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# Determination of Reference Temperature $T_0$ for Steel JRQ in an Unirradiated State and Construction of Master Curve

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# Определение эталонной температуры $T_0$ для стали JRQ и построение "Master curve"

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Рассмотрены результаты исследования трещиностойкости корпусной реакторной стали JRQ и построена зависимость коэффициента интенсивности напряжений от температуры (-196...+50°C), которая является так называемой "Master curve". При исследовании вязкости разрушения использовали малые компактные образцы толщиной 1/2T, которые вырезали из заготовки стали JRQ размером 225×198×165 мм, поставленной МАГАТЭ.

Испытания проводили в соответствии со стандартом ASTM E 1921-97 "Determination of Reference Temperature  $T_0$  for Ferritic Steels in the Transition Range". Для уточнения температуры  $T_0$  и выполнения необходимых расчетов проводили также испытания на ударную вязкость и динамические испытания по определению модулей упругости E и G в интервале температур -196...+350°C.

Построена "Master curve" по результатам испытаний двух типов компактных образцов на различном оборудовании и определены ее доверительные интервалы для различных веро-ятностей.

*Ключевые слова*: вязкость разрушения, температура вязко-хрупкого перехода, *J*-интеграл, "Master curve", прочность.

**Introduction**. The known methods for obtaining reliable data on fracture toughness under conditions of plane deformation require tests to be conducted on long specimens and involve high consumption of hard-to-get materials. For reactor steels, for which it is necessary to determine the transition temperature

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shift in the irradiated state as compared to the unirradiated one, the use of large-size specimens is unacceptable at all, and surveillance specimens are small in size and their tests involving the methods of nonlinear fracture mechanics are rather labor consuming and expensive. Employing Master Curve approach for ferritic steels enables obtaining reliable data and the temperature dependence of the crack-growth resistance variation at the lower boundary of transition over the wide temperature range by testing small specimens [1]. According to the recent standardizing documents, Master Curve is constructed on the basis of the results of testing small-size specimens at a single temperature value such that slow steady growth of a crack before the beginning of instability is practically precluded. This makes it possible to simplify considerably the procedure for determining the *J*-integral and to use the experimental basic approach of nonlinear fracture mechanics. Below we consider the results of determining the reference temperature  $T_0$  and constructing Master Curve on the basis of the experiments performed for reactor steel in an unirradiated state.

**Material and Specimens**. Compact-tension specimens for the construction of Master Curve and standard Charpy specimens for the determination of brittle-to-ductile transition temperature were fabricated from blocks of steel JRQ with dimensions of  $225 \times 198 \times 165$  mm, its chemical composition is given in Table 1.

Т	а	b	1	e	-1

**Chemical Composition of Steel JRQ** 

ľ	С	Si	Mn	Ni	Мо	Cr	Р	Cu	S	Al	V
	0.18%	0.24%	1.42%	0.84%	0.51%	0.12%	0.017%	0.14%	0.004%	0.014%	0.002%

For static fracture toughness tests we used 1/2T CT specimens of two types (Fig. 1), whose orientation corresponded to the T–L direction [2] of the JRQ steel billet. These specimens differed in dimensions and design of the attachment points of the displacement transducer. Specimens of the second type had side notches made after growing an initial fatigue crack (see Fig. 1b). The dimensions of the second type specimens corresponded to those of the surveillance specimens installed in reactor pressure vessels of the nuclear power plants in Ukraine. Specimens of these two types were tested to assess how differences in dimensions and design affect the magnitude of the reference temperature and the trend of Master Curve for steel JRQ in an unirradiated state. Fracture toughness tests were performed in accordance with [1] at a temperature of  $T_{28j} - 28$ . The temperature  $T_{28j}$  was determined from the results of testing Charpy specimens ( $10 \times 10 \times 55$  mm) using a two-column pendulum impact testing machine with a store of impact energy 300 J in the temperature range from  $-196^{\circ}$ C to  $+270^{\circ}$ C.

