

# DYNAMIC MASTER CURVE and DUCTILE-BRITTLE TRANSITION TEMPERATURE OF 9Cr-1Mo STEEL

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## ABSTRACT

The DBTT of 9Cr-1Mo steel has been characterised by reference temperature ( $T_0$ ) based Master Curve (MC) approach. The MC was developed at the dynamic loading conditions (stress intensity factor change rate  $\sim 10^6$  MPa $\sqrt{m/s}$  at a loading rate of 5.12 m/s) using pre-cracked Charpy specimens (PCVN) following ASTM E 1921 guidelines. To minimise the inertial oscillations effect on load determination, the initial  $T_0$  was determined by tests conducted at a hammer velocity of  $\sim 1.12$  m/s; this was further converted to  $T_0$  at 5.12 m/s applying an test velocity dependent shift to  $T_0$ . Thus, the  $T_0$  for 9Cr-1Mo steel at dynamic condition was determined to be  $-52$  °C. Using a modified Schindler procedure to evaluate  $K_{Jd}$  from instrumented Charpy V-notch tests,  $T_0$  was also estimated for the 9Cr-1Mo steel and the result shows close agreement with the  $T_0$  evaluated from the pre-cracked Charpy tests. ASME  $K_{IR}$ -curve approach proves to be too conservative compared to the real trend of the fracture toughness with temperature.

## 1. INTRODUCTION

Irradiation induced shift in ductile-brittle transition temperature (DBTT) of 9Cr-1Mo steel is a matter of concern in its application as a wrapper materials in fast reactors [1,2]. Thus it is essential to characterise the DBTT of the unirradiated material on the basis of dynamic fracture toughness variation in transition regime.

In describing the DBTT, the  $RT_{NDT}$  based  $K_{IR}$  curve [3,4] approach, being used since 1972, is purely empirical and based on the data obtained from reactor pressure vessel steels only. However, it is now well understood that the size, shape and distribution of the carbides ahead of the crack tip leads to scatter in fracture toughness. A description of brittle fracture mechanism through 'weakest link' theory [5] and subsequent modeling of scatter using a three parameter Weibull distribution recently has led to the development of Master Curve (MC) [6,7], which describes the temperature dependence of fracture toughness of ferritic steels, indexed by a material specific reference temperature,  $T_0$ . ASTM E 1921-97 [8] has recently standardised the process of evaluation of  $T_0$  and the Master Curve under quasi-static loading conditions. However, its application to higher strain rates needs further verification/validation by dynamic fracture toughness evaluation. In this paper, the reference temperature,  $T_0$ , has been determined for the 9Cr-1Mo steel in a dynamic loading condition. A parallel effort has been made to evaluate  $T_0$  with conventional Charpy (CVN) tests also. The Master Curve thus determined was compared with the  $RT_{NDT}$  based ASME  $K_{IR}$  curve. The results would enable evaluating the DBTT of 9Cr-1Mo steel in the light of dynamic fracture toughness.

## 2. MATERIAL AND $T_0$ DETERMINATION PROCEDURE

### 2.1. Material

The 20 mm thick plate of 9Cr-1Mo steel were supplied by M/s Creusot-Loire Industrie, France. The detailed chemical composition (in wt %) is given as C: 0.10, Cr: 8.44, Mo: 0.94, Ni: 0.17, Cu: 0.10, Si: 0.48, S: 0.002, P: 0.007, Al: 0.011, Fe: balance. The plate was normalised at 950 °C for 30 minutes and tempered at 750 °C for 60 minutes.

