Fractographic Study of the Creep Behavior of High Density Polyethylene (HDPE) for High Pressure Pipe Application

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**Abstract**

Slow crack growth resistance, often estimated by the bent strip environmental stress crack resistance (ESCR) test and full notch creep test (FNCT). The FNCT is an important performance parameter for different applications of HDPE, from the blow moulding segment like bottles and containers up to the extrusion moulding segments such as high pressure gas pipes. Fractographic analysis was carried out on the fracture surfaces of the FNCT specimens of the HDPE samples to understand the deformation micromechanism of fracture due to slow crack propagation. This paper presents the study of the fractured surface of different samples with different crack resistance characteristics using optical microscope (OM) and scanning electron microscope (SEM). The experimental results showed that increase of micro fibrous structures is directly related to an increase in full notch creep test (FNCT) failure time.

**Introduction**

The term slow crack growth (SCG) resistance is an important parameter for the HDPE material. This property resulted into specific application such as pipe application. This can be resulted into the lifetime of final product like HDPE pipe [9,8]. In the industries, there are several ways to measure slow crack resistance characteristic of the HDPE. The most widely used and oldest SCG test is the Bent Strip environmental stress crack resistant (ESCR) test. However, due to limitations of the Bent Strip ESCR method, several alternative ESCR tests have been developed for use in the United States and Europe including Pennsylvania Notch Test (PENT), Full Notch Creep Test (FNCT), cone test and notch pipe test (NPT) [1].

Among these tests, full notch creep test is one of the widely accepted test to determine the slow crack resistance of materials particularly for polyethylene material in accelerated environment. This type of analysis is an important performance parameter for different applications of HDPE, from blow moulding segment like bottles and containers up to the extrusion moulding segment such as gas pipes [12].

The slow crack growth in polyethylene has been widely studied in the past [4,17,18,19,25,10,5,26,21, 20]. In general, the failure process begins with the crazes, where it has been defined as deformation accompanied by volume increase [15,6]. Kramer reported that, plane strain is more likely to open up such voids, than plane stress in the craze nucleation mechanism [14]. After the craze, they propagate and terminate turning into a crack.

Several molecular mechanisms also have been proposed for polyethylene [20,3,22,27,26]. Lustiger et al. [21,20] have proposed “interlamellar failure” as the controlling mechanism of environmental stress crack (ESC), with the concentration of the tie molecules as a factor in ESCR. Brown et al. [4] concluded that the mechanism of slow crack growth involves the disentanglement of the tie molecules from the crystals. The number of tie molecules and the strength of the crystals that anchor them are considered the controlling factors.

This study indicates the insight into the micromechanism of deformation and fracture due to slow crack propagation during the FNCT test. The effects of fracture surface in relation to different FNCT failure times were also studied.

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**Methodology:**

**Material:**
High density polyethylene (HDPE) samples are received from Tasnee, Saudi Arabia. All the samples received are HDPE pipe grade and selected based on their relative FNCT failure time. The details of the samples are presented in Table 1.

<table>
<thead>
<tr>
<th>No.</th>
<th>Sample code</th>
<th>Relative FNCT results</th>
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<tbody>
<tr>
<td>1</td>
<td>HDPE-1</td>
<td>1</td>
</tr>
<tr>
<td>2</td>
<td>HDPE-2</td>
<td>2.5</td>
</tr>
<tr>
<td>3</td>
<td>HDPE-3</td>
<td>3.4</td>
</tr>
<tr>
<td>4</td>
<td>HDPE-4</td>
<td>6.3</td>
</tr>
</tbody>
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**Morphological analysis:**

For the morphological characterization, the samples received are in the form of fractured FNCT bar where the area dimension is 10 mm x 10mm. The fracture surface was cut into small piece using the knife cutter. The samples are directly subjected to optical light microscope (LEICA M205) with Leica Application Suite V.4 software. For higher resolution images, the samples were then sputter coated and microscopically analysed using scanning electron microscope (Jeol JSM 6510LA).

**RESULTS AND DISCUSSIONS**

Four different samples based on the FNCT results are taken for the study. The samples received are fractured FNCT bars after completing the FNCT analysis. These samples are taken for the morphology study. The side surface of these fracture samples are scanned under optical microscope and the micrographs of these samples are shown in Figure 1. All the samples show many broken ligament from the side. The side view does not indicate any significant difference in the failure mode. All these fracture FNCT samples surface shows more brittle failure in terms of FNCT terminology according to the ISO 16770 [11]. As defined by the ISO 16770, there are two typical types of failure mode for the FNCT test specimen for HDPE samples i.e. brittle failure and ductile failure. The images of these failures are shown in Figure 2. Brittle failure is defined in which the fracture surface exhibits no permanent material deformation to the naked eye, i.e. stretching, elongation or necking down. In some tougher materials also shows an extended ligament may form in the center (Figure 1b). However, ductile failure shows permanent material deformation with stretching, elongation and necking down. The analyzed samples of the study are in alignment with the definition of the brittle behaviors as shown in figure 3b, where the material deformed randomly and many ligaments started from notch end with the stretching, elongation and necking down and breaks from various places.