The first type specimens (see Fig. 1a) were tested at a conventional laboratory of the Institute of Problems of Strength of the National Academy of Sciences of Ukraine using Instron-1126 testing machine with a hydromechanical converter with an ultimate load of 250 kN that allowed us to perform both stress-and strain-controlled tests. Most of the second type specimens was tested using an electromechanically driven Instron-8500 testing machine with an ultimate load of 100 kN equipped with remote control and installed in a hot chamber of the

Institute for Nuclear Research of the National Academy of Sciences of Ukraine to correspond to the requirements of the ENUCRA program (see Fig. 1b). Only some of those specimens were tested in the Instron-1126 testing machine for comparison.



Fig. 1. Dimensions of 1/2T CT specimens of the first (a) and second (b) types in mm.

In impact toughness testing the specimens were heated in an electrical resistance furnace, and cooled in a mixture of liquid nitrogen with alcohol. In fracture toughness testing using the Instron-1126 testing machine 1/2T CT specimens were cooled in a cryogenic chamber also using the mixture of liquid nitrogen with alcohol (see Fig. 1a), specimens of the second type tested in the hot chamber on the Instron-8500 testing machine were cooled in the vapors of nitrogen (see Fig. 1b). Both methods of cooling allowed obtaining a stable low temperature in the range under study.

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The temperature was measured with thermometers and thermal converters, which had been put to metrological tests. For measuring loads and crack opening displacement, elements of the measurement systems (devices, gauges, and transducers) of the Instron testing machines were used that allowed determining loads and displacements to a high accuracy, and obtaining reliable P - V diagrams. The procedure for measuring temperature, load and displacement, and also the technique for growing initial fatigue cracks met the requirements of [1].

To get a deep insight into the mechanical behavior of the steel investigated over the operating temperature range, and to obtain data on strength and elasticity characteristics essential in the determination of the load causing initial fatigue crack, and the limiting value of  $K_{Jc}$  and its current values with respect to the *J*-integral values, short-term strength tests were performed with an Instron testing machine and elasticity was determined by the dynamic method with the use of a special resonant equipment [3].

When testing short-term strength, standard five-fold specimens were employed with the working section 10 mm in diameter and 50 mm in length. The modulus of elasticity E and shear modulus G were found for cylindrical specimens 8 mm in diameter and 120 mm in length from their resonance frequency in bending and torsional vibrations, respectively.

**Construction of Master Curve**. The Master Curve concept is based on the results of fundamental investigations performed by Wallin [4, 5] and involves the following principles:

– probability of brittle fracture  $P_f$  for a specimen chosen arbitrarily from the batch is described by a three-parametric equation of Weibull [4]:

$$P_{f} = 1 - \exp\left[-\left(\frac{K_{\rm Ic} - K_{\rm min}}{K_{0} - K_{\rm min}}\right)^{4}\right],\tag{1}$$

where  $K_0$  is the scale parameter, whose magnitude is governed by the specimen thickness and test temperature, and  $K_{\min}$  is the least crack-growth resistance, whose magnitude according to [1] is assumed to be equal to 20 MPa $\sqrt{m}$ ;

- the level of the crack-growth resistance depends on the specimen thickness and this relationship is described by the following formula [5]:

$$\frac{K_{\rm Ic}^{X} - K_{\rm min}}{K_{\rm Ic}^{Y} - K_{\rm min}} = \left(\frac{B_{Y}}{B_{X}}\right)^{1/4},\tag{2}$$

where  $K_{Ic}^X$  and  $K_{Ic}^Y$  are the values of the stress intensity factors for specimens of thickness  $B_X$  and  $B_Y$  at the same probability of brittle fracture  $P_f$ ;

- the median value of the crack-growth resistance (at  $P_f = -0.5$ ) as a function of the temperature for 1T CT specimens is described by the equation, which is the equation of Master Curve [1]:

$$K_{Jc(med)} \approx 30 + 70 \exp[0.019(T - T_0)],$$
 (3)

where  $T_0$  is the reference temperature at which  $K_{Jc(med)}$  is taken to be equal to 100 MPa $\sqrt{m}$  [1].

