doi:10.1017/S143192761600948X
Microsc. Microanal. 22 (Suppl 3), 2016

Troubleshooting Fabricated Products via Microscopic Defect and Failure Analysis

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Troubleshooting product issues requires a disciplined approach utilizing a variety of analytical tools. Tools such as light, scanning and transmission electron microscopies play key roles in material analysis and product development at Dow [1-2]. Light microscopy (LM) can cover a broad range of magnifications ranging from analyses of large fabricated parts, to more detailed cross sectional analyses. Under proper illumination, LM can provide details about fracture surfaces, interfaces, crystallinity and residual stresses [3]. Once regions of interest have been identified, scanning electron microscopy (SEM) can be used to locate and identify defects and mechanisms which induced product failures.

Case studies will be presented to demonstrate the utility of microscopic analyses in investigating defects and failures associated with products ranging from films to fabricated articles. For each example, sample preparation methods are discussed to provide insight regarding experimental techniques and instrumentation required. Established protocols for morphological analyses of semi crystalline and amorphous polymers are well documented in the literature [4].

The first study involved a manufacturer of polyethylene based six-pack ring holders for beverage bottles. The polyethylene ring holders were slipping off product labels adhered to the bottles. The customer suspected the labels were at fault and provided three labels for analyses. Two labels had passed a ring holder support test and a third had not. Topographical comparison of the labels was done using reflected light microscopy on an Olympus Vanox-S compound LM (Figures 1a and 1b). Fish-eye type defects were found throughout the outer surface of a label having slippage of the ring holder. A Leica UC7 microtome was used to collect 5um thick cross sections at -80^{o}C across the fish-eye defects. Label cross sections were examined on an FEI Nova Nano600 SEM coupled with a 30mm^2 Bruker AXS silicon drift energy dispersive xray detector (EDX). SEM analysis showed that fish-eye defects were comprised of inorganic spheres of approximately 1-2um in diameter causing bumps along the outermost film surface (Figure 1c). Elemental analysis detected Si and O in the spheres, which was consistent with a silicon dioxide slip additive. The surface roughness introduced by the spheres was causing the ring holders to slip from bottle labels. The label film manufacturer verified that a slip additive was used in other formulations and should not have been present in this application.

A second study dealt with a manufacturer of carbon black filled high density polyethylene pipe. The customer indicated that a high concentration of pit-like defects were present on the exterior pipe surface. A Leica MZ-16 stereo microscope was used to survey the pipe and determine that dark flake-like defects and white defects were present within the surface pits (Figures 2a and 3a). Defect cross sections were cryogenically sectioned at -80ºC to approximately 5um in thickness and placed on a glass slide containing immersion oil. A compound LM was used under transmitted polarized light to determine that the dark defects were brown and polymeric (Figures 2b and 2c). The white defects appeared inorganic and contained two types of crystalline material (Figure 3b). Epi-fluorescence imaging using an ultraviolet light source caused an intense emission of the brown defects, indicative of thermal oxidation (Figure 2d). Infrared micro spectroscopy verify that the brown defects were oxidized polyethylene that likely formed on the hot metal surfaces of the extruder and flaked off during processing. The white crystalline defects were examined using SEM and EDX from cryopolished block faces used to collect thin sections for light microscopy. A backscatter electron image of a white defect is shown in Figure 3c. Elemental analyses coupled with false color elemental mapping determined that the defects had a plate-like matrix phase containing Si, Al and O and solid particles containing Ca and O. The morphology and composition of the defects were consistent with clay and $CaCO_3$ and not part of the resin formulation. Defects causing pipe surface pits were introduced from improperly purged equipment during pipe fabrication and not related with the resin formulation.

