa √m) | $\delta_i$ | 1° member | 2° member |
|---|---|---|---|---|---|
| 4.1 A13 | -50 | 67.9 | 1 | 0.0109 | 0.0025 |
| 3.1 B02 | -45 | 53.1 | 1 | 0.0111 | 0.0004 |
| 3.1 B08 | -40 | 80.9 | 1 | 0.0112 | 0.0035 |
| 4.2 B03 | -35 | 94.9 | 0 | 0.0000 | 0.0058 |
| 3.1 B05 | -30 | 101.3 | 0 | 0.0000 | 0.0059 |
| 4.1 B03 | -25 | 108.4 | 0 | 0.0000 | 0.0059 |
| 4.1 B09 | -20 | 116.3 | 0 | 0.0000 | 0.0060 |
| 3.1 B13 | -15 | 124.9 | 0 | 0.0000 | 0.0060 |
| 3.1 B11 | -10 | 126.5 | 1 | 0.0119 | 0.0046 |
| 3.1 B07 | -5 | 144.7 | 0 | 0.0000 | 0.0061 |
| 4.2 A08 | -40 | 80.9 | 1 | 0.0112 | 0.0035 |
| 4.2 A01 | 5 | 168.7 | 0 | 0.0000 | 0.0061 |

*Sum of 1° member:* 0.056

*Sum of 2° member:* 0.056

*Difference :* 0.000

**$T_0$ = -35.3 °C**

**$T_{0(stepn)} \geq T_{0(stepn-1)}$ + 0.5 °C ?**
**NO - Analysis Completed**

**$T_{0scrn}$ = -31.0 °C**

**Screening Criterion**
THE MATERIAL IS
HOMOGENEOUS

# Appendix H: Master Curve Analysis for Tests on SE(B) 20/40 Specimens of Biblis C Weld Material

**Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range**
**ASTM E1921-20**

Analysis for Homogeneous Materials - Multi-Temperature Approach

1. Material characteristics

*Material specifications:* **Biblis C weld - SE(B) 20/40 specimens**

| | Fit coefficients | | |
|---|---|---|---|
| | $\sigma_{YS}$ (MPa) | E (GPa) | $\sigma_{YS,RT}$ (MPa) |
| ($2^{nd}$ order term) A = | 0.00E+00 | 0 | |
| ($1^{st}$ order term) B = | -1.445 | -0.051 | |
| (Intercept) C = | 594.8 | 211.093 | |

2. Dimensional and crack growth requirements

Poisson's Ratio = 0.3

| Specimen code | T (°C) | $a_o$ (mm) | W (mm) | B (mm) | $b_o$ (mm) | $\Delta a$ (mm) {Excessive crack growth} | $K_{Jc}$ (MPa√m) {Above $K_{limit}$} | $\sigma_{ys}$ (MPa) | E (GPa) | $K_{lim}$ (MPa√m) | Censored? (YES/NO) | $K_{Jc analysis}$ (MPa√m) | Test Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| SV2D3 | -75 | 20.882 | 40 | 20 | 19.12 | 0.000 | 94.42 | 703.18 | 214.92 | 325.32 | NO | 94.42 | 3.17E+02 |
| SV2C6 | -65 | 21.043 | 40 | 20 | 18.96 | 0.000 | 90.24 | 688.73 | 214.41 | 320.22 | NO | 90.24 | 3.47E+02 |
| SV2C2 | -60 | 20.905 | 40 | 20 | 19.09 | 0.000 | 123.28 | 681.50 | 214.15 | 319.50 | NO | 123.28 | 3.51E+02 |
| SV2B9 | -55 | 20.935 | 40 | 20 | 19.07 | 0.000 | 255.81 | 674.28 | 213.90 | 317.37 | NO | 255.81 | 3.83E+02 |
| SV2D7 | -54 | 20.877 | 40 | 20 | 19.12 | 0.000 | 127.67 | 672.83 | 213.85 | 317.47 | NO | 127.67 | 3.50E+02 |
| SV2C10 | -50 | 21.162 | 40 | 20 | 18.84 | 0.000 | 105.43 | 667.05 | 213.64 | 313.59 | NO | 105.43 | 3.31E+02 |
| SV2A4 | -50 | 21.089 | 40 | 20 | 18.91 | 0.000 | 197.23 | 667.05 | 213.64 | 314.20 | NO | 197.23 | 3.77E+02 |
| SV2B5 | -45 | 20.884 | 40 | 20 | 19.12 | 0.000 | 202.96 | 659.83 | 213.39 | 313.99 | NO | 202.96 | 3.69E+02 |
| SV2B01 | -45 | 20.923 | 40 | 20 | 19.08 | 0.000 | 243.30 | 659.83 | 213.39 | 313.67 | NO | 243.30 | 3.76E+02 |
| SV2A08 | -40 | 20.655 | 40 | 20 | 19.35 | 0.000 | 300.32 | 652.60 | 213.13 | 313.95 | NO | 300.32 | 3.54E+02 |

3. Application of the multi-temperature approach for the calculation of the reference temperature

| Specimen code | T (°C) | $K_{Jc(anal)}$ (MPa√m) | $K_{Jc,1T}$ (MPa√m) | $\delta_i$ | $r_i$ | $n_i$ | $1^{st}$ member | $2^{st}$ member |
|---|---|---|---|---|---|---|---|---|
| SV2D3 | -75 | 94.4 | 90.1 | 1 | 1 | 0.167 | 0.0117 | 0.0018 |
| SV2C6 | -65 | 90.2 | 86.2 | 1 | 1 | 0.167 | 0.0119 | 0.0007 |
| SV2C2 | -60 | 123.3 | 117.3 | 1 | 1 | 0.167 | 0.0120 | 0.0024 |
| SV2B9 | -55 | 255.8 | 242.1 | 1 | 1 | 0.167 | 0.0121 | 0.0455 |
| SV2D7 | -54 | 127.7 | 121.4 | 1 | 1 | 0.167 | 0.0121 | 0.0018 |
| SV2C10 | -50 | 105.4 | 100.5 | 1 | 1 | 0.167 | 0.0122 | 0.0006 |
| SV2A4 | -50 | 197.2 | 187.0 | 1 | 1 | 0.167 | 0.0122 | 0.0102 |
| SV2B5 | -45 | 203.0 | 192.3 | 1 | 1 | 0.167 | 0.0122 | 0.0082 |
| SV2B01 | -45 | 243.3 | 230.3 | 1 | 1 | 0.167 | 0.0122 | 0.0182 |
| SV2A08 | -40 | 300.3 | 284.1 | 1 | 1 | 0.167 | 0.0123 | 0.0317 |

USE TEMPERATURE LIMITS? YES
($T_o$ - 50 °C ≤ T ≤ $T_o$ + 50 °C)

