Investigation of the Fracture Mechanics Properties of Small Tubes from Oxide Dispersion Strengthened Steels

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The paper presents one part from the RATEN ICN contribution to the European FP7 MatISSE Project objectives, which is focused on the fracture mechanics properties of small tubes made from ODS steels (Oxide Dispersion Strengthened steels). The ODS tubes are foreseen as cladding tubes for gen IV reactors, and therefore the mechanical properties are very important for working in the most aggressive environment (irradiation and high temperatures) during the gen IV reactor operation. The fracture toughness, K_{IC}, could be obtained for tubes with small diameters by means of the PLT-type mechanical test (acronym for Pin-Loading Test). This kind of test is a non-standard (ASTM) mechanical test, and during last decade it is still worldwide under development. The specific specimens for PLT-tests has been prepared from ODS tubes, which were provided to the project by the CEA France, a partner in the FP 7 MatISSE, in two different compositions: Fe -9cr ODS and Fe -14 Cr ODS. The paper highlights the PLT experimental test methodology, starting with obtaining of the geometric function, description of the experimental set-up and results processing.

Keywords: Pin-Loading Test, ODS steels, K_{IC} parameter, geometric function

Thin-walled tubes with small diameter are used on a large scale in nuclear power plants. More often, these tubes are used as heat exchanger tubes, steam generator tubes and nuclear cladding tubes [1]. The operating conditions of these tubes are severe due to high temperatures and mechanical stresses combined with the effects of radiation on the material properties [2]. The cracking behavior of thin-walled tubes is an important feature for the structural integrity assessment during operation.

Given these considerations, to ensure the structural integrity of components which are on use in nuclear installations, it was necessary to know their resistance to the initiation and propagation of crack (fracture toughness). Because of their dimensions, it is not possible to prepare the standard fracture mechanics specimens for the classical ASTM tests. Therefore it is need to introduce the new methods for assessing the fracture material properties of tubes with thin walls [3]. One of these has been introduced by Grigoriev [4, 5], and is called Pin-Loading Test (PLT), that is described in the next.

Experimental part

Fracture mechanics parameter, K_{IC}, could be obtained by means of the Pin-Loading Test, and this method requires the existence of tube sample with a very sharp artificial mechanical defect [6]. In the case of PLT method, the tension is applied at one end of the sample in comparison with an axial tensile test on the same sample type [7-9]. The advantage is that the tension required for the deformation of the sample and the elastic deformation of the gripping system are lower than the uniaxial tensile test. Therefore, the elongation of the sample is bigger and the recorded data is enough to characterize the material in contrast with the uniaxial tensile test, where the elastic deformation of the gripping system has a major influence, especially for materials with low ductility, causing rupture of the sample at extremely small elongation, and the recorded data is insufficient to characterize the material (fig.1) [10-12].

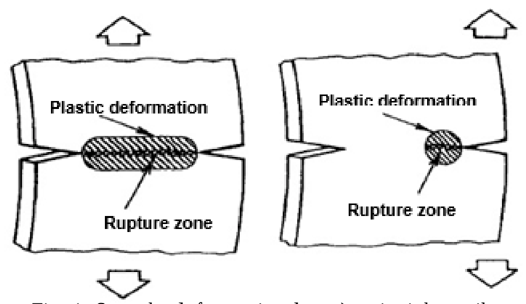

Fig. 1. Sample deformation by: a) uniaxial tensile test, b) PLT test

The material used for the PLT tests was distributed by CEA France within the framework of European FP7 MatISSE Project and consists of ODS steel tubes in two different compositions: Fe-9Cr ODS and Fe-14Cr ODS. The dimensional characteristics are:

-inner diameter: 9.70 mm

-wall thickness: 0.60 mm;

The PLT type specimens were obtained by machining processes that included cutting, milling and electro-erosion. Figure 2 shows the sketch of PLT sample and the figure 3 presents the samples used for testing.

At each end of the sample were performed two diametrically opposed clefts. The clefts from the bottom part of the sample have a depth of 2 mm and a width of 0.5 mm, and their role is a technological one [13-15]. The clefts

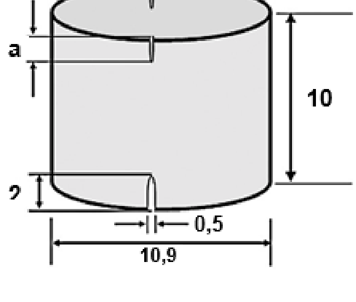

Fig. 2. PLT sample dimensions in mm (crack length a =1.5 mm; 3 mm; 5 mm)

