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# Fracture behaviour of 20MnMoNi55 steel in DBT region under corrosive environment

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**Abstract**

Different mechanical properties like strength, fracture toughness and fatigue life of structural components reduce due to corrosion of the material. In this paper the effect of corrosion on fracture toughness in ductile to brittle transition (DBT) region of 20MnMoNi55 steel is investigated. The corrosion properties of the material are measured by electrochemical impedance spectroscopy (EIS) test. The loss of ductility and fracture toughness due corrosion is measured by measuring master curve reference temperature in DBT region. Both single and multi–temperature method is used to measure the master curve reference temperature for both base material and corroded specimen. A comparative study is made to measure embrittlement that occurs due to corrosion in DBT region. From the EIS study it has been observed that the corrosion resistance properties decrease with time when the material corroded. Also the embrittlement occurring due to corrosion is reflected in the shift in master curve.


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*Keywords*: EIS study; fracture toughness; reference temperature; ductile to brittle transition region.

## 1. Introduction

The mechanical properties of structural components of reactor pressure vessels are degraded due to numerous mechanisms and threaten the integrity, safety and service life of structural components. The wear, stress cycles, mechanical shock and impact, creep rupture, embrittlement and corrosion are the some notable mechanisms which affect the mechanical properties of structural components. The reactor pressure vessel is the most critical pressure

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**Nomenclature**

| | |
|---|---|
| B | gross thickness of the specimen |
| E | young's modulus |
| $B_{1T}$ | thickness of one inch thick compact tension specimen |
| $b_0$ | initial ligament length |
| $E_{corr}$ | corrosion potential |
| $I_{corr}$ | corrosion current density |
| $J_C$ | critical *J*-integral at onset of cleavage fracture |
| $K_{JC}$ | elastic plastic critical stress intensity factor determined from J-integral |
| $K_{JC(med)}$ | median fracture toughness value for 50% cumulative failure probability |
| $K_{JC(X)}$ | fracture toughness of tested specimen |
| $K_{min}$ | deterministic constant of Weibull distribution |
| $K_0$ | normalizing fracture toughness corresponding to 63.2% probability |
| $P_f$ | cumulative probability failure |
| $T$ | test temperature |
| $T_0$ | master curve reference temperature |
| ν | poisson's ratio |

boundary component and most reactor pressure vessel (RPV) parts are normally protected by a stainless steel cladding with a high resistance against the intergranular stress corrosion cracking (SSC) and environmentally assisted cracking [1, 2]. Due to the simultaneous action and synergy of a mechanical tension and a corrosive media the stress corrosion cracking is produced. The result is the appearance of microscopic cracks that penetrate and induce the brittle failure. The fracture toughness is significantly reduced when the material is exposed to corrosive media. The environment assisted crack initiation and growth are governed by complex interaction of environmental, loading and material parameters [3]. The ASME code for nuclear fatigue design and evaluation have been quite successful in preventing fatigue cracks and failures in low alloy steel components and therefore, seem to be adequate or conservative under most operating circumstances. But during plant operation, oxidizing agents and hydrogen chemistry water, usually dissolved oxygen and relevant dynamic straining always play an important role in material corrosion. For most of the metals, corrosion is an inevitable process. Corrosion affects structural integrity by reducing fracture strength and fatigue life. It is important to understand and evaluate the corrosion effects on overall fracture and corresponding local strength, in order to estimate the plant life accurately. To quantify the corrosion by measuring the loss of weight suffered by a material exposed to the corrosive environment, the electrochemical studies are used. Among the various electrochemical test viz. electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization studies are the present generation studies used to evaluate the corrosion parameters like open circuit potential, corrosion current density, charge transfer resistance, double layer capacitance, corrosion rate etc. of a material [4–6]. Moreover, these electrochemical tests give an idea of the mechanistic pathway of corrosion. For safe design and operation of reactor pressure vessels (RPVs), it is necessary to develop a systematic fracture toughness database and method to apply this information in integrity assessment [7]. The loss of ductility and fracture in RPV materials due to continuous irradiation is reflected in displacement of fracture toughness – temperature curves along with temperature axis [8]. The master curve approach proposed by Kim Wallin [7] with the ASTM E1921–02 prescribes a three parameter Weibull distribution for the cumulative probability of cleavage fracture toughness in terms of $K_{JC}$ at each temperature over the ductile to brittle transition (DBT) region. The master curve can be used to measure the loss of ductility by measuring $T_0$ in ductile to brittle transition temperature. This $T_0$ is defined as the temperature at which median fracture toughness for 1T (one inch thick) specimen equals 100 MPa√m. In this method, $T_0$ the only fracture toughness characterizing parameter can be evaluated using different methods like single temperature and multi-temperature and also using specimens having different size, shape, crack depth and loading type.