Using Eqs. (1)–(3) for specimens of any thickness at any fracture probability temperature dependences of the crack-growth resistance can be calculated from the results of testing the material for crack-growth resistance at a single temperature value. The procedure of such tests and regulating requirements for them are described in detail in [1]; they were used for obtaining the data considered in the present study.

The temperature at which the fracture toughness tests were performed was defined as

$$T = T_{28\,i} + C, \tag{4}$$

where  $C = -28^{\circ}$ C for 1/2T specimens, and the temperature  $T_{28j}$ , as noted above, was determined from the results of impact toughness testing the Charpy specimens. The data obtained from such tests for the specimens with the L–T (along the rolling direction) and T–L (across the rolling direction) orientations are given in Fig. 2. As follows from these results, a considerable scatter of the impact toughness values is observed in the region of the brittle-to-ductile transition, that is indicative of instability of the property of the JRQ steel billet to resist brittle fracture that was also confirmed by the results of fracture toughness tests. The  $T_{28j}$  value was determined for the T–L orientation and amounted to  $-52^{\circ}$ C. Thus, the crack-growth resistance tests according to the guidelines of [1] and on the basis of Eq. (4) should be carried out at the temperature  $T = -80^{\circ}$ C.



Fig. 2. Results of Charpy testing of unirradiated JRQ material.

To calculate the limiting value of the stress intensity factor at this temperature we used equation

$$K_{Jc(\text{limit})} = \sqrt{Eb_0 \sigma_Y / 30},\tag{5}$$

where E is the modulus of elongation,  $b_0$  is the ligament of the compact specimen  $b_0 = W - a_0$ , and  $\sigma_Y$  is the yield strength of the material at this temperature. The mechanical characteristics of JRQ steel obtained for the operating temperature range are given in Table 2, and the temperature dependences of the moduli E and G are illustrated in Fig. 3.

Mechanical Characteristics of Steel JRQ

Temperature, ℃	Ultimate strength $\sigma_u$ , MPa	Yield strength $\sigma_Y$ , MPa	Residual elongation $\delta, \%$		
20	640.0	490.0	18.0		
-80	790.0	630.0	18.5		
-196	990.0	960.0	0.5		



Fig. 3. Temperature dependences of the elasticity and shear moduli E and G.

It should be emphasized that the break of monotonicity of the curve trend was observed for the elastic modulus E in the region of the brittle-to-ductile transition temperatures that is indicative of abnormal changes in the structure of the material in this region as the temperature decreases.

For the values of E and  $\sigma_Y$  obtained at -80°C,  $K_{Jc(limit)} = 233.0 \text{ MPa}\sqrt{\text{m}}$ .

1/2T CT specimens were tested in the mixture of liquid nitrogen with alcohol or vapors of nitrogen at  $-80^{\circ}$ C with a simultaneous recording of P - V diagrams. From the results of processing the diagrams we determined the values of the *J*-integral as a sum of its elastic and plastic components:

$$J_c = J_e + J_p. \tag{6}$$

The elastic and plastic components were defined as

$$J_e = \frac{K_e^2}{E} \tag{7}$$

and

$$J_p = \frac{\eta A_p}{Bb_0},\tag{8}$$

respectively, where  $K_e$  is the value of the stress intensity factor determined by the known procedure [1],  $\eta$  is the coefficient dependent on the dimensions of the CT specimen  $b_0$  and W,  $A_p$  is the work of plastic deformation determined from the corresponding area of the P - V diagram, and B is the specimen thickness.

The quantity  $K_{Jc}$  was calculated for each individual specimen depending on the particular  $J_c$  value from the relation

$$K_{Jc} = \sqrt{J_c E}.$$
(9)

The values of  $J_c$  and  $K_{Jc}$  for qualification specimens of two types, which remained after the rejection performed according to the requirements of the standard [1], are listed in Tables 3, 4.

