INTEGRATION OF STATISTICAL TECHNIQUES IN THE ANALYSIS OF EXPERIMENTAL FRACTURE MECHANICS DATA FOR THE CANDU PRESSURE TUBE, ZR-2.5%NB ALLOY

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Abstract

The experimental data obtained in the fracture mechanics tests on the Curved-Compact-Test (CCT) type specimens made from the CANDU pressure tube, Zr-2.5Nb alloy, requires special attention, due to the intrinsic character of variability. Tested material may have imperfections and inhomogeneities which can come from the manufacturing process, sample preparation, test conditions, etc. The *t*-Student distribution was integrated into the statistical analysis of the experimental data obtained during the fracture mechanics tests on CCT specimens. The fracture toughness values were represented by the two parameters obtained at maximum load: stress intensity factor, K_{ml}, and J-integral, J_{ml}. The parameters of interest values were obtained in the statistical analysis (variance, standard deviation, confidence interval, etc.) for Zr-2.5%Nb alloy, considering the 60-80 ppm hydrogen concentration range. The paper approach will be used for conservative assessments in the structural integrity analyses of CANDU pressure tubes.

Keywords: *Zr-2.5%Nb pressure tube, Fracture toughness, t-Student distribution, Statistical analysis*

Introduction

Understanding the characteristics of fracture toughness is essential for the in-service assessment of Zr-2.5% Nb pressure tubes as well as for initiating rupture and confirming the Leak-Before-Break (LBB) argument. This is especially true for comparatively high hydrogen concentrations and a range of hydride morphologies. The equivalent hydrogen concentration rises throughout the tube's volume, making this particularly crucial for tubes that have been running in the reactor for an extended period. The fracture toughness of Zr-2.5% Nb and its influence on hydrogen concentration and hydride morphology are generally poorly studied, particularly at high hydrogen concentrations and hydride morphology unique to the CANDU reactor's operating conditions [1-5].

To learn more and comprehend the fracture properties of Zr-2.5% Nb material, cold processed, irradiated and non-irradiated, based on the hydrogen concentration and hydride morphology, representative for reactor operating conditions, a comprehensive research program was needed [6-10]. The present paper studies are included in a research program dedicated to performing of fracture mechanics tests on Curved-Compact-Test (CCT)-type samples taken from pressure tube segments, Zr-2.5%Nb alloy, with high hydrogen concentrations. The fracture mechanics parameters of interest, namely the stress intensity factor, K_{ml}, and the J-integral, J_{ml}, were calculated at the maximum mechanical test load. Micro-structural and SEM analyses complement the study of fracture toughness on CCT samples at high hydrogen concentrations. The results are compared with those obtained in some Canadian studies and previous works.

Because the spread of the values obtained in such tests has an intrinsic variability, it is especially important to make appropriate use of the experimental data obtained in the fracture mechanics tests on the CCT- type specimens made from the CANDU pressure tube, Zr-2.5Nb alloy. This spreading of the experimental values characterizing the phenomena is present in all cases involving propagation of a break in a metallic material. This is due to the possibility that the tested material contains flaws and inhomogeneities resulting from the production process, sample preparation for the experiment, test settings, etc.

The statistical analysis of the experimental data collected on CCT specimens during the fracture mechanics testing incorporated the *t*-Student distribution. The two parameters obtained at maximum load - the stress intensity factor, K_{ml}, and the *J*-integral, J_{ml} - represented the fracture toughness values. This type of statistical analysis is appropriate for a limited set of experimental data, and it is used in the study to analyze the values acquired from three earlier studies.

The statistical analysis for the K_{ml} and J_{ml} parameters of the Zr-2.5%Nb alloy in the range of 65–82 ppm hydrogen concentration (variance, standard deviation, confidence interval, etc.) was carried out. Ultimately, a good agreement was established with the results obtained in the Canadian investigations for a similar concentration range. For cautious evaluations in the structural integrity analyses of CANDU pressure tubes, the paper approach will be applied. The fracture mechanics topic, within the Fuel Channel Research Program, is integrated within the activities assessing the structural integrity of the pressure tubes in Units 1 and 2 from Cernavodă Nuclear Power Plant.