The effect of corrosion on fracture toughness in ductile to brittle transition region of 20MnMoNi55 steel is investigated, in this paper. To study the effect of corrosion in ductile to brittle transition (DBT) region corrosion exposure cycles of 2 hours are considered. The corrosion properties of the base material and corroded materials are determined by using electrochemical impedance spectroscopy test and compared. Now the master curve methodology is used to quantify the loss of ductility due to corrosion and effect of corrosion on fracture property. The fracture toughness tests have been carried out on the base material and corroded material and compared.

## 2. Methodology

The transition fracture toughness curve definition for ferritic steels, as specified in ASTM E1921-02 [9] was originally derived in 1991 from data measured on various quenched and tempered structural steel. Wallin showed that the brittle fracture probability ($P_f$) for a given temperature in the transition region can be described by a three parameter Weibull model in the form [10],

$$P_f = 1 - \exp\left[-\left(\frac{K_{JC} - K_{\min}}{K_0 - K_{\min}}\right)^4\right] \quad (1)$$

where $P_f$ is the probability of fracture at $K_{JC}$ for an arbitrary chosen specimen from a specimen set, $K_{JC}$ is the cleavage fracture toughness in MPa√m, $K_0$ is a scale parameter dependent on the test temperature and specimen thickness located at 63.2% cumulative failure probability level, and $K_{min}$ is the minimum possible fracture toughness. Wallin and International Atomic Energy Agency have suggested that $K_{min}$ should be assumed to be equal to 20 MPa√m for all the ferritic RPV material. The temperature dependence of the median fracture toughness in the transition region is given by equation (2) [11, 12].

$$K_{JC(median)} = 30 + 70\exp\left[0.019\left(T - T_0\right)\right] \quad (2)$$

where $T_0$ is master curve reference temperature in °C. At $T=T_0$, the median fracture toughness is 100 MPa√m. Once $T_0$ is known for a given material, the fracture toughness distribution can be obtained as a function of temperature. The measured $K_{JC}$ values should be checked to verify whether they fulfill the defined validity criterion. A $K_{JC}$ datum is invalid if the specimen size requirement is exceeded. In addition to size requirement there is the maximum ductile crack growth limit of $0.05(W–a_0)$ or 1 mm, whichever is smaller. $K_{JC}$ values beyond the validity criteria will be censored.

The value of $T_0$ is calculated after inclusion of all valid values according to the single or multi-temperature methods. For the single temperature method, multiple tests are done at the chosen test temperature. However multi-temperature method is more effective way than the single temperature method. The critical fracture toughness ($K_{JC}$) is determined from the $J_C$ value obtained from experiment using following equation.

$$K_{JC} = \sqrt{\frac{J_C \times E}{1-\nu^2}} \quad (3)$$

The statistical weakest link theory is used to consider the effect of specimen size on the probability of failure in the transition region. Now the measured $K_{JC}$ values are adjusted to a specimen size of 1T-CT specimen using the following equation.

$$K_{JC(1T)} = K_{\min} + \left[ K_{JC(x)} - K_{\min} \right] \left( \frac{b_0}{B_{1T}} \right)^{\frac{1}{4}} \quad (4)$$

where $b_0$ is the thickness of the tested specimen (side grooves are not considered), $B_{1T}$ is the thickness of 1T-CT specimen, $K_{JC(1T)}$ is the fracture toughness of equivalent 1T-CT specimen, $K_{JC(X)}$ is the fracture toughness of the tested specimen. The $K_{JC}$ values below 50 MPa√m are not size adjusted. Replicate fracture toughness tests at a constant temperature are performed to determine $T_0$. The ASTM standard recommends minimum 6 tests and these data are used to determine $K_0$ at the test temperature.

