A NEW METHOD TO CHARACTERIZE ENVIRONMENTAL STRESS CRACKING RESISTANCE (ESCR) OF POLYETHYLENE PIPES

Ben Jar, Patrick Ward, Chester Jar and Yi Zhang, University of Alberta, Edmonton, Alberta, Canada
Wajdy Ateerah, Polytyubes, Edmonton, Alberta, Canada

Abstract

A new test method has been developed to evaluate environmental stress cracking resistance (ESCR) for polyethylene (PE). The new test method applies transverse loading to the central area of a plate specimen, to generate local stretch that results in a truncated cone. Time for crack initiation in the truncated cone, during the exposure to an aggressive agent (10% Igepal CO-630 solution), is used to characterize ESCR. Results from the new test method are consistent with those from ASTM D1693, but the former does not require any pre-notch and takes less than 3% of the time required for the latter. Based on the new test method, a stand-alone device has been developed to characterize ESCR, which uses change in electrical conductivity to measure the time for the crack development. The device is compact and easy to operate. Using this device, time for crack initiation can be determined automatically and accurately without the use of a commercial test machine.

Introduction

Environmental stress cracking (ESC) is a serious problem that needs to be considered for many polymers in load-bearing applications. Most of the current standards for characterizing ESC resistance (ESCR) [1-2] require the use of a pre-notch to accelerate crack growth during the exposure to an aggressive agent (such as 10% Igepal CO-630 solution). Most of the test methods use time for crack growth in an aggressive environment to characterize ESCR. However, variation of the pre-notch quality has been suspected to cause inconsistency of the test results. In view of this problem, the current project is aimed at developing a new test method that uses notch-free specimens to characterize ESCR. The use of notch-free specimens also enables the new test method to evaluate the dependence of ESCR on the applied stress level, to facilitate the prediction of time for crack formation as a function of loading condition and environment. This new test method also has the potential to be used to evaluate resistance to the in-service environment, not merely for the qualitative comparison and characterization of ESCR.

In addition to the above functional capability, the new test method takes a much shorter time to complete than the current standards. Therefore, it can replace the current standards for characterizing ESCR for PEs that take too long to generate crack growth using methods as ASTM D1693.

This paper details principles for the new test method, describes a stand-alone device that has been developed based on the new test method, and evaluates and compares results from the new test method with those from ASTM D1693.

Principles of the New Test Method

The new test method measures ESCR by firstly applying transverse loading to a plate specimen to generate deformation. Cylindrical indenter with a flat end is used to introduce the transverse loading. The deformed region is then exposed to 10% Igepal CO-630 solution to accelerate the crack formation. Time for the crack formation during the exposure to the 10% Igepal CO-630 solution is used to characterize ESCR for the specimen. Since the indenter is kept at a fixed position during the exposure to the 10% Igepal CO-630 solution, the new test method applies essentially a similar loading mode as that used in ASTM D1693 during the exposure to the aggressive agent for crack development.

Deformation introduced in the new test method is to transform the central part of a plate specimen to a truncated cone that contains a highly stretched annular region around the area in contact with the indenter. By exposing the outer surface of the truncated cone to 10% Igepal CO-630 solution, cracks are generated in the highly stretched annular region, and time for crack generation in that region is used to characterize ESCR. The new test method does not rely on a pre-notch to accelerate crack formation. Therefore, its results can be used to evaluate the influence of stress state and initial loading speed on the time for crack formation, which cannot be achieved using any of the current standard test methods.

Specimens used in the current study have a size of 40x40 mm$^2$ and 3 mm in the nominal thickness. This size can be further reduced if required. Specimen of the above size is placed on the top of a solution container that has the inner diameter of 15 mm. For pipe specimens, arc-shaped plates of the above size were prepared from two types of PE pipe, which were then bolted down flat along the edges before the testing.

Indenter used in this study has a diameter of 13 mm. Such an indenter diameter of only 2 mm smaller than the inner diameter of the solution container, and is used to increase shear deformation involved in the specimen
deformation before the commencement of local stretch to form the truncated cone.

