ENVIRONMENTAL STRESS CRACKING – EXAMPLES FROM THE AUTOMOTIVE INDSUTRY

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Abstract

Four case studies are presented to illustrate environmental stress cracking (ESC) within automotive components. ESC is a very important mode of plastic component failure. The presented cases illustrate how the failure analysis process was used to identify the failure mechanism as well as the primary factors responsible for the failures. The four cases depict representative automotive failures involving varied designs and service conditions.

Background

Environmental stress cracking (ESC) is widely regarded as the leading cause of plastic product failure. A recent study indicated that approximately 25% of plastic component failures are associated with ESC[1]. ESC is a failure mechanism whereby a plastic resin is effected by a chemical agent while simultaneously under tensile loading. It is a solvent-induced failure mode brought about by the synergistic effects of the chemical agent and the mechanical stress.

As part of ESC, there is no chemical attack or molecular degradation. Conversely, the chemical permeates into the molecular structure and interferes with the intermolecular forces binding the polymer chains, causing molecular disentanglement.[2] ESC is a brittle fracture mode and the steps involved in the mechanism include fluid absorption, plasticization, craze initiation, crack growth, and finally fracture. Recent comparisons have illustrated similarities between ESC and creep, sometimes referred to as static fatigue.[3] Creep is the application of static stresses, below the yield point, over an extended period of time. The presence of the ESC agent simply reduces the time to failure, or alternatively, reduces the level of stress required to initiate the failure.

Experimental

Surfaces of the exemplar failed components 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 surfaces were gold sputter coated to enhance the imaging.

Samples representing the materials were analyzed using micro-Fourier transform infrared spectroscopy (FTIR) in the attenuated total reflectance (ATR) mode. A Nicolet Magna 550 spectrometer interfaced with a Nic-Plan® IR microscope was used for the analysis.

Sample materials from failed components were analyzed using thermogravimetric analysis (TGA). The testing was performed on a TA Instruments 2950 TGA. The thermal program involved dynamic heating at 20 °C/min. using sequential nitrogen and air purge atmospheres. Material samples were evaluated using differential scanning calorimetry (DSC). The testing was conducted using a TA Instruments 2010 DSC. The analysis involved heating the samples at 10 °C/min. through the transition. Thermomechanical analysis (TMA) was used to evaluate materials from the failed parts. The testing was performed on a TA Instruments 2940 TMA. A heating rate of 5 °C/min. was utilized in conjunction with a 0.05 N loaded expansion probe.

The molecular weights of the failed part materials were evaluated indirectly by melt flow rate. Tests were conducted in accordance with ASTM D 1238 using an appropriate test temperature and constant load.

Tests and Results

Mirror Housing Failure

A set of prototype external mirror housings had failed during a thermal/vibration engineering evaluation. The testing included exposing the assembled parts to extremes in temperature while under vibrational testing. The plastic mirror housings were secured to the mating steel rod with a coated steel clamp. The housings had been molded from a poly(acrylonitrile:butadiene:styrene) (ABS) resin , and this represented a change from the previous polypropylene resin.

The visual examination of the mirror housings confirmed the presence of a substantial level of cracking immediately surrounding the fastening hole. The cracking was most heavily concentrated within the area that had been in contact with the clamp that secures the housing to the pole. A microscopic inspection of the opened fracture surfaces revealed features indicative of crack initiation along the exterior surface of the housings, as shown in Figure 1. Additionally, an oily residue was found covering the fracture surfaces. At slightly higher magnifications, radiating features, suggestive of a progressive failure mode were evident. These features covered approximately 80% of the fracture surface extending from the exterior wall, with the remaining 20% closest to the interior wall showing features indicative of tensile overload, associated with the final fracture zone.

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Representative fracture surfaces were examined via SEM, and the observed features confirmed that multiple individual cracks had initiated along the exterior edge of the mirror housing wall. The morphology within the crack origin zone was relatively, smooth, indicating slow crack initiation. Outside of the origin zone, the fracture surface exhibited signs of moderate micro ductility in the form of stretched flaps. Additionally, the surface outside of the crack origin area displayed a series of radiating features. At high magnification these features were present as bands of opened craze remnants. The fracture surface along the interior housing wall, remote to the area of crack initiation, showed contrasting features. The morphology within this area included a high concentration of stretched fibrils, characteristic of ductile cracking, associated with mechanical overload.

Analytical testing of the mirror housings via FTIR, DSC, and TGA generated results consistent with an unfilled ABS resin. No signs were found to indicate contamination or material irregularities within the mirror housing material. Melt flow rate testing produced results consistent with adequate retention of molecular weight, without degradation from either production or service.

The oily residue present on the fracture surfaces was analyzed using FTIR. The obtained results were denotative of a phthalate-based ester, such as dioctyl phthalate, as presented in Figure 2. The coating covering the steel camp was also analyzed and the results represented a poly(vinyl chloride) (PVC) resin containing a significant level of a phthalate ester, which closely matched the results obtained on the fracture surface residue.