For single temperature evaluation, the determination of $T_0$ with $K_{JC}$ values is distributed over a restricted temperature range, namely, $T_0 = \pm 50°C$. For single temperature evaluation, the estimation of the scale parameter $K_0$, is performed according to the following equation.

$$K_0 = \left[ \sum_{i=1}^{N} \frac{\left( K_{JC(i)} - K_{\min} \right)^4}{N} \right]^{\frac{1}{4}} + K_{\min} \quad (5)$$

Here $K_{JC(i)}$ is the individual $K_{JC(1T)}$ value and $N$ is the number of $K_{JC}$ values. The term $N$ is replaced by the number of valid $K_{JC}$ values in the calculation. The fracture toughness for a median (50%) cumulative probability of fracture is estimated according to the following equation.

$$K_{JC(median)} = K_{\min} + \left[ K_0 - K_{\min} \right] (\ln)^{\frac{1}{4}} \quad (6)$$

Now the $K_{JC(median)}$ value determined at the test temperature is used to calculate $T_0$ by using the following equation.

$$T_0 = T - \left( \frac{1}{0.019} \right) \ln \left( \frac{K_{JC(median)} - 30}{70} \right) \quad (7)$$

In case of multi-temperature evaluation the reference temperature $T_0$ is directly determined. The value of $T_0$ is evaluated by an iterative solution of the following equation.

$$\sum_{i=1}^{N} \frac{\delta_i \exp\left[ 0.019 (T_i - T_0) \right]}{11 + 77 \exp\left[ 0.019 (T_i - T_0) \right]} - \sum_{i=1}^{N} \frac{\left( K_{JC(i)} - K_{\min} \right)^4 \exp\left[ 0.019 (T_i - T_0) \right]}{\left\{ 11 + 77 \exp\left[ 0.019 (T_i - T_0) \right] \right\}^5} = 0 \quad (8)$$

Here $T_i$ is the test temperature corresponding to $K_{JC(i)}$ and $\delta_i$ is the censoring parameter.

$\delta_i = 1$, if the $K_{JC(i)}$ datum is valid.

$= 0$, if the $K_{JC(i)}$ datum is not valid and censored.

Margins are usually added to cover the uncertainty in $T_0$ that is associated with the use of only a few specimens to establish this reference temperature.

## 3. Material and experimental details

The material investigated in this work is 20MnMoNi55 steel used for pressure vessel applications. The chemical composition of the material is given in table 1.

Table 1. Chemical composition of 20MnMoNi55 steel

| Name of element | C | Si | Mn | P | S | Al | Ni | Mo | Cr | Nb |
|---|---|---|---|---|---|---|---|---|---|---|
| Percentage composition (in wt.) | 0.20 | 0.24 | 1.38 | 0.011 | 0.005 | 0.068 | 0.52 | 0.30 | 0.06 | 0.032 |

To study the effect of corrosion on fracture toughness in ductile to brittle transition region, the standard tensile and ½T–CT specimen are exposed to corrosion in 3.5% NaCl solution. The specimens are kept in the solution for 2 hour in ambient temperature of 25 °C. After that, the electrochemical impedance spectroscopy test has been performed on base material and corroded specimen to estimate the corrosion properties of both the materials. The photograph of corrosion experiment setup is shown in Fig. 1.

Now standard tensile and *J*–integral tests have been performed using base material and corroded specimen sequentially in the temperature range –80 °C to –140 °C to measure fracture toughness of the base material and corroded specimen. The tensile and *J*–integral tests are done in the cryo–chamber attached to a computer controlled universal testing machine with 100 kN grip capacity. The required zero and sub-zero temperatures are attained by flowing liquid nitrogen. The tensile tests are performed at different temperatures on ASTM E8 [13] standard tensile specimen using Bluehill software. The *J*–integral tests are done on ASTM E399–90 [14] standard pre-cracked ½T–CT specimen according to ASTM E1820 [15] standard test procedure using $J_{IC}$ software. The photograph of *J*–integral experiment setup is shown in Fig. 2.