### 2.2. $T_0^{dy}$ from Pre-Cracked Charpy Test (PCVN)

The dynamic fracture toughness ( $K_{Jd}$ ) was determined from the pre-cracked Charpy test. To minimise the inertial oscillations effect on load determination, the initial  $T_0$  was determined by tests conducted at a hammer velocity of ~1.12 m/s; this was further converted to  $T_0$  at 5.12 m/s applying an test velocity dependent shift in  $T_0$ . ASTM E 1921 suggests that the test temperature should be selected such that it yields fracture toughness values (corresponding to 1” thickness specimens) close to 100 MPa√m. As per this guideline, following some trial and error tests, the test temperature was selected as -50 °C. The initial crack length was measured by a travelling microscope, using ten point averaging method. The test temperature, test velocity, validity of test results and 1” corrected  $K_{Jd}$  values are reported in Table. 1. Two tests have been conducted at a hammer velocity of 5.12 m/s, at -20 °C and the corresponding results are also shown in Table 1.

Table.1  $K_{Jd}$  with validity limits and  $T_0$  from the PCVN tests of 9Cr-1Mo steel.

Test Temp(°C)	$V_0$ , (m.s <sup>-1</sup> )	$a_0$ (mm)	$K_{Jd}$ (MPa.√m)	$\sigma_{yd}$ (Mpa)	Validity limit, (MPa.√m)	Validity results	$K_{Jd}$ (1 inch) (MPa.√m)
-70	1.08	4.40	57.84	-	-	-	49.97
-55	1.06	4.42	56.38	-	-	-	48.82
-50	1.11	4.50	137.13	717.34	166.13	Valid	112.78
-50	1.11	4.39	162.26	707.60	166.64	Valid	132.68
-50	1.14	4.70	186.98	742.62	165.90	Invalid	152.21*
-50	1.15	4.38	83.528	713.61	167.53	Valid	70.32
-50	1.12	4.60	118.87	745.56	167.74	Valid	98.31
-50	1.14	4.54	$P_F$ not clear	717	165.55	-	-
-50	1.10	4.60	243.87	721.56	165.16	Invalid	197.33*
-50	1.10	4.80	60.00	$P_F < P_{GY}$	-	Valid	51.70
-50	1.13	4.88	163.00	762.5	165.23	Valid	136.44
-50	1.10	5.09	70	$P_F < P_{GY}$	-	Valid	60
-50	1.12	4.63	133.95	712.10	163.60	Valid	110.26
-20	5.12	4.87	286.70	779.70	166.70	Invalid	230.57*
-20	5.12	4.62	296.50	789.20	171.60	Invalid	233.44*

The cumulative probability of failure,  $p_f$ , the scale parameter of the Weibull distribution,  $K_0$  and the median  $K_{Jd}$  were calculated according to ASTM E 1921. The  $T_0^{dy}$  thus determined was  $-59.4$  °C. The corresponding Weibull plot is shown in Fig. 1. A near slope four agreement has been observed in the Weibull plot.

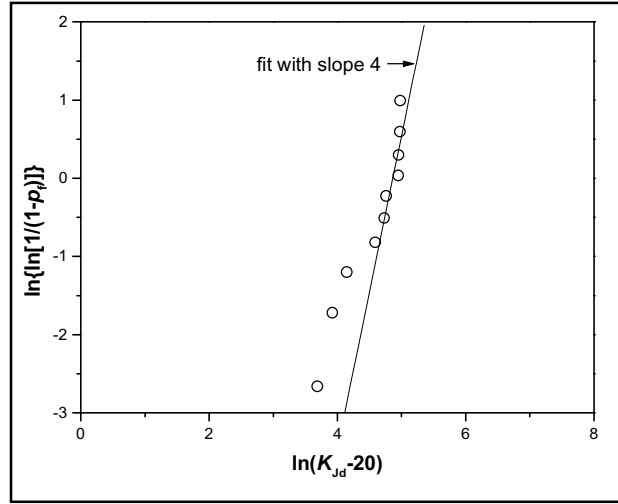


Figure 1. Weibull plot from the  $K_{Jd}$  data, obtained from PCVN tests.

To account for the test velocity effect on the reference temperature,  $T_0$ , amongst various equations proposed by Yoon *et al.*[9], the following represent the highest and the lowest values respectively:

$$\text{For A515 steel,} \quad T_0 = 6.1 \ln (dK/dt) - 18 \quad [1a]$$

$$\text{For A533B weld,} \quad T_0 = 2.7 \ln (dK/dt) - 87 \quad [1b]$$

$dK/dt$  is the rate of change of stress intensity factor.