It was the conclusion of the investigation that the mirror housings failed through ESC. The ESC agent in this case was the phthalate-based ester plasticizer present in the PVC coating on the steel clamp used to secure the housing to the mating pole. The plasticizer likely migrated from the coating under the conditions of the engineering testing, including exposure to elevated temperature and the compressive loading inherent to the design. Phthalate esters are known to be aggressive ESC agent in concert with ABS resins. The obvious mechanical stress driving the failures was the interference loading between the mirror housing and the clamp. The failure was identified as ESC through the visual, microscopic, and SEM evaluations. The designative features included multiple crack initiation sites that subsequently coalesced into a unified fracture. Additional features were found within the mid-fracture zone, including a series of radiating markings corresponding to opened craze remnants. Such features are consistent with the ESC failure mechanism in which the crack front alternately forms crazes leading to cracking, followed by arrest. Once the crack progressed to a critical size, final overload occurred. The primary factor in the failure was the incompatibility between the PVC-based coating and the ABS housing. Previously, the parts had been molded from polypropylene, which was not effected by the phthalate plasticizer.

Head Light Lens Cracking

A headlight lens had cracked after a brief time in service. The part was considered to be typical of many other parts that had also cracked in service. The failure rate had remained low, but steady, over the course of several years. The lenses were injection molded from a polycarbonate resin and subsequently covered with an acrylic resin hard coat. It had been discovered that most of the failures had been observed shortly after the cars had been in for unrelated service at the respective dealership.

A visual examination of the headlight assembly confirmed the presence of a high concentration of cracks within the lens, as shown in Figure 3. The cracking was most heavily massed around the area adjacent to the lamp. The cracks were arranged in a network pattern and appeared to be randomly oriented. The cracks presented signs of brittle fracture, with no apparent macro ductility.

A cross section was prepared through a typical area of the lens exhibiting the cracking. Examination of the cross section indicated that the cracking was limited to the exterior portion of the lens. The crack network was primarily present within the hard surface coating, but also penetrated into the lens base material. The cracking ranged from 0.35 to 0.50 mm in depth.

A SEM examination was performed on a typical crack surface. The inspection showed characteristic brittle fracture features. Specifically, the appearance indicated crack initiation at multiple locations along the exterior surface of the lens hard coat as illustrated in Figure 4. This morphology extended though approximately 95% of the coating thickness, with the final 5% showing features associated with overload. The cracking subsequently progressed into the lens base material, where craze remnants were readily apparent at high magnification.

Analysis of the lens materials using FTIR produced results corresponding to an acrylic resin and polycarbonate for the hard coating and the base lens, respectively. The base lens material was tested via DSC and a glass transition temperature of 146 °C was obtained. No evidence was found in the either the FTIR or DSC results to suggest the presence of contamination or degradation. A sample of the base lens material was evaluated using TMA. The resulting thermogram showed linear expansion through 135 °C. Above that, the sample underwent moderate expansion, indicating that the lens was under a modest level of molded-in residual stress as produced. Melt flow rate testing of the base lens material produced results indicating very good retention of molecular weight through the molding process and service.

The failure analysis results indicated that the headlight lens cracked through ESC. The stresses responsible for the failure likely included both molded-in residual stress and external stress from securing the lens into the complete assembly. The analysis of the cracked lens did not produce results pinpointing a responsible chemical agent. However, detailed background information indicated that most of the parts, including the subject lens, had failed shortly after a dealer service call. A review of common operating procedures revealed that many dealers place a protective rubber mat over the front fender, including the headlight, while be serviced. Further, many of the service technicians were instructed to remove any residual grease from the headlights with a solution primarily used as window wiper fluid, which in general contain isopropanol. Thus, two likely sources of the chemical agent include the plasticizer from the rubber mats and the isopropanol from the cleaning fluid. Given this, the principle factor in the failure appears to be the improper handling and/or cleaning of the headlight lens during servicing. The concentration of cracking adjacent to the headlight lamp suggests that the heat generated by the bulb significantly accelerates the failure. The identification of ESC as the failure mode was facilitated by the random, intersecting crack pattern observed during the visual examination, by the presence of multiple crack origin locations, and particularly by the craze remnants seen during the SEM evaluation.

Latch Handle Failure

A high number of latch handles failed after a relatively short time in service. The normal service conditions included periodic actuation of the handles at normal ambient automotive passenger compartment temperatures. The handles were injection molded from a poly(acrylonitrile:butadiene:styrene) / polycarbonate (ABS/PC) blend. The design utilized an integral metallic roll pin and a spring.

Visual and microscopic examinations of the failed parts revealed significant cracking, which was consistent across all of the handles. The cracks were present within the molded boss that secured the roll pin. When the parts were disassembled, numerous additional, non-catastrophic cracks were apparent at similar locations around the boss hole. The crack surfaces, as represented in Figure 5, exhibited features characteristic of brittle fracture, and the crack origins were primarily located adjacent to the inner diameter. An oily residue was evident on and adjacent to the fracture surfaces, and on the mating roll pin, as shown in Figure 6.

Typical fracture surfaces were further examined via scanning electron microscopy (SEM). The SEM evaluation confirmed the presence of multiple crack origins at the inner diameter of the boss hole, at areas that had been in direct contact with the roll pin. The surface morphology within the crack origin areas included craze remnants, suggestive of craze formation as part of the crack initiation mechanism. The fracture surface outside of the origin locations showed features indicative of brittle fracture, including hackle marks and secondary cracking.

