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Root Cause Failure Analysis of Polyethylene Tubing Utilizing Microscopy

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Polyolefin-based tubing applications for liquid and gas transport enhance and simplify our everyday lives. Analytical tools such as light, scanning and transmission electron microscopies play key roles in material development and design at Dow [1-2]. Light microscopy (LM) covers a broad range of magnifications ranging from imaging large fabricated parts, to elucidating internal morphological structures. Under proper illumination, LM can provide details about failure, crystallinity, orientation and residual stresses [3]. Once regions of interest have been identified, scanning and transmission electron microscopies (SEM, TEM) can be used to further interrogate material morphology and failure [4].

Case studies will be presented to demonstrate how microscopy techniques were leveraged to identify the root cause of failures in polyethylene-based tubing applications. Sample preparation protocols, experimental techniques are discussed to provide insight on how each material was investigated.

The first case study identified the root cause of premature field failure in black agricultural micro-irrigation tubing produced from linear low density polyethylene (LLDPE) containing carbon black (CB). The client indicated that tubing was produced using LLDPE tubing grade resin and had prematurely failed after 5 years of service. It was hypothesized that environmental stress cracking (ESC) from chemicals used for pest control and fertilization had accelerated failure. Visual examination of the tubing showed severe surface embrittlement and micro-cracking consistent with ESC (Figures 1a). To investigate the root cause of failure, a Leica UC7 microtome was used to collect 5um thick cross sections at -80ºC along a crack arrest. An Olympus Vanox compound optical microscope used under transmitted brightfield and epi-fluorescence illumination determined that cracks initiated along the inner tubing surface and propagated outward (Figure 1b). A thin ductile layer of approximately 70µm thick, displaying a more oriented CB pigment dispersion, was present along the outermost tubing surface (Figure 1c). The presence of a ductile layer suggested that a co-extruded structure was used to produce the tubing. Backscatter SEM images collected on an FEI Nova Nano600 SEM verified the presence of a ductile surface layer and showed extensive surface mud-cracking consistent with ultraviolet oxidation (Figure 1d). The tubing manufacturer verified that LLDPE tubing grade resin had been co-extruded as a protective outer surface layer on the tubing. The bulk of the tubing was produced using a combination of LLDPE tubing grade resin and regrind containing tubing with emitters from the manufacturing process, and was depleted in UV stabilizers required for extend service life.

A second case study dealt with flexible tubing produced from a polyolefin elastomer (POE) resin. The tubing manufacturer had installed a new feed die and experienced linear cracks along barbed tubbing connectors (Figure 2a). Prior to die modification, failures did not occur along tubing connectors. This study focused on identifying the root cause of linear cracks in tubing produced using a new die. A Leica MZ-16 stereo microscope was used under cross polarized light to locate and examine tubing cracks and stress fields around connectors (Figures 2b). Tubing cross sections were cryogenically sectioned at -80ºC to approximately 300um (Figure 2c) and 5um (Figure 2d) in thickness, and examined using a compound LM under transmitted cross polarized light. Stress fields and parabolic polymer flow fronts (blue arrows in Figure 2c) were observed on both sides of a crack which developed internally within the tubing wall. Examination of a 5μm thick cross section 2-3mm directly ahead of a crack arrest (red dotted line in Figure 2b), identified a linear birefringent streak forming a knit line between two parabolic flow fronts (red arrows in Figure 2d). The greater birefringence associated with the knit-line suggested a higher density, more crystalline material. Transmission electron microscopy was used to examine the knit lines and determine they were comprised of an impact modified polypropylene (PP) resin that was not part of the tubing formulation. The PP-based knit-lines were not miscible with the POE tubing matrix and formed sharp interfacial boundaries (Figures 2f and 2g). A control tubing produced with the original die also showed evidence of resin contamination, however, the oval-like flow patterns produced with the old die (green ovals in Figure 2h) did not form linear knit lines in the thickness direction which induced failures.