T limits (°C)
-139.6
-39.6

Sum of 1° member: 0.121

Sum of 2° member: 0.121

Difference: 0.000

**$T_o$ = -89.6 °C**
**(valid per ASTM E1921)**

$\Sigma_i$ $r_i n_i$ = 1.7

# tests = 10
N = 10
r = 10

$K_{min}$ = 20 MPa√m

**$K_{o,eq}$ = 184.6 MPa√m**

**$K_{med,eq}$ = 170.2 MPa√m**

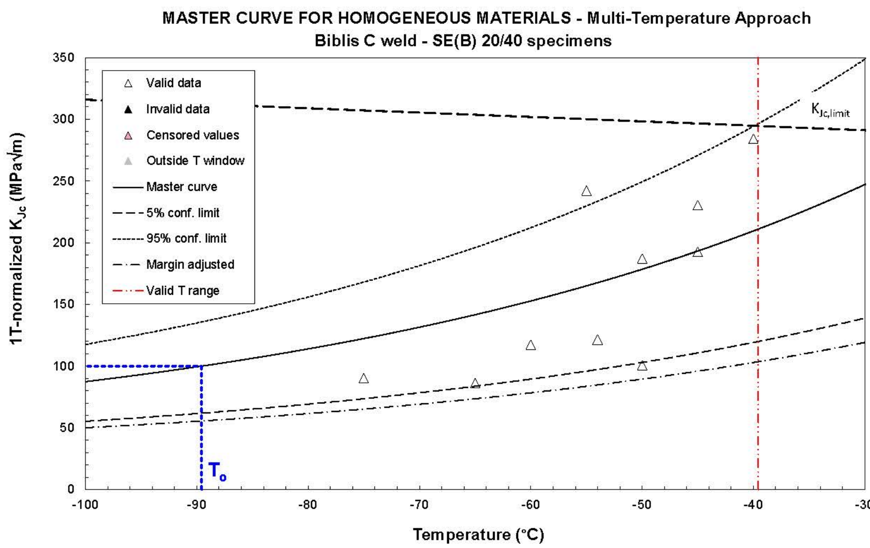
**dK/dt = 3.55E+02 MPa√m/s**

4. Master curve fit to data

Margin adj. (85 % conf.): 10.0 °C (est.) Stand. dev. on $T_o$ = 7.0 °C (est.)

| T (°C) | $K_{Jc(exp)}$ (MPa√m) | $K_{Jc,1T}$ (MPa√m) | $K_{MC(1T)}$ (MPa√m) | *5% conf.* (MPa√m) | *95% conf.* (MPa√m) | *5% L.B.* (MPa√m) |
|---|---|---|---|---|---|---|
| -75 | 94.4 | 90.1 | | | | |
| -65 | 90.2 | 86.2 | | | | |
| -60 | 123.3 | 117.3 | | | | |
| -55 | 255.8 | 242.1 | | | | |
| -54 | 127.7 | 121.4 | | | | |
| -50 | 105.4 | 100.5 | | | | |
| -50 | 197.2 | 187.0 | | | | |
| -45 | 203.0 | 192.3 | | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | -45 | 243.3 | 230.3 | | | | |
| | -40 | 300.3 | 284.1 | | | | |
| Start T (°C) | -100 | | | 87.4 | 55.3 | 117.6 | 50.1 |
| -100 | -95 | | | 93.1 | 58.3 | 125.8 | 52.6 |
| Step T (°C) | -90 | | | 99.4 | 61.6 | 134.9 | 55.3 |
| 5 | -85 | | | 106.3 | 65.2 | 145.0 | 58.3 |
| Min test T (°C) | -80 | | | 114.0 | 69.2 | 156.0 | 61.6 |
| -75 | -75 | | | 122.3 | 73.6 | 168.1 | 65.2 |
| Max test T (°C) | -70 | | | 131.5 | 78.4 | 181.4 | 69.2 |
| -40 | -65 | | | 141.6 | 83.7 | 196.0 | 73.5 |
| Max $K_{Jc,1T}$ | -60 | | | 152.8 | 89.5 | 212.1 | 78.4 |
| 284.1 | -55 | | | 165.0 | 95.9 | 229.8 | 83.7 |
| | -50 | | | 178.5 | 103.0 | 249.3 | 89.5 |
| | -45 | | | 193.3 | 110.7 | 270.7 | 95.9 |
| | -40 | | | 209.5 | 119.2 | 294.3 | 102.9 |
| | -35 | | | 227.4 | 128.6 | 320.2 | 110.7 |
| | -30 | | | 247.1 | 138.9 | 348.6 | 119.2 |
| | -25 | | | 268.7 | 150.2 | 379.9 | 128.5 |
| | -20 | | | 292.5 | 162.7 | 414.4 | 138.8 |




Plot $K_{Jc,limit}$?
YES

**Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range**
**ASTM E1921-20**

Homogeneity Screening Procedure based on SINTAP method

1. Data censoring

| *Specimen code* | *T (°C)* | *$K_{Jc(exp)}$ (MPa √m)* | *$K_{Jcl,1T}$ (MPa √m)* | *$K_{CENS}$ (MPa √m)* | *$\delta_i$* | *$K_{analysis}$ (MPa √m)* |
|---|---|---|---|---|---|---|
| SV2D3 | -75 | 94.4 | 90.1 | 104.1 | 1 | 90.1 |
| SV2C6 | -65 | 90.2 | 86.2 | 119.6 | 1 | 86.2 |
| SV2C2 | -60 | 123.3 | 117.3 | 128.6 | 1 | 117.3 |
| SV2B9 | -55 | 255.8 | 242.1 | 138.4 | 0 | 138.4 |
| SV2D7 | -54 | 127.7 | 121.4 | 140.5 | 1 | 121.4 |
| SV2C10 | -50 | 105.4 | 100.5 | 149.2 | 1 | 100.5 |
| SV2A4 | -50 | 197.2 | 187.0 | 149.2 | 0 | 149.2 |
| SV2B5 | -45 | 203.0 | 192.3 | 161.1 | 0 | 161.1 |
| SV2B01 | -45 | 243.3 | 230.3 | 161.1 | 0 | 161.1 |
| SV2A08 | -40 | 300.3 | 284.1 | 174.1 | 0 | 174.1 |

Benchmark $T_o$ = -78.0 °C

2. Analysis of the censored data and obtainment of a new estimate of $T_o$

USE LIMITS : YES

T limits (°C)
-127.5
-27.5

| *Specimen code* | *T (°C)* | *$K_{analysis}$ (MPa √m)* | *$\delta_i$* | *1° member* | *2° member* |
|---|---|---|---|---|---|
| SV2D3 | -75 | 90.1 | 1 | 0.0114 | 0.0039 |
| SV2C6 | -65 | 86.2 | 1 | 0.0117 | 0.0016 |
| SV2C2 | -60 | 117.3 | 1 | 0.0118 | 0.0054 |
| SV2B9 | -55 | 138.4 | 0 | 0.0000 | 0.0084 |
| SV2D7 | -54 | 121.4 | 1 | 0.0119 | 0.0042 |
| SV2C10 | -50 | 100.5 | 1 | 0.0120 | 0.0013 |
| SV2A4 | -50 | 149.2 | 0 | 0.0000 | 0.0084 |
| SV2B5 | -45 | 161.1 | 0 | 0.0000 | 0.0085 |
| SV2B01 | -45 | 161.1 | 0 | 0.0000 | 0.0085 |
| SV2A08 | -40 | 174.1 | 0 | 0.0000 | 0.0086 |

*Sum of 1° member:* 0.059

*Sum of 2° member:* 0.059

*Difference :* 0.000

**$T_o$ = -77.5 °C**

**$T_{0(stepn)} \geq T_{0(stepn-1)}$ + 0.5 °C ?**
**NO - Analysis Completed**

**$T_{0scrn}$ = -77.5 °C**

**Screening Criterion**
THE MATERIAL IS
INHOMOGENEOUS

**Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range**
**ASTM E1921-20**

Determination of the Reference Temperature for Inhomogeneous Materials (Simplified Method)

