TECHNICAL SPECIFICATION

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Thermoplastics pipes for the conveyance of fluids — Determination of the slow cracking resistance of pipes and fittings using the Notched Ring Test (NRT)

Tubes en matières thermoplastiques pour le transport des fluides — Détermination de la résistance des tubes et raccords à la propagation lente de la fissure au moyen d'essais sur sections fendues



Reference number ISO/TS 16479:2012(E)

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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of document:

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Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TS 16479 was prepared by Technical Committee ISO/TC 138, *Plastics pipes, fittings and valves for the transport of fluids*, Subcommittee SC 5, *General properties of pipes, fittings and valves of plastic materials and their accessories* — *Test methods and basic specifications*.

Introduction

At the present time ISO/TC 138 provides several test methods for determining the resistance to slow cracking which are essential for assessing properties and durability of thermoplastics pipes and fittings. In fact, they constitute a basis for determining the long term performance characteristics of these parts. In line with the development of better slow crack resistant materials, new accelerated test methods for evaluating slow crack resistance of thermoplastic piping parts are frequently under investigation [1-3]. In consideration of this, the Notched Ring Test (NRT) method has been developed and applied to determine its applicability as the standard test method and has demonstrated its effectiveness in generating slow cracking within the time frame of interest [4-7]. This Technical Specification provides details of the NRT method in terms of test implementation, its SCG performance evaluation, and the application of the method utilizing the stress intensity factor approach.

Thermoplastics pipes for the conveyance of fluids — Determination of the slow cracking resistance of pipes and fittings using the Notched Ring Test (NRT)

1 Scope

This Technical Specification determines the slow cracking resistance of pipes and fittings using the Notched Ring Test (NRT) which is a method for determining the slow crack resistance of thermoplastics resins and compounds in pipe form and/or finished products (e.g. pipes and fittings) from which a ring specimen can be cut and notched. The test is performed under sustained constant loading in a test medium at a specified temperature and the time to the on-set of slow cracking is measured. This method applies to the rings from pipes or fittings having a wall thickness greater than 5 mm. This Technical Specification specifies the method of testing using notched ring specimens directly obtained from pipes or fittings.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 6108, Double equal angle cutters with plain bore and key drive

ISO 13479, Polyolefin pipes for the conveyance of fluids — Determination of resistance to crack propagation — Test method for slow crack growth on notched pipes (notch test)

3 Terms and definitions

3.1

slow crack growth

SCG

crack growth that occurs below the critical stress intensity factor at which the unstable rapid crack propagation takes place

NOTE The crack growth is stable and hence can grow to a substantial length before causing material to fail either by rapid fracture or ligament yielding. In some polyolefin materials, distinct fracture surface features characteristic of slow crack growth are apparent and are often used as an indicator of slow crack growth

3.2

ring deflection curve

time dependent deflection curve obtained from the sustained constant flexure loading of the ring specimen

NOTE 1 See Annex A.

NOTE 2 The curve may have similar features of a creep curve, normally consisting of primary, steady state secondary and tertiary regions. Deflection is normally taken at the notch position and directly above the line of load application.

3.3

on-set slow cracking time

*t*_i (h)

time for first noticeable abnormal change to appear on the recorded ring deflection curve

NOTE 1 See Annex A.

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For this definition to be complete, the fracture surface must show features of slow cracking in any degree or features defined otherwise.

EXAMPLE Examples of abnormal change are discontinuity, slope change etc.

3.4 applied load L(N)

load applied in three point flexure mode

3.5

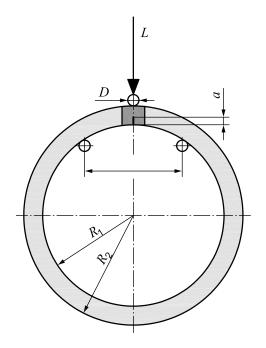
applied stress intensity factor

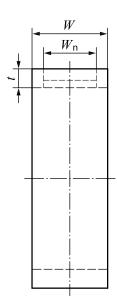
 $K_0 (N/m^{3/2})$

crack-tip stress intensity factor calculated based on the initial notch dimension

Principle

A suitable NRT specimen is an undisturbed full ring of certain width directly removed from the pipes or fittings (see Figure 1), having features that include an inner wall notch along the sample width between the tips of two coplanar sidewall notches (see Figure 5). The inner wall and sidewall notches are all in the same plane and the notch and specimen dimensions are such that conditions for the planestrain slow crack growth are met at the notch tip. Coplanar sidewall notches are introduced to further maintain the plane strain condition as well as to act to guide the loading rod accurately along the line of the crack tip. NRT specimens are subjected to sustained and constant flexure load in a temperature controlled medium and the time to on-set cracking as defined in 3.3 is determined from the measured ring deflection curve (see 3.2 and Annex A). In the NRT specimen, the slow cracking can be initiated in a relatively shorter time due to the possibility of constrained crack tip deformation induced by the sample and the loading geometry, hence allowing higher stress intensity to develop at the crack tip without gross yielding.