References:
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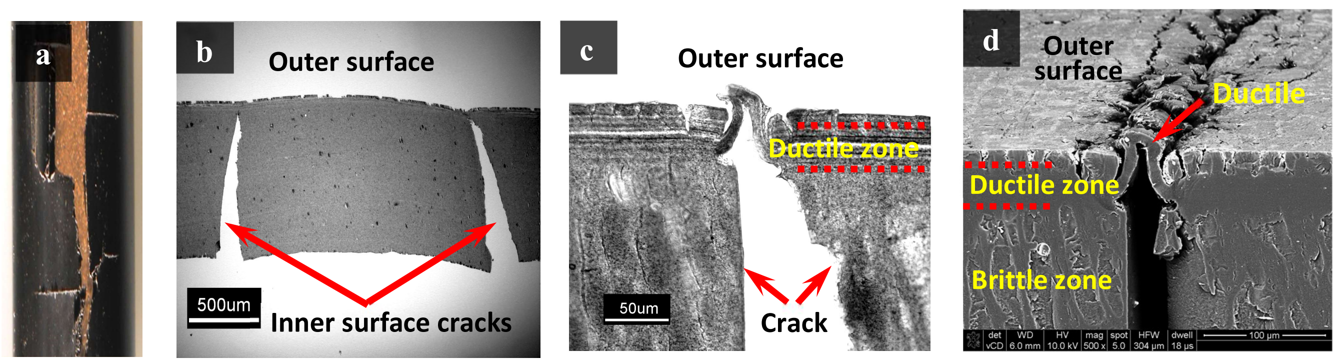

Figure 1. (**a**) Failed tubing (**b**) LM cross section of inner surface, crack initiation (**c**) LM cross of crack and ductile outer layer with streaked pigmentation (d) backscatter SEM of crack and ductile outer layer.

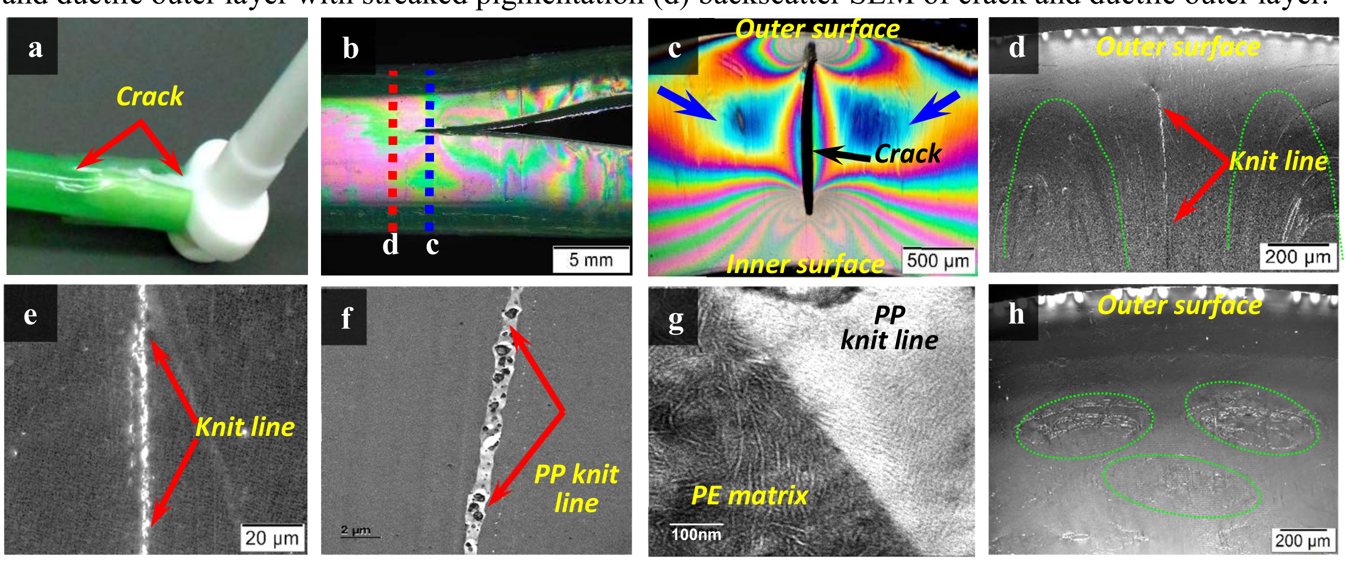

Figure 2. (**a**) Tubing crack over connector (**b**) Linear crack and arrest under cross polarized light (CPL) (**c**) Tubing cross section under CPL showing internal crack and stress fields (**d** and **e**) CPL images ahead of a crack arrest showing parabolic flows and knit line (**f** and **g**) TEM images of knit line morphology and interfacial boundary (**h**) CPL image of control tubing cross section showing oval flow patterns.

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