1. Calculation of the maximum value of $T_o$ (based on a single data point) and establishment of $T_o$ for the data set

| Specimen id | T (°C) | $K_{Jc[exp]}$ (MPa√m) | $K_{Jci}$ (MPa√m) | $\delta_i$ | $T_{oi}$ (°C) |
|---|---|---|---|---|---|
| SV2D3 | -75 | 94.4 | 90.1 | 1 | -101.0 |
| SV2C6 | -65 | 90.2 | 86.2 | 1 | -87.7 |
| SV2C2 | -60 | 123.3 | 117.3 | 1 | -104.5 |
| SV2B9 | -55 | 255.8 | 242.1 | 1 | -144.7 |
| SV2D7 | -54 | 127.7 | 121.4 | 1 | -100.8 |
| SV2C10 | -50 | 105.4 | 100.5 | 1 | -83.8 |
| SV2A4 | -50 | 197.2 | 187.0 | 1 | -124.2 |
| SV2B5 | -45 | 203.0 | 192.3 | 1 | -121.0 |
| SV2B01 | -45 | 243.3 | 230.3 | 1 | -131.8 |
| SV2A08 | -40 | 300.3 | 284.1 | 1 | -139.0 |

$T_{omax}$ = -83.8 °C

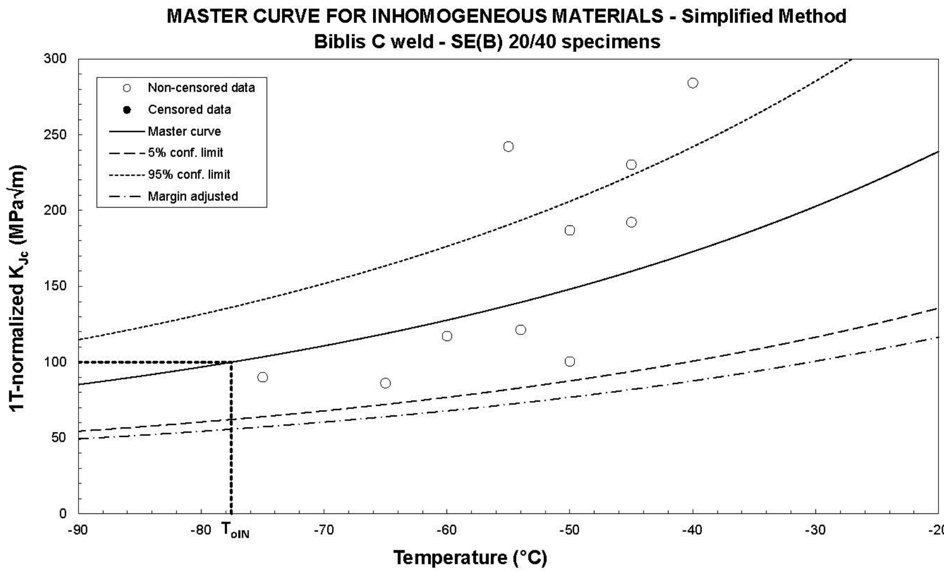
$T_{oscrn}$ = -77.5 °C

$T_{omax} - T_{oscrn} > 8$ °C : **NO**
Number of tests N = 10
**$T_{oIN}$ = -77.5 °C**

2. Final Master Curve fit to data

Margin adj. (85 % conf.): 10.0 °C (est.) Stand. dev. on $T_o$ = 7.0 °C (est.)

| | T (°C) | $K_{Jc(exp)}$ (MPa √m) | $K_{Jc(1T)}$ (MPa √m) | $K_{MC(1T)}$ (MPa √m) | 5% conf. (MPa √m) | 95% conf. (MPa √m) | 5% L.B. (MPa √m) |
|---|---|---|---|---|---|---|---|
| | -75.0 | 94.4 | 90.1 | | | | |
| | -65.0 | 90.2 | 86.2 | | | | |
| | -60.0 | 123.3 | 117.3 | | | | |
| | -55.0 | 255.8 | 242.1 | | | | |
| | -54.0 | 127.7 | 121.4 | | | | |
| | -50.0 | 105.4 | 100.5 | | | | |
| | -50.0 | 197.2 | 187.0 | | | | |
| | -45.0 | 203.0 | 192.3 | | | | |
| | -45.0 | 243.3 | 230.3 | | | | |
| | -40.0 | 300.3 | 284.1 | | | | |
| Start T (°C) | -100 | | | 75.7 | 49.4 | 100.9 | 45.2 |
| -100 | -95 | | | 80.2 | 51.8 | 107.5 | 47.2 |
| Step T (°C) | -90 | | | 85.3 | 54.5 | 114.7 | 49.4 |
| 5 | -85 | | | 90.8 | 57.4 | 122.7 | 51.8 |
| Min test T (°C) | -80 | | | 96.8 | 60.6 | 131.5 | 54.4 |
| -75 | -75 | | | 103.5 | 64.1 | 141.2 | 57.3 |
| Max test T (°C) | -70 | | | 110.8 | 67.9 | 151.8 | 60.5 |
| -40 | -65 | | | 118.8 | 72.2 | 163.5 | 64.1 |
| Max $K_{Jci}$ (MPa√m) | -60 | | | 127.7 | 76.9 | 176.4 | 67.9 |
| 300.3179701 | -55 | | | 137.4 | 82.0 | 190.5 | 72.2 |
| | -50 | | | 148.1 | 87.7 | 206.1 | 76.8 |
| | -45 | | | 159.9 | 93.9 | 223.2 | 82.0 |
| | -40 | | | 172.9 | 100.7 | 242.0 | 87.6 |
| | -35 | | | 187.1 | 108.2 | 262.6 | 93.9 |
| | -30 | | | 202.8 | 116.5 | 285.4 | 100.7 |
| | -25 | | | 220.0 | 125.6 | 310.4 | 108.2 |
| | -20 | | | 238.9 | 135.6 | 337.9 | 116.5 |



MASTER CURVE FOR INHOMOGENEOUS MATERIALS - Simplified Method
Biblis C weld - SE(B) 20/40 specimens
Non-censored data
Censored data
Master curve
5% conf. limit
95% conf. limit
Margin adjusted
1T-normalized K_Jc (MPa√m)
Temperature (°C)
T_oIN

# Appendix I: Master Curve Analysis for Tests on 1TC(T) Specimens of S590QL Steel

**Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range**
**ASTM E1921-20**

Analysis for Homogeneous Materials - Multi-Temperature Approach

1. Material characteristics

Material specifications: **S690QL - 1TC(T) specimens - 2.5 mm/s**

| | Fit coefficients $\sigma_{YS}$ (MPa) | Fit coefficients E (GPa) | $\sigma_{YS,RT}$ (MPa) |
|---|---|---|---|
| (2[nd] order term) A = | -3.13E-03 | 0 | |
| (1[st] order term) B = | -0.8875 | -0.051 | |
| (Intercept) C = | 494.24992 | 210.5 | |