Kev

- *W* width of the ring
- W_n width between sidewall notches
- R₁ inside radius
- R₂ outside radius
- t wall thickness

- S span length
- a razor notch depth
- L applied load
- D diameter of loading and supporting rods

Figure 1 — The diagram and loading configuration of NRT specimen

5 Apparatus

5.1 Loading apparatus

A suitable device for loading the ring specimen in a sustained and constant manner under flexure mode shall be used. The device may utilize dead weights, pneumatically actuated loading or any other means for producing a sustained constant load with accuracy better than $\pm 1,0$ % of the applied load. The loading, supporting and guiding rods (see Figure 1) are made from a 316 stainless steel having the same ground surface and they are positioned parallel to each other. The guiding rod guides the loading rod right on top of the inner wall notch by means of adapting the sidewall notches (see Figures 1 and 5). An impression of a typical loading jig is shown in Figure 2.

5.2 Ring deflection measuring device

Ring deflection of the mid-span containing the plane of the crack front (see Figure 1) shall be measured by means of a suitable device that will not influence the specimen in any way. Particular caution should be practiced to ensure the stability of the measuring device with time, temperature and humidity. The device shall be capable of deflection measurement with accuracy better than $\pm 1,0\%$ and a resolution of 0,01 mm. Since it is important to continuously monitor the deflection, it is recommended to use an automatic deflection measuring system. An example of an electronic displacement gauge and its assembly to an overall loading-measuring device is shown in Annex A. The measurement shall not be influenced by any mechanical and/or electric disturbances that may end up in the deflection curve being obtained.

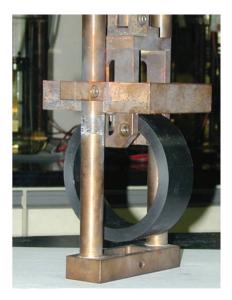


Figure 2 — Flexure apparatus for loading notched ring specimen

Temperature chamber 5.3

A suitable chamber should contain the environment needed for this test and ensure complete immersion of the specimen. The construction material of this chamber shall not affect the environment or vice versa. The chamber can usually be closed with a lid to prevent medium evaporation as well as to protect the loading-measuring apparatus. The temperature of the environment is controlled to maintain the specimens within ±1,0 °C of the specified test temperature. Replenishment of the lost medium due to evaporation is achieved best through the use of an auxiliary container using gravity feeding to minimize temperature variation during feeding.

5.4 Test medium

The tests are usually performed in water, unless stated otherwise.

5.5 Timing device

A suitable timing device that can monitor time accumulation shall be used. The accuracy of the measurement is recommended to be better than ±1 % of the elapsed time.

5.6 Microscope

A travelling microscope or an equivalent device is used to measure the initial razor notch length accurately after the slow crack test. The recommended resolution for this method is $\pm 100~\mu m$.

A combination of digital camera and image processing software is considered a readily available solution. This method can provide a razor notch depth measurement with required accuracy and convenience.

5.7 Notching apparatus

5.7.1 Inner wall notching device

The device shall be capable of producing a sharp notch at the inner wall of the ring specimen at a cutting speed of less than $300\mu m$ per minute.

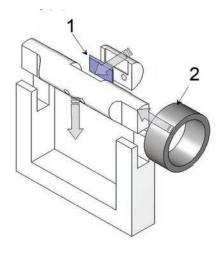
NOTE Such cutting speeds are generally available in a typical universal testing machine and the load-displacement curve can be readily obtained simultaneously by installing the notching device on such machines [see Figure 3b)]. Higher notching speed is known to create abnormality at the notch tip which can affect the slow crack initiation.