Fracture mechanics tests on CCT samples

The characteristics and dimensions of the CCT sample used in the experimental program were similar to those used in [11]. Samples were taken from circumferentially sectioned rings on the pressure tube. In Fig. 1(a) the dimensions of the CCT sample are mentioned, Fig. 1(b) showing an aspect of the sample before testing. It should be noted that the plan dimensions of the CCT sample meet the proportionality requirements (in terms of relation to the width, W) of the standard flat Compact Tension (CT) sample, as specified in ASTM E1820 [12], but retain the original curvature of the pressure tube.

Due to limitations imposed by the original pressure tube material thickness (4.2 mm), thickness B does not meet the requirements of ASTM E1820 standard, which specifies for B a value between 0.25 W and 0.5 W (4.25-8.5 mm). However, if the original pressure tube thickness is used in the test, then the measured fracture toughness must be representative for the actual component. The design of the specimen used includes two knife edges for attaching a strain gauge to monitor the fatigue pre-crack, as can be seen in Fig. 1(b). The results from the model given in [1] are equivalent, for a whole range of fracture toughness values, to the results obtained on flat CT specimens according to ASTM, in the same size range. CCT samples were processed from pressure tube ring sections with various hydrogen concentrations. For the introduction of hydrogen into the CCT samples, a controlled hydrogenation of the pressure tube ring type samples was carried out by the method of electrolytic deposition of a hydrogen film layer, followed by an appropriate heat treatment, necessary for diffusion and homogenization of the hydrogen amount in the sample. This method, often used in specialized literature, especially in case of experiments on controlled hydration of the Zr-2.5%Nb alloy, was developed in the Metallography laboratory for the Zircaloy-4 alloy. Later on, the method was adapted and applied to the Zr-2.5%Nb alloy.

In order to perform the fracture mechanics tests, the steps described in [11] were followed after adapting for the CCT-type samples taken from the CANDU pressure tube, 65-82 ppm of hydrogen. The fracture mechanics tests on CCT samples were performed at room temperature.

The clamping of the CCT sample in the Walter+Bai traction machine, before the fatigue tests and the fatigue diagram are shown in Fig. 2.