2. Dimensional and crack growth requirements

Poisson's Ratio = 0.3

| Specimen code | T (°C) | $a_o$ (mm) | W (mm) | B (mm) | $b_o$ (mm) | {Excessive crack growth} $\Delta a$ (mm) | {Above $K_{limit}$} $K_{Jc}$ (MPa√m) | $\sigma_{ys}$ (MPa) | E (GPa) | $K_{lim}$ (MPa√m) | Censored? (YES/NO) | $K_{Jc analysis}$ (MPa√m) | Test Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| BB09 | -50 | 25.802 | 50 | 25 | 24.20 | 0.000 | 146.17 | 530.81 | 213.05 | 316.61 | NO | 146.17 | 1.46E+02 |
| AB12 | -45 | 25.573 | 50 | 25 | 24.43 | 0.000 | 107.47 | 527.86 | 212.80 | 317.02 | NO | 107.47 | 1.07E+02 |
| AA03 | -45 | 25.353 | 50 | 25 | 24.65 | 0.000 | 63.20 | 527.86 | 212.80 | 318.45 | NO | 63.20 | 6.32E+01 |
| BD06 | -40 | 25.689 | 50 | 25 | 24.31 | 0.000 | 93.32 | 524.75 | 212.54 | 315.15 | NO | 93.32 | 9.33E+01 |
| AC09 | -40 | 25.708 | 50 | 25 | 24.29 | 0.000 | 112.97 | 524.75 | 212.54 | 315.03 | NO | 112.97 | 1.13E+02 |
| AB06 | -35 | 25.019 | 50 | 25 | 24.98 | 0.000 | 63.83 | 521.48 | 212.29 | 318.28 | NO | 63.83 | 6.38E+01 |
| BC03 | -32 | 26.276 | 50 | 25 | 23.72 | 0.000 | 114.82 | 519.45 | 212.13 | 309.45 | NO | 114.82 | 1.15E+02 |
| BE03 | -30 | 25.523 | 50 | 25 | 24.48 | 0.000 | 84.55 | 518.06 | 212.03 | 313.82 | NO | 84.55 | 8.45E+01 |
| BC12 | -27 | 25.799 | 50 | 25 | 24.20 | 0.000 | 137.27 | 515.93 | 211.88 | 311.30 | NO | 137.27 | 1.37E+02 |
| AE06 | -25 | 25.478 | 50 | 25 | 24.52 | 0.000 | 116.05 | 514.48 | 211.78 | 312.84 | NO | 116.05 | 1.16E+02 |
| AD03 | -20 | 26.470 | 50 | 25 | 23.53 | 0.000 | 178.07 | 510.75 | 211.52 | 305.15 | NO | 178.07 | 1.78E+02 |
| BA06 | -20 | 25.622 | 50 | 25 | 24.38 | 0.000 | 191.32 | 510.75 | 211.52 | 310.60 | NO | 191.32 | 1.91E+02 |
| BA12 | -15 | 26.297 | 50 | 25 | 23.70 | 0.000 | 187.75 | 506.86 | 211.27 | 304.91 | NO | 187.75 | 1.88E+02 |
| AD12 | -10 | 25.489 | 50 | 25 | 24.51 | 0.000 | 68.57 | 502.81 | 211.01 | 308.64 | NO | 68.57 | 6.86E+01 |

3. Application of the multi-temperature approach for the calculation of the reference temperature

| Specimen code | T (°C) | $K_{Jc(anal)}$ (MPa√m) | $K_{Jc,1T}$ (MPa√m) | $\delta_i$ | $r_i$ | $n_i$ | 1[st] member | 2[st] member |
|---|---|---|---|---|---|---|---|---|
| BB09 | -50 | 146.2 | 145.7 | 1 | 1 | 0.167 | 0.0113 | 0.0575 |
| AB12 | -45 | 107.5 | 107.1 | 1 | 1 | 0.167 | 0.0114 | 0.0096 |
| AA03 | -45 | 63.2 | 63.0 | 1 | 1 | 0.167 | 0.0114 | 0.0006 |
| BD06 | -40 | 93.3 | 93.0 | 1 | 1 | 0.167 | 0.0115 | 0.0034 |
| AC09 | -40 | 113.0 | 112.6 | 1 | 1 | 0.167 | 0.0115 | 0.0089 |
| AB06 | -35 | 63.8 | 63.7 | 1 | 1 | 0.167 | 0.0117 | 0.0003 |
| BC03 | -32 | 114.8 | 114.4 | 1 | 1 | 0.167 | 0.0117 | 0.0057 |
| BE03 | -30 | 84.5 | 84.3 | 1 | 1 | 0.167 | 0.0118 | 0.0011 |
| BC12 | -27 | 137.3 | 136.8 | 1 | 1 | 0.167 | 0.0118 | 0.0095 |
| AE06 | -25 | 116.1 | 115.7 | 1 | 1 | 0.167 | 0.0119 | 0.0037 |
| AD03 | -20 | 178.1 | 177.4 | 1 | 1 | 0.167 | 0.0120 | 0.0194 |
| BA06 | -20 | 191.3 | 190.6 | 1 | 1 | 0.167 | 0.0120 | 0.0268 |
| BA12 | -15 | 187.8 | 187.1 | 1 | 1 | 0.167 | 0.0120 | 0.0175 |
| AD12 | -10 | 68.6 | 68.4 | 1 | 1 | 0.167 | 0.0121 | 0.0001 |

**USE TEMPERATURE LIMITS?** YES
**($T_o$ - 50 °C ≤ T ≤ $T_o$ + 50 °C)**

T limits (°C)
-96.9
3.1

Sum of 1° member: 0.164

Sum of 2° member: 0.164

Difference: 0.000

**$T_o$ = -46.9 °C**
**(valid per ASTM E1921)**

$\Sigma_i$ $r_i n_i$ = 2.3

# tests = 14
N = 14
r = 14

$K_{min}$ = 20 MPa√m

**$K_{o,eq}$ = 137.4 MPa√m**

**$K_{med,eq}$ = 127.1 MPa√m**

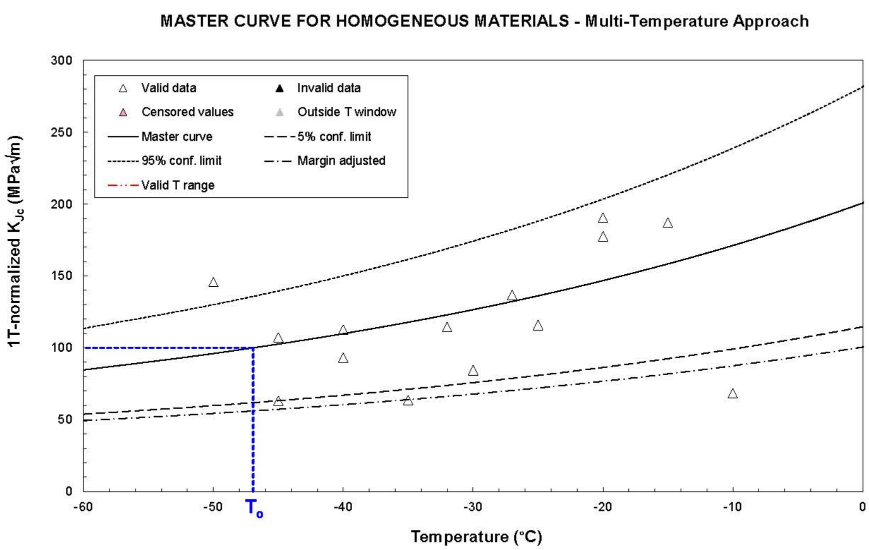
**dK/dt = 1.19E+02 MPa√m/s**

4. Master curve fit to data

Margin adj. (85 % conf.): 9.0 °C (est.) Stand. dev. on $T_o$ = 6.3 °C (est.)