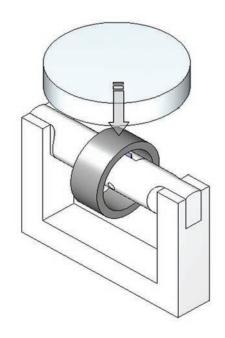
It is recommended that the device utilized is also able to produce load-razor blade displacement curve during notching for the purpose of checking consistency and uniformity of notching. The notch tip radius produced depends on the type of razor blade (blade thickness not exceeding 0,2 mm) and shall be less than $10~\mu m$. A broaching device is also acceptable provided that the notch tip radius is also less than $10~\mu m$.

Figure 3 shows an inner wall notching device. It consists of a removable arm for accommodating the razor blade holder, depth adjusting parts and the ring specimen, a base to hold the arm perpendicular to the compression load and a mechanism for firmly holding the base to the universal testing machine.

5.7.2 Sidewall notching device

A suitable device capable of machining a 90° angle "V"-notch at each sidewall of the ring specimen opposite each other and the notch tips in the same plane (see Figure 1) shall be used. The device should provide a notching rate of 0.010 ± 0.002 (mm/rev)/tooth (see ISO 13479 for details) and the cutter subject to a pre-treatment amounting to 10 m of notching prior to its first use. It is strongly recommended to use the cutter solely for this purpose and only on this material, to be replaced after 100 m of notching. The 90°-included-angle "V"-cutter employed shall conform to ISO 6108.





a)



b) Notching on a universal testing machine

Key

- razor blade
- notched ring specimen

Figure 3 — Inner wall notching device

14

6 Test specimen

6.1 Specimen geometry and dimensions

The specimen configuration and the loading geometry of the NRT specimen is illustrated in Figure 1 and their dimensions are shown in Table 1 for 63, 110, 250 and 400 mm nominal diameter SDR 11 ring specimens. Other ring dimensions may also be used. The sidewall notches shall be made parallel to the pipe axis and the angle between the surfaces in the V-notch is 90° . The depth of the inner wall notch is 30% of the sample wall thickness.

SDR 11 ^a	63 ^a	110	250	400		
Wb	> 35		> 50	> 50		
MNc	> 25 ± 0.2					
S	25	50	125	200		

12

Table 1 — NRT specimen dimensions in mm

 $0.3 \times e$

6.0

a d

D

6.2 Specimen preparation

The NRT specimen may be prepared by the process of direct ring removal from a pipe or a fitting. This ring is dimensioned to size after which the sidewall notching takes place and finally the inner wall notching. At every stage of preparation, the method employed is designed to minimize ring deformation and/or excessive heating of the specimen.

6.2.1 Ring removal and dimensioning

The test specimens are cut from a pipe or a fitting using a suitable method (e.g. band saw cutting) and are machined to a final width dimension (W) within ± 0.2 mm tolerance (e.g. lathe machining). In both cases the method utilized shall produce minimum deformation of the removed ring in addition to having two sidewalls produced parallel to each other. It is useful to have a ring holder (see Figure 4) which would grip the ring from its circumference, as this would cause the ring specimen axis to position themselves parallel to the axis of the holder as well as causing minimum deformation of the ring. When one sidewall of the ring specimen is machined on the holder this wall surface becomes perpendicular to the ring specimen axis. By removing the ring and reinserting the machined sidewall backed against the holder the second side wall being machined is ensured to be parallel to the first sidewall produced. During preparation, the specimen should not be subjected to excessive heating or to cooling or lubricating agents.

6.2.2 Specimen notching

The specimens are notched (inner wall and sidewall notches) at room temperature (23 ± 2 °C). Care must be exercised to avoid notch tip damage such as blunting and introduction of residual stresses during notching (e.g. excessive speed/force) as this will compromise the results. In order to obtain accurate results with NRT specimens, all three notches need to be in the same plane.

Other dimensions can also be used.

b Dimension chosen shall be at least WN + D.

c The crack tip stress intensity factor value varies with WN.

d e = actual wall thickness.



Figure 4 — Secondary machining on lathe to obtain final ring width dimension. The holder is used to conveniently obtain parallel sidewall surfaces as well as to cause minimum ring deformation during machining

6.2.2.1 Sidewall notching

When using a 90° "V"-notch cutter, it is recommended that a cutting rate of 0.010 ± 0.002 (mm/rev)/ tooth be utilized. The sidewall notches are cut parallel to the ring axis and the notch tips are in the same plane through the thickness of the specimen (see Figure 5). It is noted that while the speed of notching is not critical, it is important to keep co-planarity of the two machined sidewall notches. It has been found that this can be best done by using a method where both notches are made without disturbing the specimen during notching.