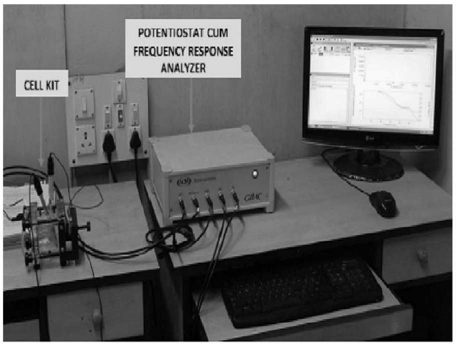


Fig.1. Experimental setup for corrosion test.

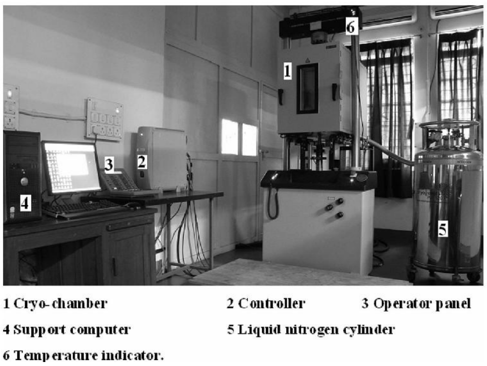


Fig.2. Experimental setup for $J_{IC}$ test.

## 4. Experimental results and discussion

### *4.1. EIS study*

The tensile and fracture toughness specimens are kept in the 3.5% NaCl solution for 2 hour at an ambient temperature of about 25 °C to find the instant effect of corrosion in master curve reference temperature. The

Nyquist plots and the polarization curves of base material and the corroded materials are obtained and shown in Fig. 3 (a) and (b) respectively.

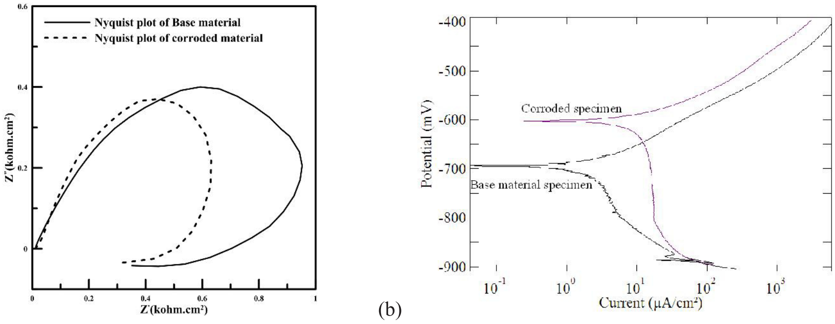


Fig. 3. (a) Nyquist plot of base and corroded materials and (b) Polarization curve of base and corroded material.

### *4.2. Tensile test results of base material and corroded specimen*

The variation of yield strength and ultimate tensile strength of base material and corroded specimen is shown in Fig. 4. It may be observed that both yield strength and ultimate tensile strength reduces due to corrosion. For all the cases with the decrease of test temperature both yield and ultimate tensile strength increases. The relations between yield and ultimate strength with test temperature (T, °C) for base material and corroded specimens are derived using the best fit curve.

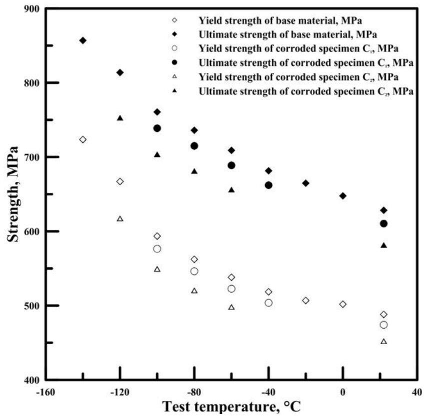


Fig. 4. Comparison of yield and ultimate strength values at different temperature for base material and corroded specimen.

