# FRACTOGRAPHIC CHARACTERIZATION OF POLYCARBONATE FAILURE MODES

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### Abstract

Polycarbonate is an important plastic molding resin used to fabricate many engineered components. Because of its widespread usage, many different types of failures can result from various service conditions. Evaluating these failures through a systematic analysis program allows an assessment of how and why the parts failed. An essential portion of the failure analysis process is the fractographic examination, which provides information about the crack origin location, and the crack initiation and extension modes. The focus of this investigation was to characterize the surfaces of intentionally cracked laboratory samples in order to gain a more thorough understanding of polycarbonate fracture mechanisms. This paper will document some of the key fracture features associated with various polycarbonate failure modes.

### Background

Polycarbonate is a key molding resin used to produce engineered components in numerous applications, including the medical, appliance, and automotive industries. The widespread use of polycarbonate makes it one of the most important plastics within the group of materials commonly referred to as "engineering resins". The estimated 2002 demand for polycarbonate was 2,000,000 tons.<sup>1</sup> Given this relatively high volume, together with the diverse usage, it is not unexpected that a wide variety of different failure modes are encountered with polycarbonate components. Additionally, while polycarbonate is known for its inherent ductility, its susceptibility to chemical and other environmental effects produces a disproportionately high failure rate, relative to other plastic materials. In the author's experience as a failure analyst, approximately 20% of all failure investigations are conducted on parts produced from polycarbonate. This is a relatively high number of failures, given the vast number of resins used to mold engineered products.

A thorough understanding of the failure mechanisms of polycarbonate is important, especially in light of the incommensurate failure rate. A key component of a proper failure investigation is the examination of the fracture surface via scanning electron microscopy (SEM), which allows for evaluation at high magnification with great depth of field. Fractogrphy is used to characterize the mode of the failure and can provide invaluable information regarding the stresses and conditions leading to the failure. In this investigation, molded polycarbonate specimens were stressed under several different conditions to intentionally create laboratory failures. A fractographic examination was subsequently conducted in order to understand and document the resulting fracture surface features. This study was conducted to further the understanding of the failure mechanisms routinely observed with polycarbonate parts.

# Experimental

A commercially available medium-viscosity polycarbonate resin was selected for the investigation. The resin was molded into plaques by a custom injection molder and subsequently machined to form the required test specimens. The prepared fracture surfaces were examined using a Hitachi S-3500N scanning electron microscope (SEM). The specimens were blown off and cleaned ultrasonically in a mixture of isopropanol and deionized water. Prior to the inspection, the samples were gold sputter coated to enhance the imaging.

# **Tests and Results**

### **Uniaxial Tensile Loading**

A uniaxial fracture was created through tensile testing using an MTS QTest QT/25LP universal mechanical tester. The evaluation was conducted in accordance with ASTM D 638 at 22 °C using a crosshead speed of 5.1 cm/min. (2 in./min.). Type I specimens were used for this evaluation. The tensile testing produced characteristically ductile results, with the samples having a nominal elongation at break of 165%. The samples exhibited substantial deformation and necking, indicative of yielding.

The SEM examination of a typical fracture surface showed that the cracking had initiated within the specimen wall at a The cracking initiated through local inhomogeneity. crazing, and the fracture surface exhibited an opened craze, indicated by a circular feature surrounding the origin, as shown in Figure 1. Radiating stress crack indications, known as hackle marks, were apparent within the remnant craze surface. These ridges were characterized by stretched flaps and represent the union of multiple individual extending cracks. At the outer boundary of the craze, the hackle marks extending toward the specimen wall exhibited a severe change in direction, consistent with the alteration of the external stresses. Within the crack origin area, no evidence was found to suggest the presence of true mirror or mist zones. Such morphologies are characterized by extremely smooth fracture surfaces, and are indicative of slow crack growth. In contrast, the observed features indicated that the crack nucleation occurred relatively rapidly, with unstable crack growth.