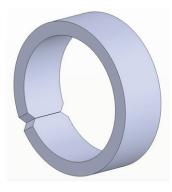
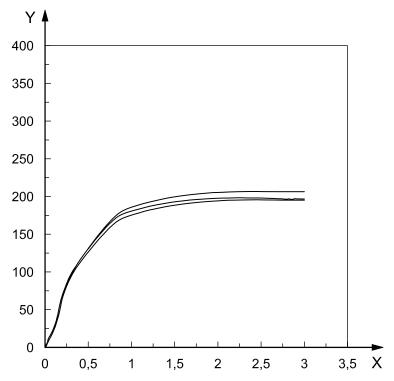


Figure 5 — Sidewall and inner wall notches

6.2.2.2 Inner wall notching

For razor blade notching the inner wall notch is made by pressing the blade at the inner wall surface at a speed of less than 300 μ m per minute. The notch covers the distance between the tips of two coplanar sidewall notches (see Figure 5) and shall be radially directed toward the outer surface to a specified depth [see Figure 3b]] indicated in Table 1. Accuracy of the notch depth produced is recommended to be ±0,1 mm and it is most important that the inner wall notch produced is coplanar with the two sidewall notches. It is recommended to record the load-razor blade displacement trace during notching and a typical load-displacement curve is illustrated in Figure 6. In practice, the razor blade is replaced after three uses (or when noticeable deviation occurs in the load-razor blade displacement trace) and shall not be used for any other materials or purposes.



Key

X displacement (mm)

Y load (N)

Figure 6 — Typical load-razor blade displacement curves obtained during inner wall notching of 110SDR11 NRT specimens. Three curves are from three separate notchings using the same razor blade

6.3 Specimen conditioning

Newly machined specimens shall be stored at (23 ± 2) °C and although the length of time between final notching and testing is not important it is recommended to precondition for a minimum of 24 h at room temperature prior to testing.

7 Procedure

7.1 Conditioning

Specimens shall be conditioned at the test temperature before applying the constant and sustained load. The samples shall be cleaned and dried to remove any traces of dirt, oil, wax or other contamination prior to measuring of the dimensions and temperature conditioning. In the liquid bath (or oven) where the test is to be performed, the specimens shall be allowed to condition for a minimum of 24 h for ring specimens of wall thickness up to 25 mm and 48 h for greater wall thickness before loading (it is convenient to condition the ring specimen as positioned in the loading device).

7.2 Load application

After conditioning at the test temperature, with the specimen completely immersed in the test medium, the load shall be applied to the specimen gradually and progressively, so as to avoid shock loading that

may blunt the notch as well as cause other damages. The load application shall be achieved within 60 s, without exceeding the required load. The applied and sustained load shall be controlled to an accuracy of better than ±1,0 % of the magnitude. Since it is important that the flexure load is applied exactly and uniformly on top of the inner wall notch front, sidewall notches are very useful means of achieving this requirement. Also, they work to conveniently hold the ring specimen in position during conditioning and testing. It is noted that the load magnitude applied shall be low enough to cause slow cracking and large enough to shorten the test duration time in the case of quality control testing.

7.3 On-set slow cracking time detection

Together with the sustained load application, the flexure displacement gauge and timing device shall be activated. The first noticeable abnormality to appear on the ring deflection curve (see Figures A.2 and A.3) is interpreted as the "on-set" of the slow cracking time. The accuracy of the deflection measurement is recommended to be better than ±1,0 % of the measured deflection and the time measured is normally within ±1,0 % of the elapsed time. The slow cracking shall be confirmed, whenever possible, through fracture surface observation on every specimen tested.

In case the on-set of the cracking time is not readily apparent on the ring deflection versus time curve (see Figure A.4) other ways of detecting on-set cracking time can be used. One such technique is the "modified displacement" method [6], which is found to bring out information regarding on-set cracking time with reasonable accuracy and success.

7.4 Notch depth measurement

After completion of the test, the specimen is removed from the test medium and cooled down to ambient temperature. A half section of the ring containing the notch at the centre is cut out and cooled down for impact fracturing to reveal the fracture surface containing a clear view of the inner wall notch. The depth of the razor produced notch shall be measured to an accuracy of ±0,1 mm with a microscope or equivalent means. The notch depth is reported as the average of measurements from at least three different locations. For accurate results, the notch depth measured shall be within 0,29 to 0,31 of e. If this is not the case, it is recommended to repeat the test.