In a more generalised form, Schindler *et al.* [10] have proposed the following equation for the shift in  $T_0$ , associated with the change in test velocity or the stress intensity factor rate as:

$$\Delta T \cong (22 - 0.016\sigma_y) \log \frac{\dot{K}_{ref}}{\dot{K}_{test}} \cong (22 - 0.016\sigma_y) \log \frac{\dot{V}_{ref}}{\dot{V}_{test}} \quad [2]$$

where  $\sigma_y$  is the yield stress at the test temperature/strain rate.

In the present campaign, the stress intensity factor rate estimated at  $\sim 1.12$  m/s for the  $-50$  °C tests yields a value of  $2.754(\pm 0.254) \cdot 10^5$  MPa $\sqrt{m/s}$ . From the two

PCVN specimens tested at 5.12 m/s, the stress intensity factor rate was estimated as  $\sim 10^6$  MPa $\sqrt{\text{m/s}}$  for the test velocity of 5.12 m/s. Applying these results in the Eqn. 1-a&b, for tests in 1.12 and 5.12 m/s, the  $T_0$  yields a shift of 14 and 6.2 °C respectively. However, the Eqn. 2 shows a shift of  $\sim 7$  °C based on the stress intensity based calculation and 7.5 °C based on velocity based estimation. It is important to mention here that the dynamic yield stress estimation from PCVN tests gives a higher value than that estimated from the Charpy V-notch tests at a particular temperature [11]. Here, the yield stress estimated from Charpy V notch tests, 660 MPa at  $-50$  °C [15], has been used to arrive at a conservative estimate (higher  $\Delta T$ ) of shift.

From the above mentioned various estimates for the test velocity induced shifts of  $T_0$ , it is observed that the Schindler,s velocity based estimation (Eqn. 2) is more tangible from a practical point of view and considering the scatter involved in all the test parameters in determining  $T_0$ , the result seem to be reasonable. So, the  $T_0^{\text{dy}}$  corresponding to test velocity of 5.12 m/s is estimated as  $-51.9$  °C. The Master Curve at test velocity 5.12 m/s is plotted using  $T_0^{\text{dy}}$  as  $-52$  °C.

### 2.3. $T_0^{\text{dy}}$ from Charpy V-Notch Tests

$T_0^{\text{dy}}$  from Charpy V-notch test was estimated from the  $K_{\text{Jd}}$  values at different temperatures.  $K_{\text{Jd}}$  values were estimated from the  $J_{\text{Id}}$ , where  $J_{\text{Id}}$  values had been evaluated from the load-displacement plots and the absorbed Charpy energy using Eqn. 3.

$$J_0 = \frac{7.33 \cdot n \cdot C_v \cdot 10^{-3}}{1 - 1.47 \cdot \left(\frac{C_v}{\sigma_{\text{Jd}}}\right)} \quad [3]$$

The original equation has been proposed by Schindler [12] and later modified by Sreenivasan *et al* [13] where the work hardening exponent  $n$  was determined directly from the load-displacement diagram obtained from the instrumented Charpy V-notch tests alone.

The validity of the estimated  $K_{\text{Jd}}$  values were evaluated as per ASTM E 1921 procedure with a ligament length of 8 mm and subsequently were converted to 1” size equivalence. An exponential fit has been obtained through the 1” size corrected and valid  $K_{\text{Jd}}$  data and the  $T$ - $K_{\text{Jd}}$  pairs between 80 to 120 MP $\sqrt{\text{m}}$  were selected and analysed for evaluating the reference temperature using the multi-temperature equation proposed by Wallin [7]. This yields a  $T_0^{\text{dy}}$  of  $-30$  °C. However, it yields a result of  $-47.3$  °C if the fit incorporates all the invalid data also. The result shows a potential in determining dynamic reference temperature from the Charpy-V notch tests alone.