### *4.3. J – integral tests results*

*J*–integral test is performed according to ASTM E399–09 standard on ½T–CT specimens of base material at different temperatures in the range between 22 °C to –140 °C and a/W ratio. Some specimens show an initial ductile stretch followed by brittle failure and some undergo direct brittle failure. Brittle fracture in the specimen has been observed at and below test temperature –80 °C [16]. The fracture toughness values for only those specimens which have undergone brittle failure have been considered for master curve. At –110 °C nine tests have been carried out using ½T–CT base material specimen and six tests have been carried using ½T–CT corroded specimen to determine the reference temperature using single temperature method. From the results it is observed that the experimental *J*–integral values are very scattered for both base material specimen and corroded specimen. All the experimental results of base material and corroded specimen using ½T–CT specimen at different temperatures are shown in Fig. 5.

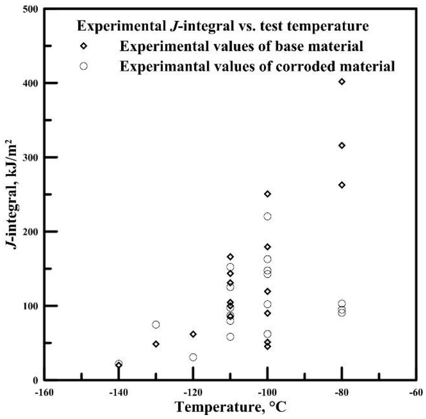


Fig. 5. Experimental *J* – integral values of base material and corroded specimen.

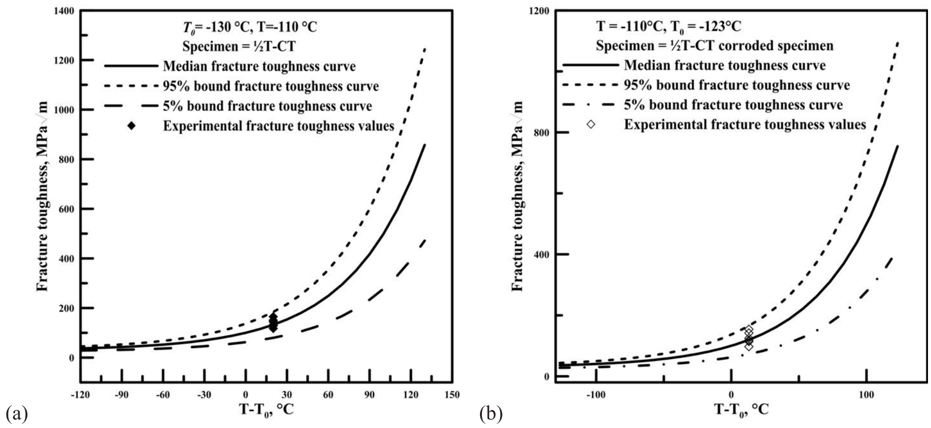


Fig. 6. Master curve at – 110 °C test temperature for (a) base material and (b) corroded specimen.

*4.4. Master curve analysis using single temperature method (–110 °C test temperature)*

Using ½T–CT specimen nine tests are done on base material specimen at –110 °C. The values of experimental fracture toughness vary from 85 to 166 MPa√m for ½T–CT specimen. The characteristic feature of these data reveals that both the upper bound and lower bound of fracture toughness increase with temperature as expected. The value of $K_0$, $K_{JC(median)}$ and $T_0$ for ½T-CT specimen are 143 MPa√m, 133 MPa√m and –130 °C respectively after the adjustment for size of specimen. The master curve at test temperature –110 °C for ½T–CT specimen is shown in Fig. 6(a). Similarly at –110 ºC six numbers of tests are done using corroded ½T–CT specimen. The value of experimental fracture toughness of corroded specimen is varying from 58.2 to 152.23 MPa√m. The value of $K_0$, $K_{JC(median)}$ and $T_0$ for corroded specimen are found to be 130 MPa√m, 120.31 MPa√m and 123 ºC respectively. The corresponding master curve is shown in Fig. 6(b). From the analysis it is observed that all the fracture toughness values of corroded specimen fall within 95% and 5% bound curve.