Figure 1(a) presents the conceptual depiction of the set-up used in the new test method in which a truncated cone is formed in the central part of the specimen. Since the indenter prevents radial contraction of the truncated cone, a bi-axial stress state is generated in the stretched annular region. As shown in the figure, outer surface of the truncated cone is exposed to the 10% Igepal CO-630 solution to accelerate the crack formation. Figure 1(b) presents the top view of the truncated cone which consists of a central area that is in contact with the end of the indenter, and an annular region where cracks are generated during the exposure to the 10% Igepal CO-630 solution. Time for crack generation in the annular region is used to characterize ESCR for the specimen.

Figure 2 presents typical load-displacement curves from the new test method, by applying monotonic transverse loading to the central part of the specimen at 23°C. The curves in Figure 2 were generated at the indentation speed of 1, 10 and 30 mm/min and without any exposure to the 10% Igepal CO-630 solution. Local stretch introduced by the indenter starts around the circumference of the central area that is in contact with the flat end of the indenter. Growth of the local stretch commences at the peak load, which with the increase of the displacement, leads to the formation of a truncated cone. Although the load drops at the beginning of the local stretch, the load maintains at a relatively constant level during the growth of the truncated cone, as shown in the stroke range of 7 to 10 mm in Figure 2. No crack was generated in the truncated cone before the unloading.

![Figure 1](image1.png)

![Figure 2](image2.png)

**Materials**

Four types of polyethylene (PE) plates, two of high-density PE (HDPE) and the other two linear-low density PE (LLDPE), were used to evaluate the new test method. Characteristics of these materials are given in Table 1. In addition, two types of PE pipe, PE2708 and PE3608, were tested under the same condition to evaluate their ESCR.

<table>
<thead>
<tr>
<th>Material</th>
<th>Plate</th>
<th>Pipe</th>
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<tbody>
<tr>
<td></td>
<td>LLDPE</td>
<td>HDPE</td>
</tr>
<tr>
<td></td>
<td>(A)</td>
<td>(A)</td>
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<tr>
<td></td>
<td>0.932</td>
<td>0.942</td>
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<tr>
<td></td>
<td>0.938</td>
<td>0.948</td>
</tr>
<tr>
<td>Density (g/cc)</td>
<td></td>
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<tr>
<td>ES CR ASTM D1693 (10%) (hr)</td>
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<td></td>
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<tr>
<td>Yield stress (MPa)</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>15.0</td>
<td>16.7</td>
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<tr>
<td></td>
<td>16.7</td>
<td>19.1</td>
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<tr>
<td></td>
<td>19.3</td>
<td>22.8</td>
</tr>
</tbody>
</table>

Note that as shown in Fig. 2, post-peak stress remains at a relatively constant level during the growth of the truncated cone, i.e., in the stroke range from 7 to 10 mm. By choosing the indenter displacement in this range for stress relaxation, the initial stress state in the truncated cone...
cone should not be much affected by any minute variation of displacement among the tests.

Typical photographs of specimens after the indentation tests in Fig. 2 are presented in Figure 3, one for each crosshead speed, as noted by the number at the right, bottom number in each photo. Figure 3 shows that no visible crack exists in the annular region of the truncated cone.

![Figure 3 Typical post-test specimens for curves shown in Fig. 2 (Number at the right bottom corner stands for the crosshead speed used to generate the truncated cone.](image)

**Stand-Alone Device (Telecom)**

Based on the above test setup, a stand-alone device has been designed and manufactured in house. The device does not rely on the occurrence of load decay to detect the crack generation in the annular region of the truncated cone. Therefore, this device is able to measure the crack development without constantly monitoring the load decay during the exposure to the Igepal CO-630 solution.