### 3. THE DYNAMIC MASTER CURVE

Using the reference temperature,  $T_0^{\text{dy}}$ , as  $-52\text{ }^{\circ}\text{C}$ , the dynamic Master Curve corresponding to 5.12 m/s is constructed as per ASTM 1921 guideline. Fig. 2 shows the dynamic Master Curve with respect to the pre cracked test results and the ASME  $K_{\text{IR}}$  curve with  $RT_{\text{NDT}} = -25\text{ }^{\circ}\text{C}$  [15]. According to the shift of  $7.5\text{ }^{\circ}\text{C}$  in  $T_0^{\text{dy}}$  at test velocity of 5.12 m/s with respect to the 1.12 m/s, the temperatures corresponding to the PCVN  $K_{\text{Jd}}$  results are also shifted by  $7.5\text{ }^{\circ}\text{C}$ . Fig. 2 also compares the trend in the  $K_{\text{Jd}}$  or  $K_{\text{Id}}$  values, obtained from the Charpy-V and drop weight tests [14,15], with the dynamic Master Curve.

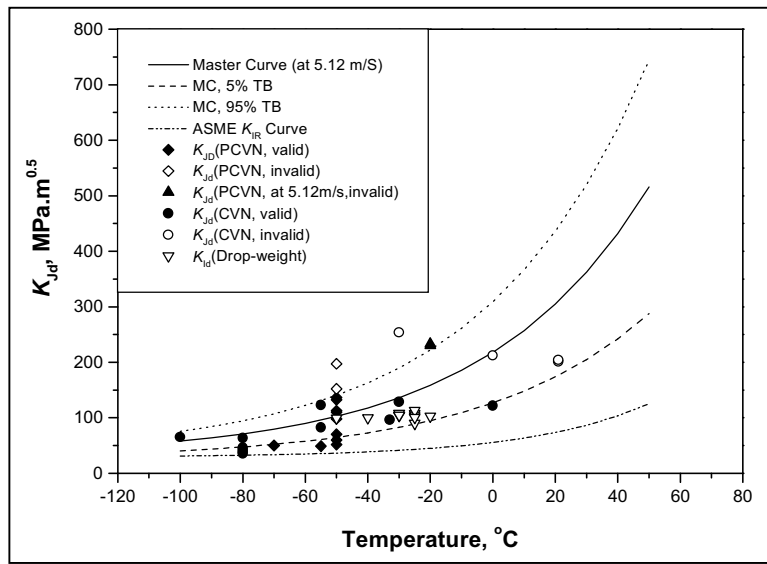


Figure 2. Master Curve (at 5.12 m/s) and  $K_{\text{IR}}$  curve for 9Cr-1Mo steel.

The trend in the  $K_{\text{Jd}}$  values obtained from the Charpy V-notch tests shows good agreement with the Master Curve; even the invalid values (except one) lie within the tolerance bound of the Master Curve. Thus, the  $T_0^{\text{dy}}$ , determined as  $-47.3\text{ }^{\circ}\text{C}$ , incorporating all the valid and invalid data from the Charpy-V notch tests seems reasonable. Compared to the Master Curve, the ASME  $K_{\text{IR}}$  curve proves to be too conservative.

### 4. CONCLUSIONS

1. The  $T_0^{\text{dy}}$  determined for 9Cr-1Mo steel at a loading rate of 5.12 m/s is  $-52\text{ }^{\circ}\text{C}$  where as the  $RT_{\text{NDT}}$  is determined as  $-25\text{ }^{\circ}\text{C}$ .
2. ASME  $K_{\text{IR}}$ -curve approach proves to be too conservative compared to the real trend of the fracture toughness with temperature, as shown by the dynamic Master Curve.

3. The  $K_{Id}$  determined from the conventional Charpy V-notch tests and the drop-weight tests follow the trend shown by the dynamic Master Curve. The methodology suggested here for evaluating  $T_0^{dy}$  using only CVN test bears potential, but needs to be verified with other materials.

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