The zone outside of the opened craze was generally smoother than inside, but still exhibited the presence of hackle marks, as illustrated in Figure 2. Remote to the crack origin area, surrounding the boundaries of the tensile specimen, the fracture surface showed a slightly increased level of ductility, as indicated by enhanced stretching within the hackle marks. Overall, the fracture surface showed primarily brittle fracture features, with no substantial evidence of fibril formation. The appearance of the fracture surface was in strong contrast to the obvious macro ductility indicated by the permanent deformation and yielding.

### **Tear Loading**

A tear fracture was created using machined specimens conforming to ASTM D 624 Type C. The specimens were stressed using the MTS universal mechanical tester and the testing was conducted at 22 °C using a crosshead speed of 5.1 cm/min. (2 in./min.). The geometry of the specimens imparted crack opening stress, commonly referred to as Mode I stress. The obtained data showed that the sample had a tear strength of 1600 ppi. The fractured sample showed little evidence of apparent macro ductility, as indicated by the relatively low level of deformation.

As anticipated, the SEM examination showed that the cracking initiated along the edge of the specimen at the formed notch, as presented in Figure 3. The fracture surface features indicated multiple, distinct crack nucleation sites. The crack origin area was relatively smooth, between the observed hackle marks. The formation of hackle marks was characterized by the presence of chevron markings, otherwise known as a plumose pattern. Such features are commonly associated with ductile fracture. Within the chevron pattern, the hackle marks exhibited slight stretching and had a feather-like appearance. Coalescence of individual cracks within the mid-wall region produced characteristic V-shaped features, as shown in Figure 4.

The fracture surface was relatively flat, which was consistent with the Mode I stress orientation, and the absence of tilting or twisting. The final 10% of the fracture surface showed a slight alteration of the observed features. A morphology characteristic of uniaxial load failure was prevalent, associated with the final overload of the test specimen. Overall, the tear fracture surface displayed features that were generally consistent with those present on the uniaxial tensile failure specimen. However, the tear fracture surface was noticeably rougher, which is associated with more rapid crack extension. This is expected given the stress concentration generated by the sample geometry.

#### **Impact Loading**

Specimens representing the molded plaques were impacted using a Dynatup 8250 instrumented impact tester. This mechanical evaluation was performed at 22 °C. A review of the obtained load / deformation data showed results typical for a polycarbonate resin, including a total energy of 80 J (59 ft.-lb.). The impacted specimens exhibited substantial permanent deformation, characteristic of a high level of macro ductility.

Examination of the fracture surface via SEM, as presented in Figure 5, revealed a single crack origin on the side of the plaque wall opposite the impact surface. This surface was under tensile loading through the impact. The origin area displayed a relatively smooth texture. The zone surrounding the crack origin showed a more coarse morphology and exhibited a series of U-shaped ridges having the general appearance of river lines, as illustrated in Figure 6. River lines are generally produced through a mixed stress field of Mode I crack opening stress and Mode III anti-plane shear stress. These indications showed a moderate level of stretching, and represented the initiation of stresses other than uniaxial loading. These markings were formed through the coalescence of multiple individual cracks, and the observed stretching was indicative of micro Approximately half way through the test ductility. specimen wall, the morphology presented a high level of folding consistent with the compressive loading and mechanical damage sustained by the impacted surface.

# Hydrolysis

A specimen machined from a molded plaque was secured into a three-point bend fixture and submersed in deionized water at 90 °C. Using the equation for maximum strain for a three-point bend from ASTM D 790:

$$r = 6Dd/L^2$$
(1)

where:

- r = maximum strain in the outer fibers, mm/mm D = maximum deflection of the center of the beam, mm d = sample thickness, mm
- L = support span, mm

it was calculated that the fixtured sample was under 3% strain. After aging for approximately 50 hours, indications of crazing were observed within the sample. Continued aging resulted in catastrophic failure after 250 hours. The sample showed no evidence of macro ductility, given the absence of permanent deformation and stress whitening.