| T (°C) | $K_{Jc(exp)}$ (MPa√m) | $K_{Jc,1T}$ (MPa√m) | $K_{MC(1T)}$ (MPa√m) | 5% conf. (MPa√m) | 95% conf. (MPa√m) | 5% L.B. (MPa√m) |
|---|---|---|---|---|---|---|
| -50 | 146.2 | 145.7 | | | | |
| -45 | 107.5 | 107.1 | | | | |
| -45 | 63.2 | 63.0 | | | | |
| -40 | 93.3 | 93.0 | | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | -40 | 113.0 | 112.6 | | | | |
| | -35 | 63.8 | 63.7 | | | | |
| | -32 | 114.8 | 114.4 | | | | |
| | -30 | 84.5 | 84.3 | | | | |
| | -27 | 137.3 | 136.8 | | | | |
| | -25 | 116.1 | 115.7 | | | | |
| | -20 | 178.1 | 177.4 | | | | |
| | -20 | 191.3 | 190.6 | | | | |
| | -15 | 187.8 | 187.1 | | | | |
| | -10 | 68.6 | 68.4 | | | | |
| Start T (°C) | -60 | | | 84.6 | 53.8 | 113.5 | 49.3 |
| -60 | -50 | | | 96.0 | 59.8 | 130.0 | 54.4 |
| Step T (°C) | -40 | | | 109.9 | 67.0 | 150.0 | 60.5 |
| 10 | -30 | | | 126.6 | 75.8 | 174.2 | 67.8 |
| Min test T (°C) | -20 | | | 146.8 | 86.4 | 203.5 | 76.7 |
| -50 | -10 | | | 171.2 | 99.2 | 238.8 | 87.5 |
| Max test T (°C) | 0 | | | 200.7 | 114.6 | 281.6 | 100.6 |
| -10 | 10 | | | 236.5 | 133.3 | 333.3 | 116.3 |
| Max $K_{Jc,1T}$ | 20 | | | 279.7 | 155.9 | 395.8 | 135.4 |
| 190.6 | 30 | | | 331.9 | 183.3 | 471.4 | 158.4 |
| | 40 | | | 395.1 | 216.4 | 562.8 | 186.3 |
| | 50 | | | 471.5 | 256.4 | 673.4 | 220.0 |
| | 60 | | | 563.9 | 304.7 | 807.1 | 260.7 |
| | 70 | | | 675.6 | 363.2 | 968.8 | 310.0 |
| | 80 | | | 810.7 | 433.9 | 1164.3 | 369.6 |
| | 90 | | | 974.0 | 519.4 | 1400.7 | 441.7 |
| | 100 | | | 1171.6 | 622.8 | 1686.5 | 528.8 |




Plot $K_{Jc,limit}$?

NO

**Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range**
**ASTM E1921-20**

Homogeneity Screening Procedure based on SINTAP method

1. Data censoring

| Specimen code | T (°C) | $K_{Jc(exp)}$ (MPa √m) | $K_{Jc,1T}$ (MPa √m) | $K_{CENS}$ (MPa √m) | $\delta_i$ | $K_{analysis}$ (MPa √m) |
|---|---|---|---|---|---|---|
| BB09 | -50 | 146.2 | 145.7 | 90.0 | 0 | 90.0 |
| AB12 | -45 | 107.5 | 107.1 | 95.9 | 0 | 95.9 |
| AA03 | -45 | 63.2 | 63.0 | 95.9 | 1 | 63.0 |
| BD06 | -40 | 93.3 | 93.0 | 102.5 | 1 | 93.0 |
| AC09 | -40 | 113.0 | 112.6 | 102.5 | 0 | 102.5 |
| AB06 | -35 | 63.8 | 63.7 | 109.7 | 1 | 63.7 |

Benchmark $T_o$ = -41.9 °C

2. Analysis of the censored data and obtainment of a new estimate of $T_o$

T limits (°C)
USE LIMITS : YES -93.2
6.8

| Specimen code | T (°C) | $K_{analysis}$ (MPa √m) | $\delta_i$ | 1° member | 2° member |
|---|---|---|---|---|---|
| BB09 | -50 | 90.0 | 0 | 0.0000 | 0.0070 |
| AB12 | -45 | 95.9 | 0 | 0.0000 | 0.0071 |
| AA03 | -45 | 63.0 | 1 | 0.0113 | 0.0007 |
| BD06 | -40 | 93.0 | 1 | 0.0114 | 0.0044 |
| AC09 | -40 | 102.5 | 0 | 0.0000 | 0.0072 |
| AB06 | -35 | 63.7 | 1 | 0.0116 | 0.0004 |
| BC03 | -32 | 114.4 | 0 | 0.0000 | 0.0073 |
| BE03 | -30 | 84.3 | 1 | 0.0117 | 0.0014 |
| BC12 | -27 | 122.8 | 0 | 0.0000 | 0.0073 |
| AE06 | -25 | 115.7 | 1 | 0.0118 | 0.0048 |
| AD03 | -20 | 136.0 | 0 | 0.0000 | 0.0074 |
| BA06 | -20 | 136.0 | 0 | 0.0000 | 0.0074 |
| BA12 | -15 | 146.6 | 0 | 0.0000 | 0.0075 |
| AD12 | -10 | 68.4 | 1 | 0.0121 | 0.0001 |

*Sum of 1° member:* 0.070

*Sum of 2° member:* 0.070

*Difference :* 0.000

**$T_o$ = -43.2 °C**

**$T_{0(stepn)} \geq T_{0(stepn-1)}$ + 0.5 °C ?**
**NO - Analysis Completed**

**$T_{0scrn}$ = -41.9 °C**

**Screening Criterion**
THE MATERIAL IS
HOMOGENEOUS

# Appendix J: Master Curve Analysis for Tests PCCv Specimens of Biblis C Base Material

**Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range**
**ASTM E1921-20**

Analysis for Homogeneous Materials - Multi-Temperature Approach

1. Material characteristics

*Material specifications:* **Biblis C base - PCCv specimens**

| | Fit coefficients | | |
|---|---|---|---|
| | $\sigma_{YS}$ (MPa) | E (GPa) | $\sigma_{YS,RT}$ (MPa) |
| (2nd order term) A = | -3.13E-03 | 0 | |
| (1st order term) B = | -0.8875 | -0.051 | |
| (Intercept) C = | 494.24992 | 210.5 | |

2. Dimensional and crack growth requirements

Poisson's Ratio = 0.3

| Specimen code | T (°C) | $a_o$ (mm) | W (mm) | B (mm) | $b_o$ (mm) | $\Delta a$ (mm) {Excessive crack growth} | $K_{Jc}$ (MPa√m) {Above $K_{limit}$} | $\sigma_{ys}$ (MPa) | E (GPa) | $K_{lim}$ (MPa√m) | Censored? (YES/NO) | $K_{Jc analysis}$ (MPa√m) | Test Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| B02-CV5 | -65 | 5.264 | 10 | 10 | 4.74 | 0.000 | 62.53 | 538.73 | 213.82 | 141.36 | NO | 62.53 | 7.28E+01 |
| B02-CV2 | -55 | 5.282 | 10 | 10 | 4.72 | 0.000 | 61.80 | 533.61 | 213.31 | 140.25 | NO | 61.80 | 7.88E+01 |
| A08-CV5 | -50 | 5.144 | 10 | 10 | 4.86 | 0.000 | 81.61 | 530.81 | 213.05 | 141.83 | NO | 81.61 | 9.83E+01 |
| B02-CV4 | -46 | 5.272 | 10 | 10 | 4.73 | 0.000 | 74.38 | 528.46 | 212.85 | 139.57 | NO | 74.38 | 8.93E+01 |
| A08-CV3 | -45 | 5.289 | 10 | 10 | 4.71 | 0.000 | 68.31 | 527.86 | 212.80 | 139.23 | NO | 68.31 | 9.22E+01 |
| A08-CV1 | -40 | 5.215 | 10 | 10 | 4.78 | 0.000 | 109.82 | 524.75 | 212.54 | 139.81 | NO | 109.82 | 1.20E+02 |
| A08-CV4 | -40 | 5.094 | 10 | 10 | 4.91 | 0.000 | 102.09 | 524.75 | 212.54 | 141.57 | NO | 102.09 | 1.17E+02 |
| A08-CV2 | -35 | 5.199 | 10 | 10 | 4.80 | 0.000 | 94.10 | 521.48 | 212.29 | 139.53 | NO | 94.10 | 1.08E+02 |
| B02-CV1 | -30 | 5.231 | 10 | 10 | 4.77 | 0.000 | 120.88 | 518.06 | 212.03 | 138.53 | NO | 120.88 | 1.33E+02 |
| B02-CV3 | -25 | 5.177 | 10 | 10 | 4.82 | 0.000 | 123.79 | 514.48 | 211.78 | 138.73 | NO | 123.79 | 1.36E+02 |