The stand-alone device is named “Telecom” because its shape is similar to a telecommunication tower in Canberra, Australia. The Telecom device has a footprint with diameter of 80 mm and height of 180 mm, and can be operated with hand tools such as screwdrivers and Allen keys. Figure 4 presents the assembly drawing of the Telecom device and its exploded view which shows that the indenter is driven by a bolt (item 9). In this study, the truncated cone is generated by the indenter displacement of 8 mm, which takes about 3.5 revolutions of the bolt till the indenter is pushed against a disk spacer (item 5). The truncated cone formed using this displacement is sufficient to generate cracks of a reasonable size in the annular region.

The Telecom device uses change in electrical conductivity to detect the crack formation. Figure 5 depicts the operation principle for the Telecom device, in which a home-made meter is used to monitor the electrical conductivity between the indenter and the solution container. As the device has all components that are in contact with both indenter and solution container made of plastics, reading of the home-made meter shows only negligible electrical conductivity before any crack is generated in the annular region of the truncated cone. Once cracks are generated, the Igepal CO-630 solution flows into the truncated cone, thus establishing an electrically conductive path between the indenter and the solution container and increasing electrical conductivity detected by the meter. The change in the electrical conductivity is used to indicate the crack formation in the annular region.

![Figure 4 Assembly drawing and its exploded view of the stand-alone device (Telecom) based on the new test method.](image)

![Figure 5 Schematic depiction of the set-up for the stand-alone device (Telecom), based on the new test method, the home-made meter and the computer for data recording.](image)

![Figure 6 presents an example of the test results from the Telecom device. Figure 6(a) is a plot of relative electrical conductivity versus time during the stress](image)
relaxation, during the exposure to 10% Igepal CO-630 solution. Time for the first occurrence of the step-wise increase in the electrical conductivity, before 40 minutes in Fig. 6(a), is the time for the first crack generation in the annular region of the truncated cone. Figure 6(b) is a photograph of the post-tested specimen for 6(a), showing visible cracks in the annular region of the truncated cone.

![Graph](image)

Figure 6 Example of test results from the Telecom device: (a) plot of electrical conductivity versus time during the stress relaxation test, and (b) photograph of the post-tested specimen for (a), showing cracks in the annular region of the truncated cone.

Note that the Telecom device is designed to be able to use indenters of any diameter from 7 to 14 mm. Increase of the indenter diameter is to increase the involvement of shear deformation in forming the truncated cone, thus reducing the initial plate deflection before the truncated cone formation.

Figure 7 summarizes results from the Telecom device for 4 PE plates and 2 pipes listed in Table 1, expressed in terms of ESCR-A based on ASTM D1693. Both abscissa and ordinate of the plot in Figure 7 are in the logarithmic scale, and value for each point from the Telecom device represents the average of four measurements, with the error bar indicating the maximum and minimum values. The results from the plate specimens were used to determine the fitting line between the number of hours using the Telecom device and that from ASTM D1693. Expression for the fitting line is also given in the figure. The figure indicate clearly that ESCR determined using the Telecom device gives the same ranking as that by ASTM D1693, but the time required using for the former is only about 2% of that from the latter, as suggested by the equation in the figure.

The numbers of hours for ASTM D1693 in Figure 7, for specimens from the two pipes in Table 1, are estimated based on the measured results using the Telecom device, using the expression given in Figure 7. Based on the estimation, the number of hours for the pipe materials, if measured using ASTM D1693 condition A, should be less than 5000.

![Graph](image)

Figure 7 Summary of test results from the Telecom device, expressed in terms of time from ASTM D1693, ESCR-A.

Conclusions

A new test method has been developed for characterizing ESCR for PE. Results show clearly that the new test method provides the same ranking for ESCR as that by ASTM D1693, but the former takes a much shorter time than the latter to complete. Since specimens for the new test method does not require any pre-notch to accelerate the crack formation, the new test method avoids the potential scattering of test results that can be caused by the uncertainty of the notch quality.

A stand-alone device, named Telecom, has been developed to quantify ESCR based on the new test method. The device is compact in size, and does not require the load measurement in order to determine the time for crack formation. Furthermore, the time for crack generation in the specimen during the exposure to the aggressive agent can be automatically recorded using a computer through a hyper terminal, thus improving efficiency and accuracy of the ESCR measurement.

Acknowledgement
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References