The SEM evaluation revealed multiple apparent crack origins along the outer diameter bend surface, as presented in Figure 7. The crack surface within the origin area exhibited a coarse morphology, suggestive of molecular degradation of the polycarbonate resin. The morphology within the origin area was consistent with crack nucleation through crazing. Between the individual crack origins, crack union ridges, showing minimal micro ductility, were evident. Stretched hackle marks were also evident within isolated locations adjacent to the origins. Immediately outside of the crack initiation sites, radiating indications were apparent. These indications, as shown in Figure 8, consisted of alternating patterns of coarse surface texture separated by smaller concentric rings having a very smooth morphology. A band of stretched, torn material was present between the two morphologies. The distance between the concentric bands increased progressively away from the crack origin.

The nature and cause of these marking are not completely apparent. Although similar in appearance, they are not thought to be present as Wallner Lines. Wallner lines are normally associated with relatively rapid crack extension and correspondingly high energy dissipation. The cracks within the hydrolysis sample extended over a relatively long period of time, characteristic of slow crack growth. As such, the observed features are thought to represent progressive propagation through alternating cycles of cracking and arrest. It is thought that this mechanism resulted through crack extension exclusively within the damage zone immediately preceding the crack tip. The damage zone is under a state of severe stress concentration and craze formation, and would be highly subject to the effects of the hot water. Given this scenario, the crack would arrest once it extended beyond the damage zone, and the process would begin again. The increase in band width with increasing distance from the crack origin supports this hypothesis, and is commonly seen in other progressive failure mechanisms, such as cyclic fatigue.

Adjacent to the inner diameter bend wall, representing the final 10% of the sample wall, the fracture surface showed raised flaps resembling river marks. These features correspond to final load failure and represent a change in the crack extension mode through simultaneous tensile loading and anti-plane shear loading. The features indicate rapid, unstable crack extension through this region.

#### **Environmental Stress Cracking**

A strip sample machined from a molded plaque was fastened into a three-point bend fixture as to impart a strain of 1.5%. The fixtured specimen was submersed in mineral spirits, a known environmental stress crack agent for polycarbonate<sup>2</sup>. After approximately 24 hours of exposure, crazing was apparent with the sample. Periodic inspection revealed that multiple cracks extended through the sample. After aging for 336 hours the sample exhibited numerous cracks, with the largest extending approximately 75% through the sample wall. At that point a manual load fracture was created within the sample. The specimen showed no indication of macro ductility.

Examination of the fracture surface via SEM revealed multiple distinct crack origins along the outer bend wall, as shown in Figure 9. The observed features indicated that the crack nucleated through craze formation, as indicated by the presence of stretched flaps corresponding to the unions between the individual cracks, and the unique morphological pattern surrounding the crack origin zone. Immediately outside of the origin area, the fracture surface presented a series of radiating indications, generally similar to those present on the hydrolysis fracture surface. However, at high magnification it was apparent that the light concentric bands consisted of craze remnants, as illustrated in Figure 10. This pattern indicated that the cracking propagated in a progressive fashion, through repeated cycles of craze formation, cracking and arrest. The separation between the progression bands extended with increased distance from the crack origin area. Approximately 75% through the sample wall, the fracture surface changed drastically. A high concentration of river markings and hackle marks were present, corresponding to the manual overload of the sample. These features were indicative of rapid and unstable crack growth.

# **Cyclic Fatigue Loading**

A machined ASTM D 638 Type I tensile specimen was evaluated through cyclic loading using an MTS IIm TestStar load frame system at 22 °C. This evaluation was conducted in load control with a triangular waveform. The loading was tension-tension at 1.0 Hz. From 70 to 3750 psi. Catastrophic failure occurred within the test specimen after approximately 10,000 cycles. The cracked specimen displayed no signs of macro ductility.

Upon SEM examination, multiple individual crack origins were apparent along the edge of the specimen. Distinct crack union ridges, displaying minimal stretching, were observed between the origins. A crack origin area is presented in Figure 11. The surface within the crack origins exhibited an extremely smooth morphology. A semicircular band was present surrounding the origins. At high magnification, this band displayed distinct micro ductility, as indicated by the presence of stretched fibrils. Within the mid-fracture zone, arrest markings were evident. At high magnification, the arrest markings were differentiated into striations, as shown in Figure 12. The striations appeared to consist of a large band having minimal micro ductility, separated by a smaller band displaying a very smooth surface morphology. With increasing distance from the crack origin, the arrest markings became wider, indicating extended crack propagation per cycle.