3. Application of the multi-temperature approach for the calculation of the reference temperature

| Specimen code | T (°C) | $K_{Jc(anal)}$ (MPa√m) | $K_{Jc,1T}$ (MPa√m) | $\delta_i$ | $r_i$ | $n_i$ | 1st member | 2nd member |
|---|---|---|---|---|---|---|---|---|
| B02-CV5 | -65 | 62.5 | 53.7 | 1 | 1 | 0.125 | 0.0095 | 0.0044 |
| B02-CV2 | -55 | 61.8 | 53.1 | 1 | 1 | 0.125 | 0.0100 | 0.0024 |
| A08-CV5 | -50 | 81.6 | 68.8 | 1 | 1 | 0.143 | 0.0102 | 0.0088 |
| B02-CV4 | -46 | 74.4 | 63.1 | 1 | 1 | 0.143 | 0.0103 | 0.0043 |
| A08-CV3 | -45 | 68.3 | 58.3 | 1 | 1 | 0.143 | 0.0104 | 0.0025 |
| A08-CV1 | -40 | 109.8 | 91.2 | 1 | 1 | 0.143 | 0.0106 | 0.0225 |
| A08-CV4 | -40 | 102.1 | 85.0 | 1 | 1 | 0.143 | 0.0106 | 0.0157 |
| A08-CV2 | -35 | 94.1 | 78.7 | 1 | 1 | 0.143 | 0.0107 | 0.0077 |
| B02-CV1 | -30 | 120.9 | 99.9 | 1 | 1 | 0.143 | 0.0109 | 0.0197 |
| B02-CV3 | -25 | 123.8 | 102.2 | 1 | 1 | 0.167 | 0.0111 | 0.0162 |

**USE TEMPERATURE LIMITS?** YES
**($T_o$ - 50 °C ≤ T ≤ $T_o$ + 50 °C)**

T limits (°C)
-65.0
35.0

Sum of 1° member: 0.104

Sum of 2° member: 0.104

Difference: 0.000

**$T_o$ = -15.0 °C**
**(valid per ASTM E1921)**

$\Sigma_i$ $r_i n_i$ = 1.4

# tests = 10
N = 10
r = 10

$K_{min}$ = 20 MPa√m

**$K_{o,eq}$ = 77.0 MPa√m**

**$K_{med,eq}$ = 72.0 MPa√m**

**dK/dt = 1.05E+02 MPa√m/s**

4. Master curve fit to data

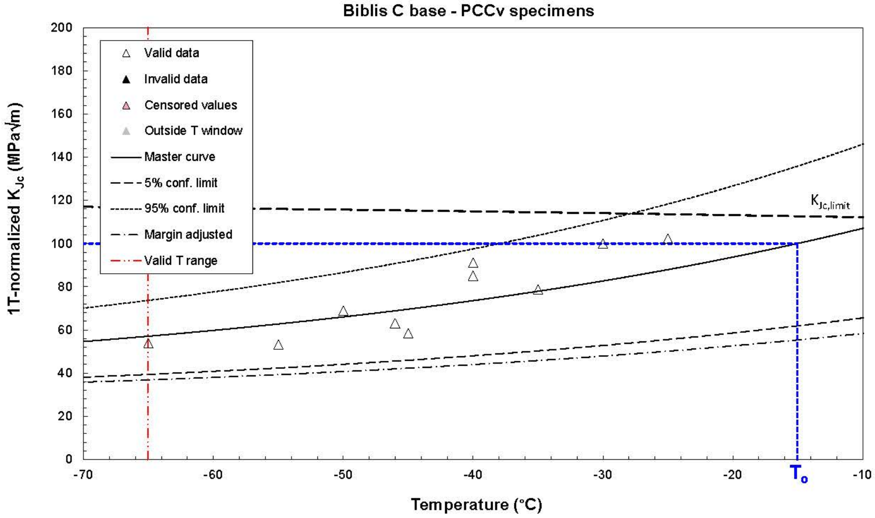
Margin adj. (85 % conf.): 10.3 °C (est.) Stand. dev. on $T_o$ = 7.2 °C (est.)

| T (°C) | $K_{Jc(exp)}$ (MPa√m) | $K_{Jc,1T}$ (MPa√m) | $K_{MC(1T)}$ (MPa√m) | 5% conf. (MPa√m) | 95% conf. (MPa√m) | 5% L.B. (MPa√m) |
|---|---|---|---|---|---|---|
| -65 | 62.5 | 53.7 | | | | |
| -55 | 61.8 | 53.1 | | | | |
| -50 | 81.6 | 68.8 | | | | |
| -46 | 74.4 | 63.1 | | | | |
| -45 | 68.3 | 58.3 | | | | |
| -40 | 109.8 | 91.2 | | | | |
| -40 | 102.1 | 85.0 | | | | |
| -35 | 94.1 | 78.7 | | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | -30 | 120.9 | 99.9 | | | | |
| | -25 | 123.8 | 102.2 | | | | |
| Start T (°C) | -90 | | | 46.8 | 34.1 | 58.9 | 32.5 |
| -90 | -80 | | | 50.4 | 35.9 | 64.0 | 34.0 |
| Step T (°C) | -70 | | | 54.6 | 38.1 | 70.1 | 35.8 |
| 10 | -60 | | | 59.8 | 40.8 | 77.6 | 38.1 |
| Min test T (°C) | -50 | | | 66.0 | 44.1 | 86.6 | 40.7 |
| -65 | -40 | | | 73.6 | 48.0 | 97.5 | 44.0 |
| Max test T (°C) | -30 | | | 82.7 | 52.8 | 110.7 | 47.9 |
| -25 | -20 | | | 93.7 | 58.6 | 126.7 | 52.6 |
| Max $K_{Jc,1T}$ | -10 | | | 107.0 | 65.6 | 146.0 | 58.4 |
| 102.2 | 0 | | | 123.2 | 74.0 | 169.3 | 65.3 |
| | 10 | | | 142.7 | 84.2 | 197.5 | 73.7 |
| | 20 | | | 166.2 | 96.5 | 231.6 | 83.9 |
| | 30 | | | 194.7 | 111.5 | 272.9 | 96.1 |
| | 40 | | | 229.2 | 129.5 | 322.8 | 111.0 |
| | 50 | | | 270.9 | 151.3 | 383.1 | 128.9 |
| | 60 | | | 321.3 | 177.7 | 456.0 | 150.6 |
| | 70 | | | 382.2 | 209.6 | 544.2 | 176.8 |

**MASTER CURVE FOR HOMOGENEOUS MATERIALS - Multi-Temperature Approach**
**Biblis C base - PCCv specimens**

Plot $K_{Jc,limit}$?
YES



**Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range**
**ASTM E1921-20**

Homogeneity Screening Procedure based on SINTAP method

1. Data censoring

| Specimen code | T (°C) | $K_{Jc(exp)}$ (MPa √m) | $K_{Jcl,1T}$ (MPa √m) | $K_{CENS}$ (MPa √m) | $\delta_i$ | $K_{analysis}$ (MPa √m) |
|---|---|---|---|---|---|---|
| B02-CV5 | -65 | 62.5 | 53.7 | 57.1 | 1 | 53.7 |
| B02-CV2 | -55 | 61.8 | 53.1 | 62.8 | 1 | 53.1 |
| A08-CV5 | -50 | 81.6 | 68.8 | 66.0 | 0 | 66.0 |
| B02-CV4 | -46 | 74.4 | 63.1 | 68.9 | 1 | 63.1 |
| A08-CV3 | -45 | 68.3 | 58.3 | 69.6 | 1 | 58.3 |
| A08-CV1 | -40 | 109.8 | 91.2 | 73.6 | 0 | 73.6 |
| A08-CV4 | -40 | 102.1 | 85.0 | 73.6 | 0 | 73.6 |
| A08-CV2 | -35 | 94.1 | 78.7 | 77.9 | 0 | 77.9 |
| B02-CV1 | -30 | 120.9 | 99.9 | 82.7 | 0 | 82.7 |
| B02-CV3 | -25 | 123.8 | 102.2 | 87.9 | 0 | 87.9 |