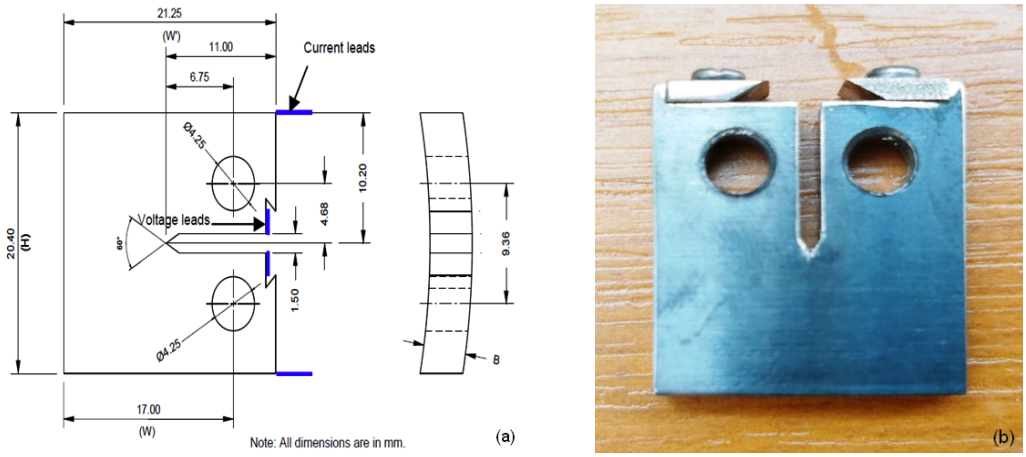


Fig. 1. CCT specimens: (a) dimensions, (b) aspect before testing

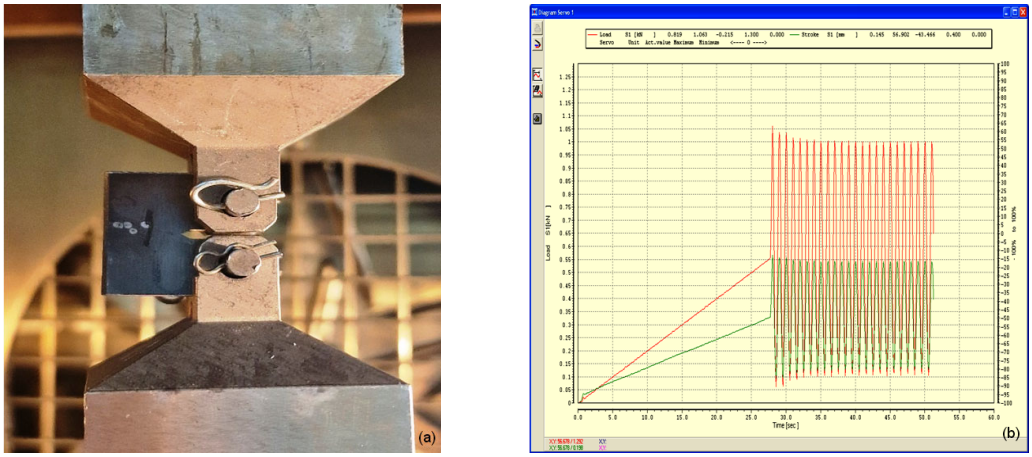


Fig. 2. CCT specimens: (a) ready for test, (b) fatigue diagram

From the fracture mechanics test sequence it was possible to determine the forces corresponding to the maximum stress at which the crack propagates to a value of about 2 mm. With the help of these values, the K_{ml} and J_{ml} values will be determined after the fatigue propagation and fracture mechanics crack size measurements.

To obtain the fracture toughness at current load, the following relationship is used [12]:

$$K_{(i)} = \frac{P_{(i)}}{(BB_N W)^{1/2}} f\left(\frac{a_i}{W}\right) \quad (1)$$

where: B = sample width; B_N = width of the sample with grooves; W = the length between the stress axis and the edge of the sample; a_i = current crack length, and

$$f\left(\frac{a_i}{W}\right) = \frac{\left(2+\frac{a_i}{W}\right)}{\left(1-\frac{a_i}{W}\right)^{3/2}}\left[0.886 + 4.64\left(\frac{a_i}{W}\right) - 13.32\left(\frac{a_i}{W}\right)^2 + 14.72\left(\frac{a_i}{W}\right)^3 - 5.6\left(\frac{a_i}{W}\right)^4\right] \quad (2)$$

For J-integral at maximum load, the relationship is used:

$$J_{ml} = \frac{K_{ml}^2}{E} \quad (3)$$

with E as the Young modulus.

Fig. 3 shows the aspects of the tested samples for optical and metallographic examinations.

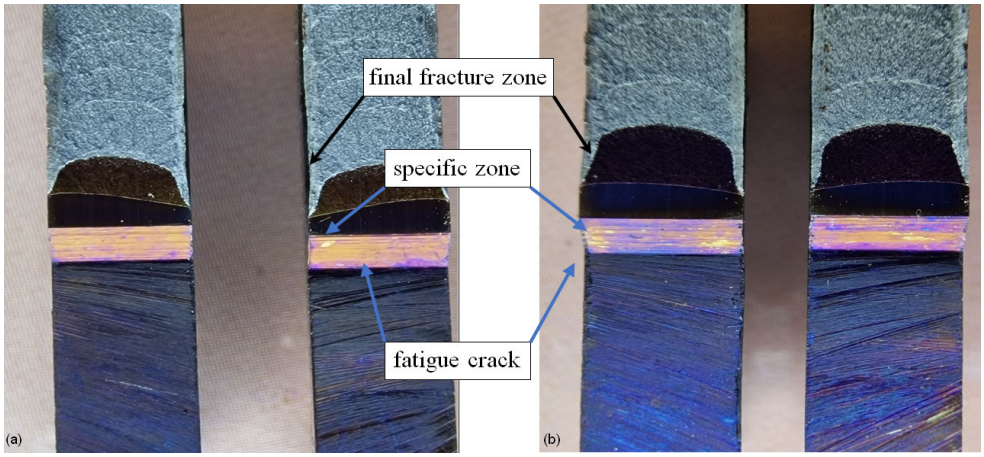


Fig. 3. Zr-2.5%Nb CCT specimens after fracture test:
(a) Sample A with 80 ppm hydrogen, (b) Sample B with 65 ppm hydrogen

In Figs. 3(a) and 2(b), the three zones in the crack plane can be noted, namely: the flat zone of the fatigue crack, the specific zone generated during the fracture mechanics test, and the final fracture zone after the test. For the fracture area, the specific "tunnelling" aspect is noted, as mentioned in the specialized literature. In this area, it is found that the crack advances more in the central area of the sample, and less at its edges. It can also be seen that the sample with 80 ppm is more fragile than the sample with 65 ppm hydrogen, which is evidenced by the size of the fracture zone during the actual test.