Remote to the crack origin locations, the fracture surface exhibited features characteristic of uniaxial tensile loading. This is consistent with the final load failure of the test specimen.

# Discussion

Based upon the SEM evaluation of laboratory fracture surfaces created through different mechanisms, it is evident that it is possible to characterize and identify the failure mode of fracture components through a thorough fractographic examination. The morphological features observed during an SEM examination can be used as a roadmap to guide the trained analyst to discover several important factors in a failure investigation including: the location of the crack origin, the mode of crack initiation, the mechanism of crack extension, and the nature of the stress and environmental factors that precipitated the failure.

As part of this evaluation it was apparent that in spite of the generally ductile macro features observed on the specimens created through uniaxial tensile loading, impact loading, and tear loading, the features observed during the SEM evaluation showed minimal micro ductility. While stretched flaps were evident on several of the fracture surfaces, the extent of deformation was relatively small and apparent only at relatively high magnification. This corresponds to the reported anomalous behavior of polycarbonate during impact testing. It has been observed that polycarbonate will show almost no energy dissipation beyond the maximum load point during instrumented falling dart impact testing.<sup>3</sup> The lack of micro ductility and the anomalous impact properties are thought to be related; resulting from a chain disentanglement mechanism unique to polycarbonate.

The fracture surfaces produced via the mechanical processes - tensile, tear, impact, and fatigue - showed features generally consistent with the respective imparted stresses. However, the failures created through chemical anomalous contact produced somewhat results. Specifically, both of these failure modes produced fracture surfaces that exhibited indications associated with progressive crack extension. The progressive nature of the failure mode is in contrast to the static stresses placed on the parts. It appears that the progressive propagation was related to the effects of the water and mineral spirits on the damage zone immediately preceding the newly formed This pattern of alternating crazing, crack crack tip. extension, and arrest, produced the observed arrest markings and striations.

The current work has raised questions regarding the mechanisms pertaining to the extension of cracks formed through crazing. Additional investigations are being undertaken to further evaluate such fracture modes. Experiments utilizing varying levels of strain in conjunction with aggressive and mild crazing conditions will be performed to assess the relative effects of the applied strain and the chemical agents on the fracture surface morphology.

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#### References

1 Explorer - Engineering Polymers, http://www.sricbi.com/Explorer/Eparchive.smtl (accessed November 20, 2003)

2 <u>Chemical Resistance Volume 1 - Thermoplastics</u>; Plastic Design Library: New York, NY, 1994; p. 399

3 Sepe, Michael P. Dickten & Masch Manufacturing Co. Private communication, 2003

#### Keywords

fractography, polycarbonate, failure analysis, brittle fracture



Fig. 1 - Electron image showing the crack origin area of the polycarbonate specimen crack via uniaxial tensile loading.



Fig. 2 - Electron image illustrating the presence of hackle marks on the tensile fracture surface.



Fig. 3 - Electron image showing the crack origin area on the fracture surface created through tear loading.



Fig. 4 - Higher magnification view of the central potion of Figure 3 showing characteristic V-shaped features on the tear fracture surface.



Fig. 6 - Higher magnification view of the left central portion of Figure 5 showing distinct U-shaped ridges, similar to river lines, on the impact fracture surface.



Fig. 7 - Electron image showing the crack origin area on the hydrolysis polycarbonate specimen.



Fig. 5 - Electron image illustrating a portion of the impact fracture surface.



Fig. 8 - Electron image illustrating the presence of concentric bands outside of the crack origin area.



Fig. 9 - Electron image showing the crack origin area on the environmental stress crack fracture surface.



Fig. 10 - Electron image illustrating the presence of concentric bands of craze remnants on the environmental stress crack fracture surface.



Fig. 11 - Electron image showing the area of crack initiation on the fatigue test specimen.



Fig. 12 - Electron image showing fatigue striations remote to the crack origin location on the fatigue test specimen.