7.5 Number of specimens

In order to be able to draw unambiguous conclusions, it is common practice to test at least three specimens.

Test report

The test report includes the following information:

- a reference to this Technical Specification, i.e. ISO/TS 16479:2012;
- all details necessary for complete identification of the product (e.g. resin, type of product, manufacture, production date, etc.);
- the notched ring specimen and loading dimensions in millimetres, given in Table 1; c)
- all details of sample preparation cutting, dimensioning, notching, etc.;
- the load-razor blade displacement curves if using razor blade compression method for the inner wall notching (should also include the razor blade information including thickness);
- the machined notch depth measurement after the completion of testing;
- the test temperature and the test medium;
- all details of the type loading equipment, deflection measuring device, etc.;
- i) the description of the method used to ensure line loading the crack front (should include deviation of the loading line in relation to two sidewall notches, if observed);

- j) the load and corresponding on-set slow cracking time data (should also include all ring deflection curves used to determine on-set cracking time) as well as the fracture surface photograph indication of the slow crack growth. If another method other than direct observation of the deflection versus time curve was used, provide the working description of the method;
- k) the stress intensity factor and corresponding on-set slow cracking time data (if applicable). Specimen dimensions for which the equation given in Annex B is not applicable, the stress intensity factor needs to be determined and the details of the stress intensity factor calculation shall be reported;
- l) details of any factors which may have affected the results, such as any incidents or any operations not specified in this document and referring standards or any possible deviation from these documents;
- m) number of samples tested;
- n) the date of the test.

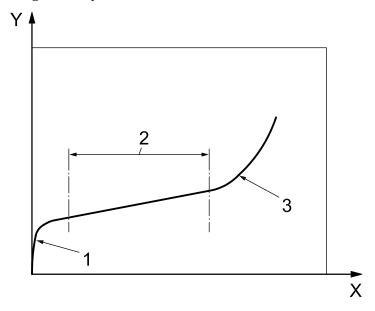
Annex A

(informative)

On-set slow cracking time detection

With NRT specimens, the on-set slow cracking time is typically determined from the ring deflection curve. The ring deflection curves are similar in appearance to that of a typical creep curve (see Figure A.1) in that primary, secondary and tertiary regions are often observed on prior deformation, slow crack initiation, crack growth and failure (see Figure A.2). In case the crack initiation occurs during the secondary region and growth occurs by the mechanism characteristic of "stick-slip" growth the deflection curve appears as shown in Figure A.3. Both in Figures A.2 and A.3, the on-set cracking time is readily determined by simple observation of the ring deflection versus the time curve, as indicted on the curves. However, where there are cases which the on-set cracking time is not apparent in the curve, as shown in Figure A.4, a method other than simple observation from the deflection curve should be found. One such technique is the "modified displacement" method and it is found to bring out information regarding on-set cracking time with reasonable accuracy and success in some cases. The complete details of the method are given in Reference [6].

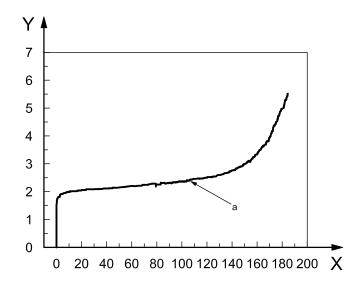
Figure A.5 shows an example of a notched ring loading device, having incorporated the electronic displacement gauge for monitoring the accumulation of ring deflection with time. The ring deflection data are acquisitioned automatically at the mid-span deflection by means of a measuring stroke movement of the pneumatic loading actuator. In this particular case the spring in the gauge was removed to prevent any influence on the loading accuracy.



Kev

- time (h) X
- displacement (mm) Υ
- 1 primary creep
- 2 steady-state (secondary) creep
- tertiary creep

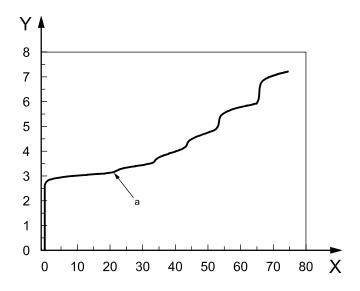
Figure A.1 — A typical creep deflection curve showing the regions of primary, secondary and tertiary creep



Key

- X time (h)
- Y displacement (mm)
- a On-set slow cracking time.