T a b l e 3 The Values of Crack-Growth-Resistance Characteristics for the First Type Specimens at  $T = -80^{\circ}$ C

No	1	2	3	4	5	6	7	8
$J_c$ , kJ/m <sup>2</sup>	8.45	31.0	32.5	41.8	51.7	21.5	34.3	86.9
$K_{Jc}$ , MPa $\sqrt{\mathrm{m}}$	43.00	82.5	84.6	95.9	106.6	68.6	84.9	58.3

Table 4

The Values of Crack-Growth-Resistance Characteristics for the Second Type Specimens at  $T = -80^{\circ}$ C

No	1	2	3	4	5	6	7	8*	9*
$J_c$ , kJ/m <sup>2</sup>	23.7	27.2	68.1	22.3	14.1	28.6	16.5	25.1	50.0
$K_{Jc}$ , MPa $\sqrt{\mathrm{m}}$	72.2	77.4	122.4	70.1	55.6	75.6	60.2	74.1	104.9

\* Specimens 8 and 9 were tested in the Instron-1126 testing machine using the mixture of alcohol with liquid nitrogen.

Master Curve was constructed and the temperature  $T_0$  was determined for the 1T specimens. For this reason, on the basis of the data given in Tables 2 and 3,  $K_0$  and  $K_{Jc(med)}$  were determined successively for specimens of thickness 1/2T and 1T from Eqs. (1) and (2). Below are given their values for the specimens of the first type:

$$K_{0(1/2T)} = \left[\frac{\sum_{i=1}^{8} (K_{Jc(i)} - 20)}{N - 0.3068}\right]^{1/4} + 20 = 86.4 \text{ MPa}\sqrt{m},$$
$$K_{0(1T)} = 20 + [K_{0(1/2T)} - 20] \left(\frac{B_i}{B_{(1T)}}\right)^{1/4} = 75.8 \text{ MPa}\sqrt{m},$$
$$K_{Jc(1T)(med)} = \frac{1}{4} [K_{0(1T)} - 20] [\ln(2)] + 20 = 70.9 \text{ MPa}\sqrt{m},$$

and for the specimens of the second type:

$$K_{0(1/2\mathrm{T})} = \left[\frac{\sum_{i=1}^{9} (K_{Jc(i)} - 20)}{9 - 0.3068}\right]^{1/4} + 20 = 90.1 \text{ MPa}\sqrt{\mathrm{m}},$$
$$K_{0(1\mathrm{T})} = 20 + [K_{0(1/2\mathrm{T})} - 20] \left(\frac{B_i}{B_{(1\mathrm{T})}}\right)^{1/4} = 74.9 \text{ MPa}\sqrt{\mathrm{m}},$$
$$K_{Jc(1\mathrm{T})(med)} = \frac{1}{4} [K_{0(1\mathrm{T})} - 20] [\ln(2)] + 20 = 70.6 \text{ MPa}\sqrt{\mathrm{m}},$$

The magnitude of the reference temperature was determined from Eq. (3):

$$T_0 = T^* - \frac{1}{0.019} \ln \left[ \frac{K_{Jc(1T)(med)} - 30}{70} \right],$$

where  $T^* = -80^{\circ}$ C.

We obtained  $T_0 = -51.7$  °C for the specimens of the first type and  $T_0 = -51.3$  °C for those of the second type.

We assumed  $T_0 = -52^{\circ}$ C for  $K_{Jc} = 100 \text{ MPa}\sqrt{\text{m}}$ .

Thus, the final equation of Master Curve for unirradiated steel JRQ has the form

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$$K_{Jc(med)} = 30 + 70 \exp[0.019(T+52)].$$
 (10)

Master Curve is depicted in Fig. 4; it is known that this curve corresponds to the 50% cumulative failure probability. Standard deviations for the  $T_0$  value obtained with allowance for the statistical character of fracture of ferritic steel JRQ are defined as

$$\sigma = \frac{\beta}{\sqrt{N}},\tag{11}$$

where N is the number of the qualification specimens and  $\beta$  is the coefficient dependent on the magnitude of  $K_{Jc(med)}$ .