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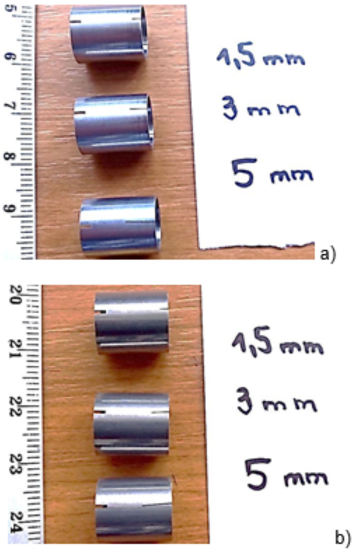

Fig. 3. PLT samples for before testing: a) Fe-9Cr ODS; b) Fe-14Cr ODS

from the top of the sample are very important because from these clefts the fracture initiate and its propagating until the sample break off. The sizes of these clefts are variable regarding to the depth, from 1.5 mm to 5 mm. The width is kept constant - about 0.15 mm.

For both compositions have been performed two sets of samples. The first set includes three samples with different lengths of test clefts (1.5 mm, 3 mm and 5 mm). These samples are used to obtain the specimen compliance. The second set comprises three samples with the same length of test clefts - 1.5 mm and are used to obtain K_{IC} from the PLT tests. The results recorded from the PLT tests are used to calculate the fracture toughness, K_{IC}, of the material.

The following relationship is used to obtain K_{IC}:

$$K_{IC} = \left[\frac{P_Q}{2t\sqrt{W}}\right] \cdot f\left(\frac{a}{W}\right) \quad (1)$$

Here:

w = distance between the load application point and the technological crack tip (fig.4)

t = wall thickness of the tube;

P_Q = applied load;

$f(a/W)$ = geometric function.

One may see, that the fracture toughness determination is subject to the determination of the applied load P_Q and geometric function $f(a/W)$. Geometric function is obtained from measurements of compliance.

Obtaining geometric function

To determine compliance, three mechanical tests were conducted for each from two kinds of materials. For this it have been used the samples with different lengths of the test clefts (1.5, 3 and 5 mm). From the experimental curve, the compliance was calculated for each sample with the equation (2).The results for samples of Fe-9Cr ODS are provided in table 1.

$$C = \frac{\Delta x}{\Delta P} \quad (2)$$

Figure 5 shows an example of calculating compliance for Fe-9Cr ODS sample with 3 mm test cleft length.

Table 1

DETERMINATION OF COMPLIANCE FOR STEEL Fe-9Cr ODS

Material	Test cleft length (mm)	Compliance (10^{-6}m/N)
Fe-9Cr ODS	1.5	0.54
	3	0.64
	5	1.14

$$C_3 = \frac{\Delta x}{\Delta P} = \frac{0{,}48mm}{750N} = 0{,}64 \cdot 10^{-3}\,\frac{mm}{N} = 0{,}64 \cdot 10^{-6}\,\frac{m}{N} \quad (3)$$

In order to perform curve fitting the following points for are chosen:

$$\left[\frac{a}{W}\right] = \left[\frac{a_1}{W}, \frac{a_2}{W}, \frac{a_3}{W}\right] \quad (4)$$

where:

$$\begin{cases} a_1 = a_{01} + b \\ a_2 = a_{02} + b \\ a_3 = a_{03} + b \end{cases} \text{but} \begin{cases} a_{01} = 1{,}5mm \\ a_{02} = 3mm \\ a_{03} = 5mm \end{cases}$$

$$\text{and } b = 7{,}5mm \Rightarrow \begin{cases} a_1 = 9mm \\ a_2 = 10{,}5mm \\ a_3 = 12{,}5mm \end{cases} \quad (5)$$

With relation (5) and W=18.5mm, the relation (4) becomes:

$$\left[\frac{a}{W}\right] = \left[\frac{9}{18{,}5}, \frac{10{,}5}{18{,}5}, \frac{12{,}5}{18{,}5}\right] = [0{,}4865; 0{,}5675; 0{,}6757] \quad (6)$$

In a first step an exponential fitting is performed on compliance parameters [C]=[C1,5, C3, C5] = [0.54; 0.64; 1.14] and [a/W]=[0.4865; 0.5675; 0.6757]. In a second step a 6th order polynomial fitting is performed on the output of the exponential fitting and the result is the following compliance relationships:

$$C\left(\frac{a}{w}\right) = 10^{-6}\left[0.3105 \cdot \left(\frac{a}{w}\right)^6 - 0.2238 \cdot \left(\frac{a}{w}\right)^5 + 0.6729 \cdot \left(\frac{a}{w}\right)^4 + 0.3671 \cdot \left(\frac{a}{w}\right)^3 + 0.6174 \cdot \left(\frac{a}{w}\right)^2 + 0.4357 \cdot \left(\frac{a}{w}\right) + 0.1663\right] \quad (7)$$