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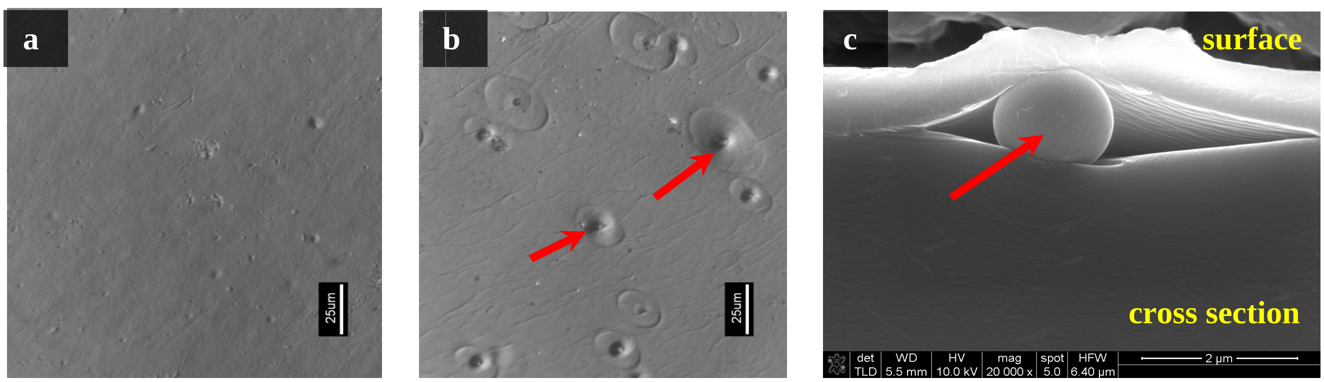

Figure 1. LM surface topography of (**a**) a control label film, (**b**) a film displaying fish-eye surface defects and (**c**) SEM image of film cross section exposing inorganic sphere causing fish-eye defects.

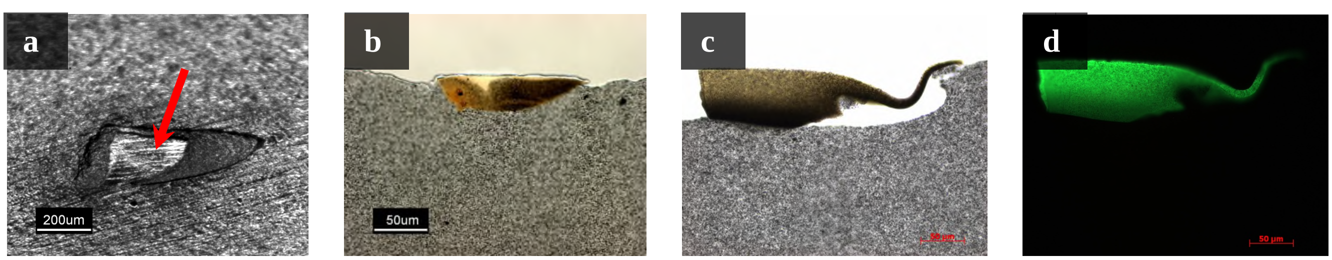

Figure 2. (a) Dark flake-like defects causing pits on pipe surface, (**b,c**) defect cross sections under transmitted polarized light and (**d**) defect under epi-fluorescence illumination.

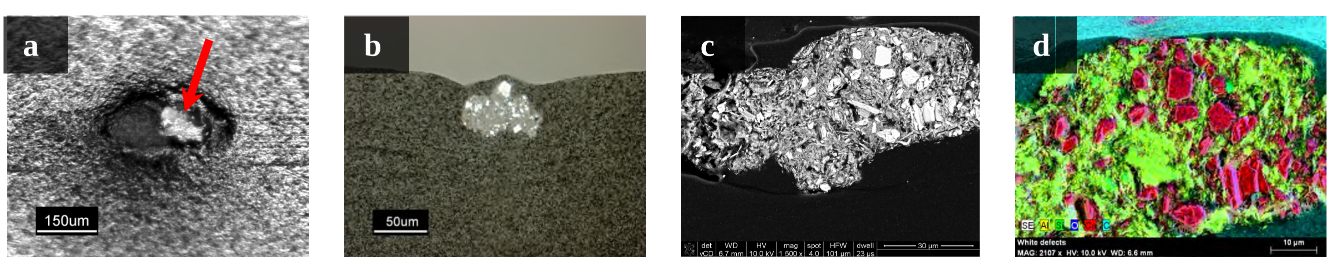

Figure 3. (a) White defects causing pits on pipe surface, (**b**) defect cross section under transmitted polarized light, (**c**) backscatter SEM image of defect and (**d**) false color elemental map of defect with red particles representing calcium, oxygen and carbon and the lime matrix, silicon, aluminum and oxygen.

https://doi.org/10.1017/S143192761600948X Published online by Cambridge University Press