SEM analyses were carried out on the fracture surfaces of the CCT samples tested to obtain the fracture mechanics parameters K_{ml} and J_{ml} of the maximum load, highlighting the specific character of the hydrate Zr-2.5%Nb samples.

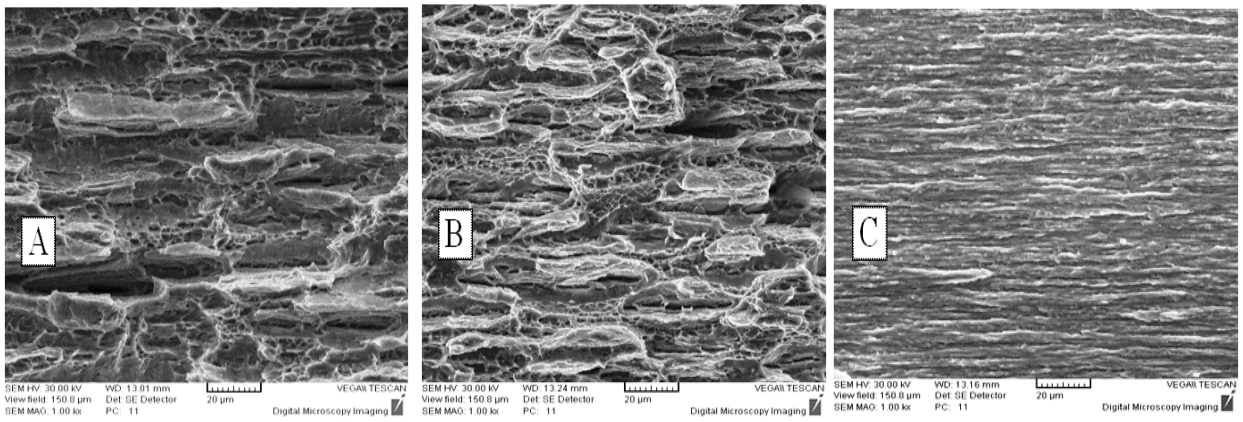


Fig. 4. SEM aspects of the three fracture surfaces of the CCT specimen

In Fig. 4, three surfaces were analyzed, namely:
- Area (A): The fatigue fracture surface, which was generated during the realization of the fatigue pre-crack;
- Area (B): The fracture surface generated during the fracture mechanics test, in the monotonically increasing application of the force up to the maximum value, after which the "pop-in" phenomenon is observed, a sudden decrease in force;
- Area (C): The fracture surface generated in the final fracture, after the colouring heat treatment of the fatigue (A) and fracture (B) surfaces, which arises in the normal fracture testing, to obtain the two sample fragments.

To obtain the fracture mechanics parameters K_{ml} and J_{ml} of the maximum load, the following quantities were used:
- thickness of the CCT sample: B = 4.0 mm;
- the distance between the line of mechanical stress and the edge of the sample: W = 17.00 mm;
- modulus of elasticity at room temperature, Zr-2.5%Nb: E =101.2 GPa.

Using relations (1), (2) and (3), as well as the optical measurement results, the values of the fracture mechanics parameters K_{ml} and J_{ml} were determined.

The Integration of Statistical Techniques in the Analysis of Experimental Fracture Mechanics Data

In order to perform the processing of the experimental data obtained in the fracture mechanics tests on the CCT samples, for the fracture toughness represented by the two parameters K_{ml} and J_{ml} we will use the values considering the 65-82 ppm hydrogen concentration range. These values characterize the mechanical behaviour specific to the last operating period designed for the CANDU pressure tube.

When the value of dispersion characterizing a population of experimental values is unknown, we can use its estimated value, s^2, based on the sample data. We consider a sample of volume n, taken from a normally distributed population $x1$, … x, …xn, with the mean and standard deviation parameters, μ and σ, unknown. Then the random variable:

$$t = \frac{x - \mu}{s/\sqrt{n}} \tag{4}$$

is distributed t-Student, and standard deviation parameters σ is replaced with sample standard deviation s.