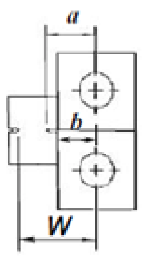

Fig. 4. Sample - gripping system assembly

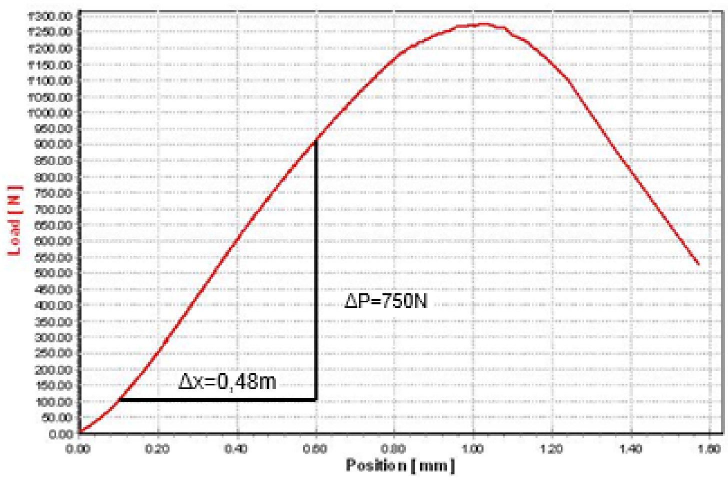

Fig. 5. Determination of compliance for Fe-9Cr ODS specimen with 3 mm test cleft length

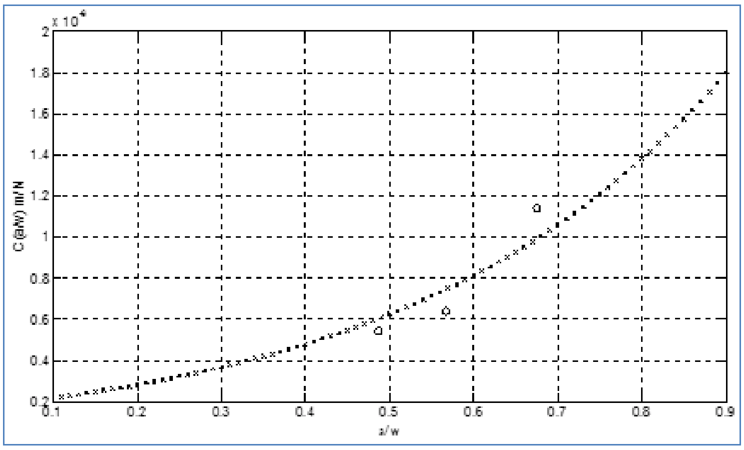

Fig. 6. Polynomial fitting of compliance forFe-9Cr ODS

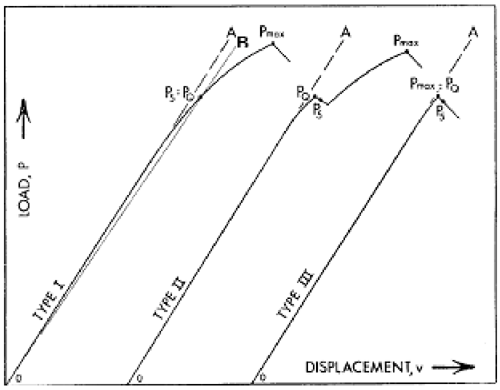

Fig. 7. Types of load – displacement curves

Further on the derived compliance is calculated and the relation is:

$$\frac{dC}{d\left(\frac{a}{w}\right)}=f_2\left(\frac{a}{w}\right)=10^{-6}\cdot\left[1.863\cdot\left(\frac{a}{w}\right)^5-1.119\cdot\left(\frac{a}{w}\right)^4+2.6916\cdot\left(\frac{a}{w}\right)^3+1.1013\cdot\left(\frac{a}{w}\right)^2+1.2348\cdot\left(\frac{a}{w}\right)+0.4357\right] \quad (8)$$

Thus, the geometric function is given by the relation:

$$f\left(\frac{a}{W}\right)=\left[t\cdot E\cdot f_2\left(\frac{a}{W}\right)\right]^{\frac{1}{2}} \quad (9)$$

Here:
t=0.6 mm;
E=200 GPa - Young modulus [16].
By putting equation (8) into equation (9) the final form of the geometric function for Fe-9Cr ODS specimen is:

$$f\left(\frac{a}{w}\right)=0.7946\cdot\left(\frac{a}{w}\right)^5-0.2936\cdot\left(\frac{a}{w}\right)^4+4.0012\cdot\left(\frac{a}{w}\right)^3+5.8040\cdot\left(\frac{a}{w}\right)^2+9.7427\cdot\left(\frac{a}{w}\right)+7.2554 \quad (10)$$

The fracture toughness K_{IC} from PLT

The fracture toughness could be obtained with equation (11):

$$K_{IC}=\left[\frac{P_Q}{2t\sqrt{W}}\right]\cdot f\left(\frac{a}{W}\right), \quad (11)$$

and here the values of the factors *a*, *t* and *w* are known. Moreover, the P_Q load values are obtained by analyzing experimental curves for the PLT tests as recommended by ASTM (fig.7) [17-19].