Figure A.2 — A typical deflection graph with on-set slow cracking time readily observed on the curve

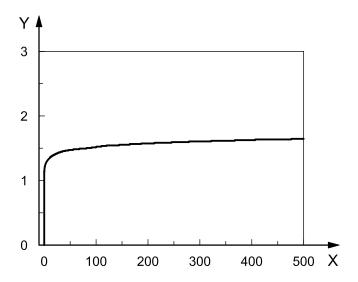


Key

- X time (h)
- Y displacement (mm)
- a On-set slow cracking time.

Figure A.3 — A typical ring deflection curve from stick-slip crack growth

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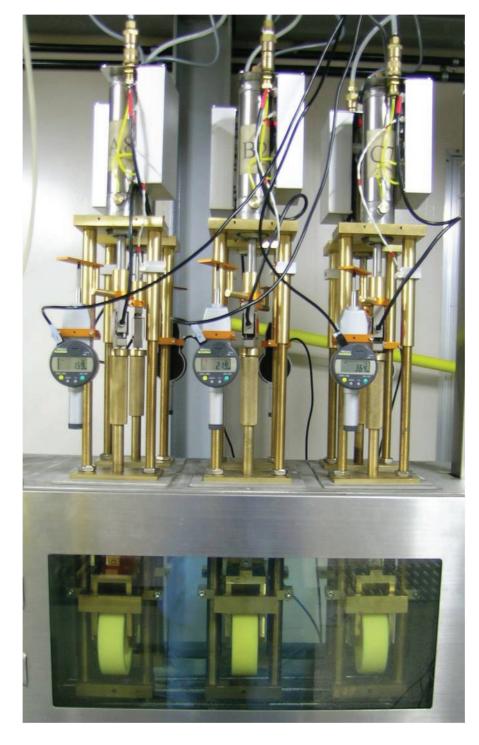


Key

time (h)

displacement (mm)

Figure A.4 — A typical ring deflection graph where the on-set slow cracking time is not apparent on the curve, modified displacement method [6] may be applied to depict on-set cracking time



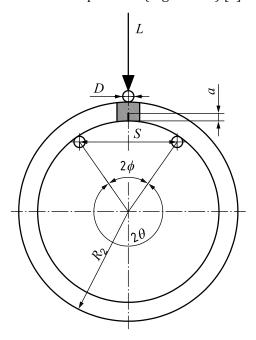
 $Figure \ A.5 - Pneumatically \ actuated \ notched \ ring \ loading \ device \ with \ electronic \ displacement \\ gauge \ assembly$

Annex B (informative)

Stress intensity factor plot

For the purpose of making a comparison of the NRT test to other available slow crack growth methodologies with different loading geometries, such as NPT (ISO 13479), the stress intensity factor approach is useful and this Annex provides the background for converting applied load into stress intensity factor.

The stress intensity factor equation for NRT specimen (see Figure B.1) is shown in Formula (B.1) [8]. This equation is applicable for sizes from 63 mm up to 400 mm nominal ring diameters and the geometric correction factor for SDR 9, 11 and 13,6 are also given in Formula (B.2). Similarly, Formula (B.3) gives the stress intensity factor equation for NPT specimen (Figure B.2) [9].



Key

applied load L

diameter of loading and supporting rods

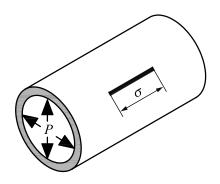
razor notch depth а

S span length wall thickness

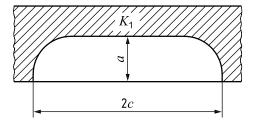
R₁ inside radius

R₂ outside radius

Figure B.1 — The loading geometry of NRT specimen



a) NPT specimen and under internal pressure



b) Outer surface crack configuration

$$K_{I.NRT} = \frac{3L\cos\phi R_2 \left(\theta - 2\sin\theta\right)}{2\pi W_N t^2} \times Y_{SDR\ NO} \sqrt{\pi a}$$
(B.1)

$$Y_{SDR9} = 53.71 \left(\frac{a}{t}\right) - 307.29 \left(\frac{a}{t}\right)^2 + 723.33 \left(\frac{a}{t}\right)^3 - 600 \left(\frac{a}{t}\right)^4$$

$$Y_{SDR11} = 21.39 \left(\frac{a}{t}\right) - 109.85 \left(\frac{a}{t}\right)^2 + 224.76 \left(\frac{a}{t}\right)^3 - 155.56 \left(\frac{a}{t}\right)^4$$
(B.2)

$$Y_{SDR13,6} = 38.08 \left(\frac{a}{t}\right) - 212.64 \left(\frac{a}{t}\right)^2 + 493.81 \left(\frac{a}{t}\right)^3 - 400 \left(\frac{a}{t}\right)^4$$