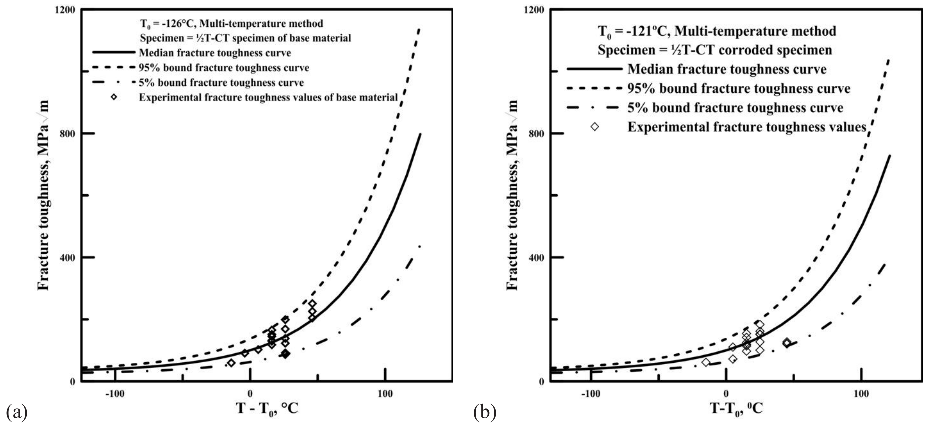


Fig. 7. Master curve using multi – temperature method for (a) base material and (b) corroded specimen.

*4.5. Master curve analysis using multi-temperature method*

The multi–temperature method for evaluating $T_0$ is more effective way than the single temperature evaluation as the scatterness and temperature dependency of the fracture toughness in DBT region is best reflected in the master curve developed using multi–temperature method. The value of $T_0$ using multi–temperature method is –126ºC for ½T–CT specimen of base material. The corresponding master curve is shown in Fig. 7(a). For the base material specimen the master curve reference temperature $T_0$ using multi–temperature method is found –129°C combining both 1T–CT and ½T–CT specimen [16]. The master curve derived from multi–temperature method combining both 1T–CT and ½T–CT specimen using multi–temperature method is considered as a reference curve to compare the embrittlement occurring due to corrosion in DBT region. The value of $T_0$ using multi–temperature method is –121 ºC for corroded ½T–CT specimen. From the result it is obtained that all the fracture toughness values fall within the 95% and 5% tolerance bound curve. The corresponding master curve is shown in Fig. 7(b).

*4.6. Comparative study between base material and corroded specimen*

Now the master curve derived from the corroded specimens are compared with the master curve derived from the base material. The master curve reference temperature $T_0$ for base material at test temperature–110 °C using

½T–CT specimen is found –130 °C. The master curve reference temperature $T_0$ for corroded specimen at test temperature –110 °C using ½T–CT specimen is found–123 °C. The master curve reference temperature using multi-temperature method combining 1T–CT and ½T–CT specimen are found as –129 °C. Now the master curve derived from corroded specimens are compared with the master curve derived at reference temperature –129 °C [16]. The corresponding comparison curve is shown in Fig. 8. From the result it is observed that the reference temperature shift is very less for this exposure time. Also it may be concluded that as the test temperature decreases the effect of corrosion becomes more prominent on the fracture property.

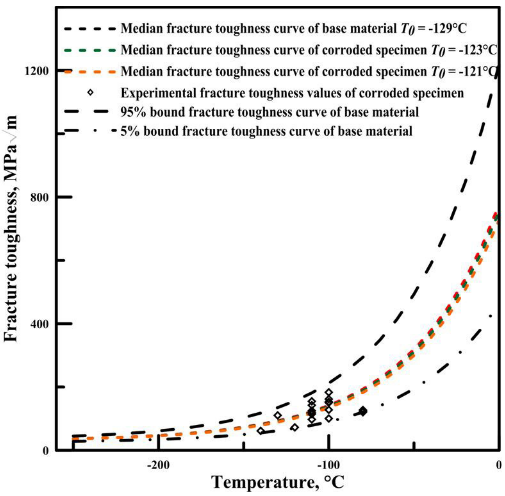


Fig. 8. Master curve comparison of corroded specimen.