The steps are the following:

-By origin, goes on a secant OA which is tangent to the elastic deformation zone;

-Another secant OB determines the formation of an origin angle which is representing 95% of OA origin angle;

-The intersection between these two secants and the experimental curve determines the corresponding point for the P_5 load [20].

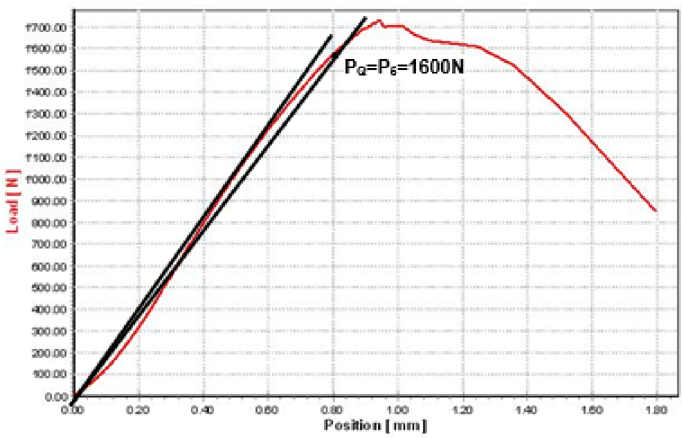

Fig. 8. P_Q load determination for „Fe-9Cr ODS – test1" sample

There are two typical situations for the determination of P_Q:

-If the points on the experimental curve which are preceding the P_5 point, have lower load values than the value corresponding to P_5 point it is considered that $P_5=P_Q$ (type I in fig. 7);

-If there is a point on the experimental curve graph preceding P_5 point where for the value of the load is higher than the load value for P_5 point, then that point will be P_Q (type II and III in fig. 7).

Figure 8 shows how the P_Q was determinate by using the tensile test curve from current test.

Results and discussions

Three samples were tested to obtain P_Q values, and these values were introduced in equation (11) to obtain the K_{IC} values of fracture toughness. The results for Fe-9Cr ODS are shown in table 2.

Table 2
VALUES of K_{IC} FOR Fe-9Cr ODS STEEL

Sample cod	P_Q (N)	K_{IC} (MPa.m$^{1/2}$)
Fe-9Cr_test1	1600	135.6
Fe-9Cr_test2	1710	144.9
Fe-9Cr_test3	1610	136.5

Table 3
VALUES OF K_{IC} FOR Fe-14Cr ODS STEEL

Sample cod	P_Q (N)	K_{IC} (MPam$^{1/2}$)
Fe-14Cr_test1	1333	122.2
Fe-14Cr_test2	1330	121.9
Fe-14Cr_test3	1336	122.5

The mean value of the toughness K_{IC} that was obtained from our tests is of 139 $MPa.m^{1/2}$. This value is in a good agreement with the mentions from MatISSE Plenary Meeting in 2017, where it was concluded that the K_{IC} values must be found within the range of 80 -180 $MPa.m^{1/2}$.

A similar methodology has been used to obtain the fracture toughness values for Fe-14Cr ODS samples and table 3 presents the results.

One may see that for the Fe-14Cr ODS steel, the average value of the fracture toughness obtained by testing (122 $MPa.m^{1/2}$) is also in good agreement with the remarks from the MatISSE Project (80 -180 $MPa.m^{1/2}$).

Conclusions

This paper outlines the non-standard test methodology to obtain the fracture toughness for ODS tubes with small diameter, and the results could be used in the structural analyses for cladding tubes in gen IV reactors. The paper highlights the Pin-Loading Test (PLT) experimental test methodology, description of the geometric function obtaining and the results processing as well, in order to obtain the fracture toughness for ODS tubes.

A part of the work has been performed inside of the contribution of RATEN ICN in the framework of activities conducted in the European FP7 Matisse Project (2014-2017). The mean values obtained in the paper work for K_{IC} are: 139 $MPa.m^{1/2}$ for Fe-9Cr ODS and 122 $MPa.m^{1/2}$ for Fe-14Cr ODS. These values are in a good agreement with the range values mentioned by the MatISSE project participants (80 – 180 $MPa.m^{1/2}$).

The non-standard PLT testing methodology is under development and improvement, in order to be applied to other candidate structural materials used in generation IV reactors.

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