Key

P internal pressure

2c machined-in crack width

 K_1 crack-tip stress intensity factor

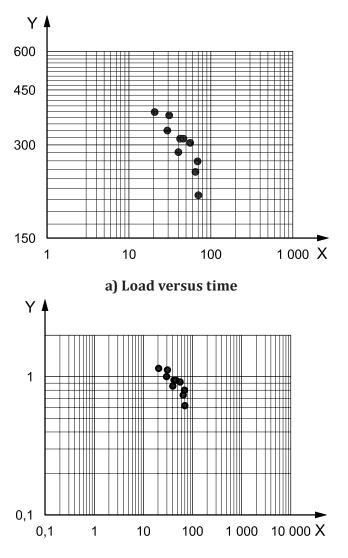
a machined-in crack depth

2c machined-in crack width

Figure B.2

$$K_{I} = \frac{P(SDR)\sqrt{\pi a}}{\frac{\pi}{4}\left(3 + \frac{a^{2}}{c^{2}}\right)}$$
(B.3)

Figures B.3a) and B.4a) illustrate a load versus slow cracking time graph and an internal pressure versus slow crack failure time curve obtained from NRT and NPT specimens, produced from the same pipe, respectively. Similarly, Figures B.3b) and B.4b) show these curves converted into stress intensity factor using Formulae (B.1) and (B.3), respectively.

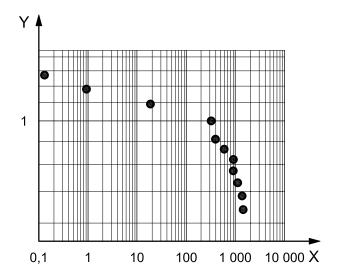


b) Stress intensity factor versus time graph of NRT method

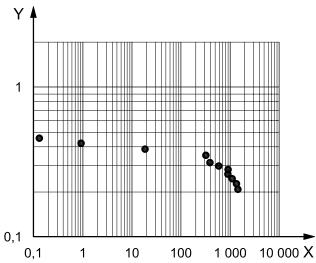
Key

- X time (h)
- Y load (N) [Figure B.3a)]
- Y stress intensity factor $(MP_a\sqrt{m})$ [Figure B.3b)]

Figure B.3



a) Internal pressure versus time



b) Stress intensity factor versus time graph of NPT method

Key

X time (h)

Y internal pressure (MPa) [Figure B.4a)]

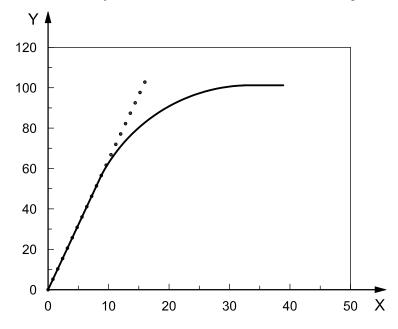
Y stress intensity factor $(MP_{a\sqrt{m}})$ [Figure B.4b)]

Figure B.4

Annex C (informative)

Flexure load estimation

To gain first approximation to the load magnitudes for producing slow crack growth in notched ring specimens, the following information may be useful. Figure C.1 exhibits a typical load-displacement curve obtained from the load-control flexure loading of 110 SDR 11 notched ring specimen. The yield load was taken as the load at which deviation from a linear line drawn through the initial slope from the origin of the curve, as shown. The loads below the 50 % of the yield load have been shown to cause slow cracking in many cases tested. In case slow cracking is not generated at 50 % of the yield load, successively decrease the load by 10 % increments until slow cracking is observed. It should be noted that the yield value seems sensitive to the exactness of the specimen and loading dimensions (e.g. initial notch length) and differences in the yield load could be observed between specimens for this reason.



Key

X displacement (mm)

Y load (N)

Figure C.1 — Load-displacement curve of a 110 SDR 11 notched ring specimen

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