A random variable t is t-Student distributed, with ν degrees of freedom, if the probability density function is of the form:

$$f_T(t) = \frac{\Gamma\left(\frac{\nu+1}{2}\right)}{\sqrt{\pi\cdot\nu}\cdot\Gamma\left(\frac{\nu}{2}\right)}\cdot\left(\frac{t^2}{\nu}+1\right)^{-\frac{\nu+1}{2}};-\infty<t<\infty,\nu>0 \tag{5}$$

$$\Gamma(x) = \int_0^\infty t^{k-1}\cdot e^{-t}\cdot dt \tag{6}$$

In various studies [13-16], the values of the quintiles of the *t*-Student distribution are calculated for different degrees of freedom.

The confidence interval, [μ_L, μ_U], containing the true value of the parameter μ, with a probability (1 - α), represents the solution of the equation:

$$\Pr(\mu_L \le \mu \le \mu_U) = 1-\alpha \tag{7}$$

Based on Eq. (4), Eq.(7) becomes:

$$\Pr\left(t_{\frac{\alpha}{2},n-1} \le \frac{\overline{X}-\mu}{s/\sqrt{n}} \le t_{1-\frac{\alpha}{2},n-1}\right) = 1-\alpha \tag{8}$$

Processing the previous equation yields the following relationship:

$$\Pr\left(\overline{x}+t_{\frac{\alpha}{2},n-1}\cdot s/\sqrt{n} \le \mu \le \overline{x}-t_{\frac{\alpha}{2},n-1}\cdot s/\sqrt{n}\right) = 1-\alpha \tag{9}$$

Making use of:

$$-t_{\frac{\alpha}{2},n-1} = t_{1-\frac{\alpha}{2},n-1} \tag{10}$$

the confidence interval [μ_L, μ_U] which contains bilaterally symmetrically the true value of the mean μ, with probability (1-α), becomes:

$$\overline{x}+t_{\frac{\alpha}{2},n-1}\cdot s/\sqrt{n} \le \mu \le \overline{x}-t_{\frac{\alpha}{2},n-1}\cdot s/\sqrt{n} \tag{11}$$

Next, we will work with sample dispersion:

$$s^2 = \frac{\sum_{i=1}^{n}(x_i-m)^2}{n-1} \tag{12}$$

where: m is the mean of the poll sample, n is the number of sampling values, and x_i represents the sample values.

Under these conditions, the sample standard deviation is given by the relationship below:

$$s = \sqrt{\frac{\sum_{i=1}^{n}(x_i-m)^2}{n-1}} \tag{13}$$

and the coefficient of variation will be:

$$CV = \frac{\sqrt{s}}{m} = \frac{\sqrt{\frac{\sum_{i=1}^{n}(x_i-m)^2}{n-1}}}{\frac{\sum_{i=1}^{n}x_i}{n}} \tag{14}$$

In the statistical analysis of the experimental data above mentioned, a confidence interval of: 1-α = 0.95 = 95% was used.

Table 1 displays the experimental data and statistics parameters for K_{ml} and J_{ml} obtained in the present study.

Table 1. Statistics for K_{ml} and J_{ml} parameters

K_{mli} $(MPa\cdot\sqrt{m})$	$\bar{K}_{ml}=\frac{\sum_{i=1}^{n}K_{mli}}{n}$ $(MPa\cdot\sqrt{m})$	$s=\sqrt{\frac{\sum_{i=1}^{n}(\bar{K}_{ml}-K_{mli})^2}{n-1}}$ $(MPa\cdot\sqrt{m})$
72.21, 40.31, 78.75, 53.41. 42.24, 53.24	56.7	15.7
J_{mli} (kJ/m^2)	$\bar{J}_{ml}=\frac{\sum_{i=1}^{n}J_{mli}}{n}$ (kJ/m^2)	$s=\sqrt{\frac{\sum_{i=1}^{n}(\bar{J}_{ml}-J_{mli})^2}{n-1}}$ (kJ/m^2)
46.81, 14.52, 55.76, 25.66, 16.04, 25.53	30.70	16.83

Statistics for K_{ml}

- Total number of data: $n = 6$ and for t-Student: $t = 2.571$

For a confidence interval of 95%, we get:

$$\frac{t\cdot s}{\sqrt{n}}=\frac{2.571\cdot 15.7}{\sqrt{6}}=\frac{40.29}{2.45}=16.47 \quad (15)$$