![Fig. 1: Side surface of FNCT fracture samples: a) HDPE-1, b) HDPE-2, c) HDPE-3 and d) HDPE-4.](image-url)

![Fig. 2: FNCT failure surfaces: a) brittle, b) brittle failure (extended ligament may form at centre) and c) ductile](image-url)
there is stretching and elongated deformation appeared on the materials, as shown in red arrows Figure 2. It also indicates center ligament appeared in all the fracture FNCT samples in a small region as indicated in 2b.

Fig. 3: Top surface of FNCT failure samples: a) HDPE-1, b) HDPE-2, c) HDPE-3 and d) HDPE-4. Red arrows: stretching and elongated deformation

To overview the fracture surface in details, scanning electron microscope (SEM) was used to study the insight for the micromechanism of deformation and fracture due to slow crack growth. For detailed fractographic study, microscopic analysis using SEM was conducted on 2 main regions: edge and central surface regions as shown in Figure 4. The edge surface regions include the region a, b, c, d and central surface region is on the region e.

Fig. 4: Regions scanned using SEM analysis: Zones-a, b, c, d and e.

The edge regions of the samples by SEM are shown in Figure 5 for one of the representative fracture of HDPE-2 sample. In the samples, the micrographs show that the crazes initiated from the notch with sharp edge and followed by the blunt tips which is usually observed in the ductile materials during the crack formation [Zhigilei 2010]. It is also called as shear lips and characteristic behavior of ductile failure mode [23,13]. The so called shear lips are shown in Figure 5 with the Red arrows.
Fig. 5: SEM micrographs of fracture sample (HDPE-2) for the edge surface: Zone- a, b, c, and d. Red arrows: shear lips; Yellow arrows: dimple patterns.

At higher magnification (Figure 6), the growth of the fracture was observed in details. The notch grows by transformation of matrix materials into fibrillar matter. This type of growth of this craze-like feature occurs through the disentanglement and breakdown of the numerous microfibrils (Figure 6a). Meanwhile, microvoids are also observed in this region, as shown in 6b, which is also known as dimples patterns consists of dense microfibrils and microvoids [23]. The dimple patterns are also shown in Figure 5 with the yellow arrows.

Fig. 6: SEM micrographs shows the dimples patterns (a) and dense microbrils & microvoids (b)

The micrographs of all the samples (Figure 7) are captured to see the impact of FNCT behaviour on the dimple pattern in the fractured surfaces. The images indicates that the area of the dimples pattern or region is in the ascending order starting from HDPE-1, HDPE-2, HDPE-3 and HDPE-4 (Figures 7a-7d) respectively. During the crazes- crack transition, as the stress applied is higher than the critical stress level of remained material’s
part, the remained part get broke and terminate with the stretching, necking down in this central region, which is shown in Figure 7.

Fig. 7: SEM micrograph of FNCT failure samples zone at the magnification of X30: a) HDPE-1, b) HDPE-2, c) HDPE-3 and d) HDPE-4.

Generally, crazes are initiated when the external stretch causes a microscopic void to open up at a stress concentration created by a precreated notch, heterogeneity in the molecular network or a foreign particle [6]. After the craze is created, then propagate into fibril matters. The formation of such dense microfibrils was reported to be responsible for the higher propagation energy absorption [23]. As reported earlier, the crystallites are connected by the tie molecules through the amorphous phase in polyethylene [4,21,20]. The tie molecules play an important role in the mechanical properties of the polymer, through the transmission of load. In the FNCT test, the solvent used acts as stress cracking agent to promote crazing and cracking. It lowers the cohesive forces which maintain the tie molecules in the crystallites, thus facilitating their “pull-out” and disentanglement from the lamellae [24]. Consequently, cracking is initiated at stress values lower than the critical stress level of the material, where it indicates the solvent embrittlement the polyethylene failure. This basically means that the resistance to craze initiation and failure in such a slow crack growth mechanism is primarily determined by the intrinsic strain hardening response of fibrils [7]. This suggests that an increase in FNCT value resulted in an increase in the area of the dimple region with fibrils, which is in alignment with the SEM micrographs.

Conclusion:

The fracture surface of FNCT bar made of HDPE shows the brittle behavior on the macroscopic level but the ductile behavior at micro scale. Material deformed with the stretching, elongation and necking down regardless of different FNCT results. The increase in FNCT value resulted in an increase in the area of the dimple region of the image. This suggests that this can be used as the qualitative test for the slow crack resistance of the HDPE material.

REFERENCE