Benchmark $T_o$ = -15.0 °C

2. Analysis of the censored data and obtainment of a new estimate of $T_o$

USE LIMITS : YES

T limits (°C)
-70.7
29.3

| Specimen code | T (°C) | $K_{analysis}$ (MPa √m) | $\delta_i$ | 1° member | 2° member |
|---|---|---|---|---|---|
| B02-CV5 | -65 | 53.7 | 1 | 0.0098 | 0.0033 |
| B02-CV2 | -55 | 53.1 | 1 | 0.0102 | 0.0018 |
| A08-CV5 | -50 | 66.0 | 0 | 0.0000 | 0.0050 |
| B02-CV4 | -46 | 63.1 | 1 | 0.0106 | 0.0031 |
| A08-CV3 | -45 | 58.3 | 1 | 0.0106 | 0.0018 |
| A08-CV1 | -40 | 73.6 | 0 | 0.0000 | 0.0052 |
| A08-CV4 | -40 | 73.6 | 0 | 0.0000 | 0.0052 |
| A08-CV2 | -35 | 77.9 | 0 | 0.0000 | 0.0052 |
| B02-CV1 | -30 | 82.7 | 0 | 0.0000 | 0.0053 |
| B02-CV3 | -25 | 87.9 | 0 | 0.0000 | 0.0053 |

*Sum of 1° member:* 0.041

*Sum of 2° member:* 0.041

*Difference :* 0.000

$T_o$ = -20.7 °C

$T_{0(stepn)} \geq T_{0(stepn-1)} + 0.5$ °C ?
NO - Analysis Completed

$T_{0scrn}$ = -15.0 °C

**Screening Criterion**
THE MATERIAL IS
HOMOGENEOUS

# Appendix K: Master Curve Analysis for Tests PCCv Specimens of Biblis C Weld Material

**Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range**
**ASTM E1921-20**

Analysis for Homogeneous Materials - Multi-Temperature Approach

1. Material characteristics

*Material specifications:* **Biblis C base - PCCv specimens**

| Fit coefficients | $\sigma_{YS}$ (MPa) | E (GPa) | $\sigma_{YS,RT}$ (MPa) |
|---|---|---|---|
| (2nd order term) A = | -3.13E-03 | 0 | |
| (1st order term) B = | -0.8875 | -0.051 | |
| (Intercept) C = | 494.24992 | 210.5 | |

2. Dimensional and crack growth requirements

Poisson's Ratio = 0.3

| Specimen code | T (°C) | $a_o$ (mm) | W (mm) | B (mm) | $b_o$ (mm) | $\Delta a$ (mm) {Excessive crack growth} | $K_{Jc}$ (MPa√m) {Above $K_{limit}$} | $\sigma_{ys}$ (MPa) | E (GPa) | $K_{lim}$ (MPa√m) | Censored? (YES/NO) | $K_{Jc analysis}$ (MPa√m) | Test Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| B02-CV5 | -65 | 5.264 | 10 | 10 | 4.74 | 0.000 | 62.53 | 538.73 | 213.82 | 141.36 | NO | 62.53 | 7.28E+01 |
| B02-CV2 | -55 | 5.282 | 10 | 10 | 4.72 | 0.000 | 61.80 | 533.61 | 213.31 | 140.25 | NO | 61.80 | 7.88E+01 |
| A08-CV5 | -50 | 5.144 | 10 | 10 | 4.86 | 0.000 | 81.61 | 530.81 | 213.05 | 141.83 | NO | 81.61 | 9.83E+01 |
| B02-CV4 | -46 | 5.272 | 10 | 10 | 4.73 | 0.000 | 74.38 | 528.46 | 212.85 | 139.57 | NO | 74.38 | 8.93E+01 |
| A08-CV3 | -45 | 5.289 | 10 | 10 | 4.71 | 0.000 | 68.31 | 527.86 | 212.80 | 139.23 | NO | 68.31 | 9.22E+01 |
| A08-CV1 | -40 | 5.215 | 10 | 10 | 4.78 | 0.000 | 109.82 | 524.75 | 212.54 | 139.81 | NO | 109.82 | 1.20E+02 |
| A08-CV4 | -40 | 5.094 | 10 | 10 | 4.91 | 0.000 | 102.09 | 524.75 | 212.54 | 141.57 | NO | 102.09 | 1.17E+02 |
| A08-CV2 | -35 | 5.199 | 10 | 10 | 4.80 | 0.000 | 94.10 | 521.48 | 212.29 | 139.53 | NO | 94.10 | 1.08E+02 |
| B02-CV1 | -30 | 5.231 | 10 | 10 | 4.77 | 0.000 | 120.88 | 518.06 | 212.03 | 138.53 | NO | 120.88 | 1.33E+02 |
| B02-CV3 | -25 | 5.177 | 10 | 10 | 4.82 | 0.000 | 123.79 | 514.48 | 211.78 | 138.73 | NO | 123.79 | 1.36E+02 |

3. Application of the multi-temperature approach for the calculation of the reference temperature

| Specimen code | T (°C) | $K_{Jc(anal)}$ (MPa√m) | $K_{Jc,1T}$ (MPa√m) | $\delta_i$ | $r_i$ | $n_i$ | 1st member | 2st member |
|---|---|---|---|---|---|---|---|---|
| B02-CV5 | -65 | 62.5 | 53.7 | 1 | 1 | 0.125 | 0.0095 | 0.0044 |
| B02-CV2 | -55 | 61.8 | 53.1 | 1 | 1 | 0.125 | 0.0100 | 0.0024 |
| A08-CV5 | -50 | 81.6 | 68.8 | 1 | 1 | 0.143 | 0.0102 | 0.0088 |
| B02-CV4 | -46 | 74.4 | 63.1 | 1 | 1 | 0.143 | 0.0103 | 0.0043 |
| A08-CV3 | -45 | 68.3 | 58.3 | 1 | 1 | 0.143 | 0.0104 | 0.0025 |
| A08-CV1 | -40 | 109.8 | 91.2 | 1 | 1 | 0.143 | 0.0106 | 0.0225 |
| A08-CV4 | -40 | 102.1 | 85.0 | 1 | 1 | 0.143 | 0.0106 | 0.0157 |
| A08-CV2 | -35 | 94.1 | 78.7 | 1 | 1 | 0.143 | 0.0107 | 0.0077 |
| B02-CV1 | -30 | 120.9 | 99.9 | 1 | 1 | 0.143 | 0.0109 | 0.0197 |
| B02-CV3 | -25 | 123.8 | 102.2 | 1 | 1 | 0.167 | 0.0111 | 0.0162 |