- Then the confidence interval, which contains bilaterally symmetrically the true value of the mean K_{ml}, with 95% probability, is:

$$K_{ml}=\bar{K}_{ml}\pm\frac{t\cdot s}{\sqrt{n}}=56.7\pm 16.47 \quad (16)$$

- The coefficient of variation is:

$$CV=\frac{\sqrt{s^2}}{m}=\frac{15.7}{56.7}=0.27 \quad (17)$$

Statistics for J_{ml}

- Total number of data: $n = 6$ and for t-Student: $t = 2.571$

For a confidence interval of 95%, we get:

$$\frac{t\cdot s}{\sqrt{n}}=\frac{2.571\cdot 16.83}{\sqrt{6}}=\frac{43.29}{2.45}=17.66 \quad (18)$$

- Then the confidence interval, which contains bilaterally symmetrically the true value of the mean J_{ml}, with 95% probability, is:

$$J_{ml}=\bar{J}_{ml}\pm\frac{t\cdot s}{\sqrt{n}}=30.7\pm 17.66 \quad (19)$$

- The coefficient of variation is:

$$CV=\frac{\sqrt{s^2}}{m}=\frac{16.83}{30.7}=0.54 \quad (20)$$

For comparison, the values obtained for the parameters K_{ml} and J_{ml} are graphically represented in Figs. 5 and 6, according to the test temperature, for all the as-received samples and the hydrated samples from the Canadian study [8].

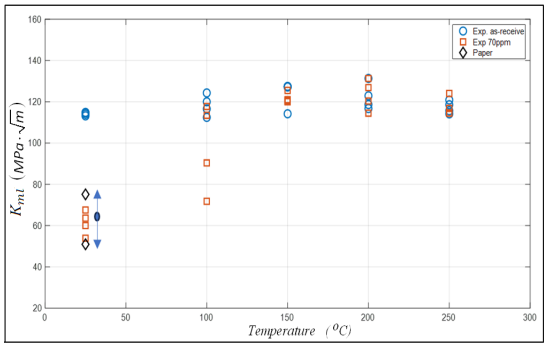


Fig. 5. Comparison of K_{ml} values in present paper and [8]

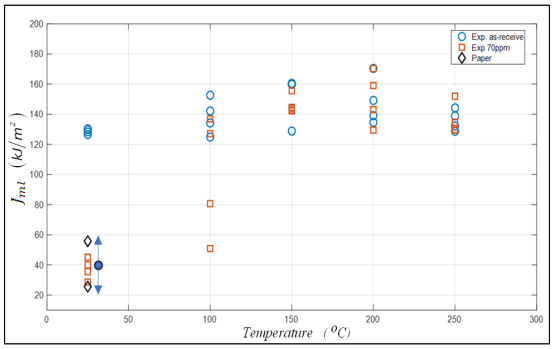


Fig. 6. Comparison of J_{ml} values in present paper and [8]

It can be noted that the values obtained in the statistical analysis for the K_{ml} and J_{ml} fracture mechanics parameters, in the concentration range of 65-82 ppm, are in full agreement with the values obtained in the Canadian studies, for the same concentration range.

Conclusions

The statistical analysis of the experimental data collected on CCT specimens during the fracture mechanics testing incorporated the t-Student distribution. The two parameters obtained at maximum load - the stress intensity factor, K_{ml}, and the J-integral, J_{ml} - represented the fracture toughness values. This type of statistical analysis is appropriate for a limited set of experimental data, and it is used in the study to analyze the values acquired from three earlier studies.

The following conclusions could be drawn:

- The room temperature testing methodology of CCT samples from Zr-2.5%Nb alloy, on the Walter + Bai installation, was improved to obtain the fracture mechanics parameters, namely the stress intensity factor, K_{ml}, and the J-integral, J_{ml}, parameters calculated at load maximum mechanical test, following ASTM E1820 standard.
- SEM analyzes highlighted the morphology of the hydrides in the tested samples as well as the character of the fracture surfaces, reflecting the influence of the hydride platelets on the mechanism of crack propagation.
- When processing the experimental data obtained during the fracture mechanics tests, performed on CCT samples, the t-Student distribution was used for the values of the fracture toughness represented by the two parameters, for statistical analysis; this statistical analysis is applied to a small number of experimental data.
- In the statistical analysis of the experimental data above mentioned, a confidence interval of 95% being used. It was found that the values obtained in the statistical analysis for the K_{ml}, and the J-integral, J_{ml}, parameters, in the 65-82 ppm concentration range, are in full agreement with the values obtained in the Canadian studies, for the same concentration range.

The obtained results will be used in the evaluation activities of the structural integrity of the pressure tubes in the CANDU nuclear power plant.

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