Fig. 4. "Master Curve" results obtained using JRQ materials: 1 - specimens I type, Instron-1126; 2 - specimens II type, Instron-8500; 3 - specimens II type, Instron-1126.

In our case, N = 8 and  $\beta = 18.8$  for  $K_{Jc(med)} = 70.9$  MPa $\sqrt{m}$ . Therefore, for the specimens of the first type,  $\sigma = 6.6^{\circ}$ C. For  $K_{Jc(med)} = 70.6$  MPa $\sqrt{m}$ , N = 9 and  $\beta = 18.8$ . Therefore, for the specimens of the second type,  $\sigma = 6.3^{\circ}$ C.

The tolerance bounds are determined from the following equations based on the use of Eq. (1):

the lower 1% tolerance bound:

$$K_{Jc(0.01)} = 23.5 + 24.4 \exp[0.019(T - T_0)];$$
(12)

the lower 5% tolerance bound:

$$K_{Jc(0.05)} = 25.2 + 36.6 \exp[0.019(T - T_0)];$$
(13)

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the upper 95% tolerance bound:

$$K_{Jc(0.95)} = 34.5 + 101.3 \exp[0.019(T - T_0)];$$
 (14)

the upper 99% tolerance bound:

$$K_{Jc(0.99)} = 36.1 + 112.8 \exp[0.019(T - T_0)].$$
<sup>(15)</sup>

Figure 4 shows the curves corresponding to the above tolerances that bound the scatter range of the results of crack-growth resistance testing with a given failure probability when realizing the method of Master Curve. Within the scatter range of Master Curve points are marked that correspond to the values of  $K_{Jc}$ obtained for 1T CT specimens on the basis of the results of crack-growth testing 1/2T CT specimens of steel JRQ at  $-50^{\circ}$ C,  $-80^{\circ}$ C,  $-100^{\circ}$ C,  $-110^{\circ}$ C, and  $-196^{\circ}$ C.

## CONCLUSIONS

1. The equation of Master Curve for steel JRQ in an unirradiated state has the following form:

$$K_{Jc(med)} = 30 + 70 \exp[0.019(T+52)].$$

2. Reference temperatures determined for the two types of 1/2T CT specimens of steel JRQ are little different and amount to -51.7 °C and -51.3 °C, for the specimens of the first and second types, respectively.

3. The procedure for determining the reference temperature  $T_0$  from testing small 1/2T CT specimens in the hot chamber was developed on the Instron-8500 testing machine within the framework of the ENUKRA program. It gives repeatable results that agree satisfactorily with those of testing using certified equipment of high accuracy on the basis of the Instron-1126 testing machine under comfortable (i.e., without remote control) laboratory conditions. Therefore, the procedure for fracture toughness testing within the framework of the ENUCRA program can be treated as efficient and dependable, and the results obtained employing this procedure can be considered as reliable.

### Резюме

Розглянуто результати дослідження корпусної реакторної сталі JRQ і побудовано залежність коефіцієнта інтенсивності напружень від температури ( $-196...+50^{\circ}$ С), яка є так званою "Master curve". При дослідженні в'язкості руйнування використовували малі компактні зразки товщиною 1/2T, що вирізали з поставляємої МАГАТЕ заготовки сталі JRQ розміром 225×198×165 мм.

Випробування проводили у відповідності до стандарту ASTM Е 1921-97 "Determination of Reference Temperature  $T_0$  for Ferritic Steels in the Transition Range". Для уточнювання температури  $T_0$  і виконання необхідних розрахунків проводили також випробування на ударну в'язкість та динамічні випробування по визначенню модулей пружності E і G в інтервалі температур  $-196...+350^{\circ}$ С.

Побудовано "Master curve" за результатами випробувань двох типів компактних зразків на різному обладнанні і визначено її довірчі інтервали для різних імовірностей.

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