## 5. Conclusion

In this work effect of corrosion on fracture toughness in DBT region of 20MnMoNi55 steel is investigated for 2 hours corrosion exposure time. The corrosion properties and fracture behavior of the material are measured and compared. From the above analysis it is observed that the corrosion resistance properties decrease with exposure to corrosive media. The fracture strength of the corroded specimen reduces due to corrosion compared to base material. The effect of corrosion in fracture toughness values in DBT region has been observed. The shift in master curve median fracture toughness curve due to corrosion is very less for this exposure time. The effect of corrosion on fracture toughness in DBT region as the test temperature is lowered. For this exposure time all the fracture toughness values and the median toughness curves fall within 95% upper bound and 5% lower bound of the base material specimen result.

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## References


[1] Seifert, H.P., Ritter, S., 2008. Stress Corrosion Cracking of Low-Alloy Reactor Pressure Vessel Steels under Boiling Water Reactor Conditions. Journal of Nuclear Materials, 372, p.114.
[2] Seifert, H.P., Ritter, S., 2008. Strain Induced Corrosion Cracking Behaviour of Low-Alloy Steels under Boiling Water Reactor Conditions. Journal of Nuclear Materials, 378, p. 312.
[3] Heldt, J., Seifert, H.P., 2001. Stress Corrosion Cracking of Low-Alloy, Reactor Pressure Vessel Steels in Oxygenated, High Temperature Water. Nuclear Engineering and Design, 206, p. 57.
[4] Lu, G., Zangari, G., 2002. Corrosion Resistance of Ternary Ni-P Based Alloys in Sulphuric acid Solutions, Electrochimica Acta, 47, p. 2969.
[5] Huang, H.H., Lee, T.H., 2005. Electrochemical Impedance Spectroscopy Study of Ti-6Al-4V Alloy in Aritificial Saliva with Fluoride and/ or Above Bovine Albumin, Dental Materials, 21, p. 749.
[6] Das, S.K., Sahoo, P., 2011. Electrochemical impedance Spectroscopy of Ni-B Coatings and Optimization by Taguchi Method and Grey Analysis, Portugaliae Electrochimica Acta, 29(4), p. 211.
[7] Wallin, K., 2010. Structural integrity assessment aspects of the Master Curve methodology, Engineering Fracture Mechanics, 77, p. 285.
[8] Hohe, J., Hebel, J., Friedmann, V., Siegele, D., 2007. Probabilistic Failure Assessment of Ferritic Steels Using the Master Curve Approach Including Constraint Effects, Engineering Fracture Mechanics, 74, p. 1274.
[9] ASTM E1921-02. Standard Test Method for Determination of Reference Temperature, $T_0$, for ferritic steel in the transition region, http://www.astm.org/DATABASE.CART/HISTORICAL/E1920.htm.
[10] Viehrig, H.W., Boehmert, J., Dzugan, J., 2002. Some Issues by Using the Master Curve Concept, Nuclear Engineering Design, 212, p. 115.
[11] Ferreño, D., Scibetta, M., Gorrochategui, I., Lacalle, R., Walle, E.V., Gutiérrez-Solana, F., 2009. Validation and Application of the Master Curve and Reconstitution Techniques to a Spanish Nuclear Vessel, Engineering Fracture Mechanics, 76, p. 2495.
[12] Serrano, M., Perosanz, F.J., Lapeña, J., 2000. Direct Measurement of Reactor Pressure Vessel Steels Fracture Toughness: Master Curve Concept and Instrumented Charpy-V test, International Journal of Pressure Vessels and Piping, 77, p. 605.
[13] ASTM E8/E8M-09. Standard Test Methods for Tension of Metallic Materials, http://www.astm.org/Standards/E8.htm.
[14] ASTM E399-90. Standard Test Method for Linear-Elastic Plastic Strain Fracture Toughness $J_{IC}$ of Metallic Materials, http://www.astm.org/Standards/E399.htm.
[15] ASTM E1820-09e1. Standard Test Method for Measurement of Fracture Toughness, http://www.astm.org/Standards/E1820.htm.
[16] Bhowmik, S., Sahoo, P., Acharyya, S.K., Chattopadhyay, J., and Dhar, S., 2012. Application and Comparative Study of Master Curve Methodology for Fracture Toughness Characterization of 20MnMoNi55 Steel. Materials and Design, 39, p. 309., Sept. 2009.