**USE TEMPERATURE LIMITS?** YES
**($T_o$ - 50 °C ≤ T ≤ $T_o$ + 50 °C)**

T limits (°C)
-65.0
35.0

Sum of 1° member: 0.104

Sum of 2° member: 0.104

Difference: 0.000

**$T_o$ = -15.0 °C**
**(valid per ASTM E1921)**

$\Sigma_i$ $r_i n_i$ = 1.4

# tests = 10
N = 10
r = 10

$K_{min}$ = 20 MPa√m

**$K_{o,eq}$ = 77.0 MPa√m**

**$K_{med,eq}$ = 72.0 MPa√m**

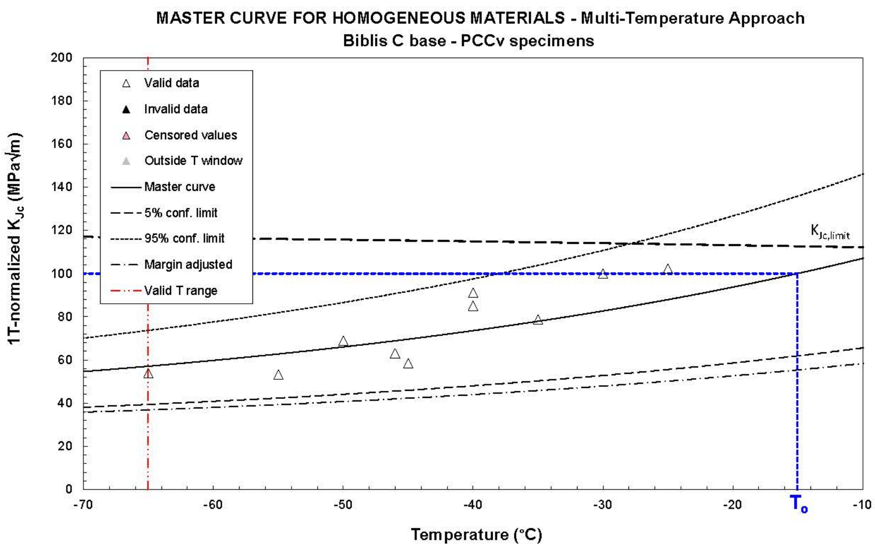
**dK/dt = 1.05E+02 MPa√m/s**

4. Master curve fit to data

Margin adj. (85 % conf.): 10.3 °C (est.) Stand. dev. on $T_o$ = 7.2 °C (est.)

| T (°C) | $K_{Jc(exp)}$ (MPa√m) | $K_{Jc,1T}$ (MPa√m) | $K_{MC(1T)}$ (MPa√m) | *5% conf.* (MPa√m) | *95% conf.* (MPa√m) | *5% L.B.* (MPa√m) |
|---|---|---|---|---|---|---|
| -65 | 62.5 | 53.7 | | | | |
| -55 | 61.8 | 53.1 | | | | |
| -50 | 81.6 | 68.8 | | | | |
| -46 | 74.4 | 63.1 | | | | |
| -45 | 68.3 | 58.3 | | | | |
| -40 | 109.8 | 91.2 | | | | |
| -40 | 102.1 | 85.0 | | | | |
| -35 | 94.1 | 78.7 | | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | -30 | 120.9 | 99.9 | | | | |
| | -25 | 123.8 | 102.2 | | | | |
| Start T (°C) | -90 | | | 46.8 | 34.1 | 58.9 | 32.5 |
| -90 | -80 | | | 50.4 | 35.9 | 64.0 | 34.0 |
| Step T (°C) | -70 | | | 54.6 | 38.1 | 70.1 | 35.8 |
| 10 | -60 | | | 59.8 | 40.8 | 77.6 | 38.1 |
| Min test T (°C) | -50 | | | 66.0 | 44.1 | 86.6 | 40.7 |
| -65 | -40 | | | 73.6 | 48.0 | 97.5 | 44.0 |
| Max test T (°C) | -30 | | | 82.7 | 52.8 | 110.7 | 47.9 |
| -25 | -20 | | | 93.7 | 58.6 | 126.7 | 52.6 |
| Max $K_{Jc,1T}$ | -10 | | | 107.0 | 65.6 | 146.0 | 58.4 |
| 102.2 | 0 | | | 123.2 | 74.0 | 169.3 | 65.3 |
| | 10 | | | 142.7 | 84.2 | 197.5 | 73.7 |
| | 20 | | | 166.2 | 96.5 | 231.6 | 83.9 |
| | 30 | | | 194.7 | 111.5 | 272.9 | 96.1 |
| | 40 | | | 229.2 | 129.5 | 322.8 | 111.0 |
| | 50 | | | 270.9 | 151.3 | 383.1 | 128.9 |
| | 60 | | | 321.3 | 177.7 | 456.0 | 150.6 |
| | 70 | | | 382.2 | 209.6 | 544.2 | 176.8 |




Plot $K_{Jc,limit}$?
YES

**Determination of Reference Temperature, $T_0$, for Ferritic Steels in the Transition Range**
**ASTM E1921-20**

Homogeneity Screening Procedure based on SINTAP method

1. Data censoring

| Specimen code | T (°C) | $K_{Jc(exp)}$ (MPa √m) | $K_{Jc(1T)}$ (MPa √m) | $K_{CENS}$ (MPa √m) | $\delta_i$ | $K_{analysis}$ (MPa √m) |
|---|---|---|---|---|---|---|
| B02-CV5 | -65 | 62.5 | 53.7 | 57.1 | 1 | 53.7 |
| B02-CV2 | -55 | 61.8 | 53.1 | 62.8 | 1 | 53.1 |
| A08-CV5 | -50 | 81.6 | 68.8 | 66.0 | 0 | 66.0 |
| B02-CV4 | -46 | 74.4 | 63.1 | 68.9 | 1 | 63.1 |
| A08-CV3 | -45 | 68.3 | 58.3 | 69.6 | 1 | 58.3 |
| A08-CV1 | -40 | 109.8 | 91.2 | 73.6 | 0 | 73.6 |
| A08-CV4 | -40 | 102.1 | 85.0 | 73.6 | 0 | 73.6 |
| A08-CV2 | -35 | 94.1 | 78.7 | 77.9 | 0 | 77.9 |
| B02-CV1 | -30 | 120.9 | 99.9 | 82.7 | 0 | 82.7 |
| B02-CV3 | -25 | 123.8 | 102.2 | 87.9 | 0 | 87.9 |

Benchmark $T_o$ = -15.0 °C

2. Analysis of the censored data and obtainment of a new estimate of $T_o$

USE LIMITS : YES

T limits (°C)
-70.7
29.3

| Specimen code | T (°C) | $K_{analysis}$ (MPa √m) | $\delta_i$ | 1° member | 2° member |
|---|---|---|---|---|---|
| B02-CV5 | -65 | 53.7 | 1 | 0.0098 | 0.0033 |
| B02-CV2 | -55 | 53.1 | 1 | 0.0102 | 0.0018 |
| A08-CV5 | -50 | 66.0 | 0 | 0.0000 | 0.0050 |
| B02-CV4 | -46 | 63.1 | 1 | 0.0106 | 0.0031 |
| A08-CV3 | -45 | 58.3 | 1 | 0.0106 | 0.0018 |
| A08-CV1 | -40 | 73.6 | 0 | 0.0000 | 0.0052 |
| A08-CV4 | -40 | 73.6 | 0 | 0.0000 | 0.0052 |
| A08-CV2 | -35 | 77.9 | 0 | 0.0000 | 0.0052 |
| B02-CV1 | -30 | 82.7 | 0 | 0.0000 | 0.0053 |
| B02-CV3 | -25 | 87.9 | 0 | 0.0000 | 0.0053 |

*Sum of 1° member:* 0.041

*Sum of 2° member:* 0.041

*Difference :* 0.000

**$T_o$ = -20.7 °C**

**$T_{0(stepn)} \geq T_{0(stepn-1)}$ + 0.5 °C ?**
**NO - Analysis Completed**

**$T_{0scrn}$ = -15.0 °C**

**Screening Criterion**
THE MATERIAL IS
HOMOGENEOUS