Analytical testing of the latch handles using FTIR, DSC, and TGA produced results characteristic of an unfilled ABS/PC blend. No evidence was found to suggest bulk contamination of the material. Melt flow rate testing of the failed latch handles produced results showing excellent retention of molecular weight, with no indication of molecular degradation.

Analysis of the oily residue found on the fracture surface using FTIR produced results indicative of a hydrocarbon oil containing an ester-based additive. The residual oil within the roll pins was also analyzed and the obtained results produced an excellent match with spectra representing the fracture surface residue. Spectral library searching generated very good matches with commercial fluids for metal processing.

The conclusion of the failure analysis was that the latch handles failed via ESC. The chemical agent responsible for the failure was identified as the fluid used in the forming and processing of the metal roll pin, a hydrocarbon oil containing an organic ester-based additive. Ester-based materials are known to be active ESC agents for both ABS and PC resins. The oil had not been properly cleaned from the roll pin, as mandated by the handle manufacturer. This inadequate cleaning is the main factor in the failure of the latch handles. The requisite stress involved in the failure apparently came from the interference fit between the roll pin and the handle boss. The failure was identified as ESC by the visual, microscopic, and SEM examinations. The telltale features included the irregular, but continuous crack pattern, the presence of multiple individual crack initiation sites, the generally brittle fracture surface morphology, and the micro-craze remnants within the crack origin locations.

Ignition Module Base Fracture

Numerous ignition base modules had failed after assembly, but prior to be placed into service. The base components were injection molded from a polycarbonate resin. The parts were subsequently joined with a metallic cylinder assembly.

The failed parts were visually and microscopically examined. Cracking was observed surrounding the area of contact with the metal cylinder housing. The surface of the black base modules also exhibited a series of gray streaks, commonly known as splay. A typical surface of a base is shown in Figure 7. Additionally, an oily film was found covering the bulk of the plastic modules. No signs of macro ductility were apparent on the failed parts, with the fracture surfaces showing features characteristic of brittle cracking.

Several fracture surfaces were evaluated through SEM inspection, with consistent observations. Multiple apparent crack origins were evident along the surface that had been in contact with the mating metal housing. The crack origins showed a pattern of radiating bands interspersed with zones of extremely smooth morphology, as illustrated in Figure 8. At high magnification, the bands were revealed to consist of craze remnants. Surrounding the crack origins, the fracture surface displayed classic brittle fracture features, including hackle marks and river markings.

Analytical testing of the module bases using FTIR, DSC, and TMA generated results consistent with an unfilled polycarbonate. No evidence was found to suggest bulk contamination of the material. However, the DSC and TMA results showed a comparatively low glass transition temperature and softening point, respectively. Correspondingly, melt flow rate testing of the failed module bases produced results indicating significant reduction in molecular weight associated with molecular degradation. Specifically, the nominal melt flow rate of the resin used to produce the failed parts ranged from 9 to 12 g/10 min. The value obtained on the failed parts, 78 g/10 min., represented an approximately increase of 700%.

Analysis of the residue found on the base modules using FTIR produced results characteristic of an aliphatic hydrocarbon oil. No signs of other chemical agents were found.

The conclusion of the failure analysis was that the ignition base modules failed via ESC. The stresses driving the failures likely originated from the interference with the mating metal cylinder housing. This included both the assembly snap fit force and the static stress. The chemical agent responsible for the failure was thought to be the aliphatic hydrocarbon oil found covering the surface of the base modules. This oil was present as residual fluid from the production of the mating metallic components. While such fluids do not normally effect polycarbonate resins, the material was severely compromised by the substantial level of molecular degradation. Thus the degradation was primary factor responsible for the failures. This degradation was evident through the reduced glass transition and softening temperatures, as well as the elevated melt flow rate. The failure was characterized as ESC by the multiple crack initiation sites and the radiating bands of opened craze remnants.

Discussion

The case studies illustrate the diversity of conditions that can produce ESC failures within automotive components. Factors involved in the cited failures included material substitution/selection, improper handling and cleaning in service, inadequate cleaning of mating metal components, and molecular degradation associated with improper molding conditions. At a minimum, this represents design, processing, and service related elements. Care must be taken throughout the product life cycle to guard against ESC. The cases also showed fracture surface features commonly associated with ESC failure. These features included multiple crack initiation sites; brittle crack features lacking apparent macro or micro ductility; a stepwise failure mechanism, indicated by a primary morphology followed by signs of final overload; and the presence of opened crazes, both as isolated features and in radiating bands. Thus, a formal failure analysis can provide information regarding both the failure mode and cause, which can be used to fix the current problem and to prevent reoccurrence.

Acknowledgements

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References

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3 David Wright, Environmetnal Stress Cracking of Plastics, Rapra Technology Ltd., United Kingdom, 1996, p. 35

Keywords

environmental stress cracking, failure analysis, automotive

Fig. 1 - A typical fracture surface from the mirror housing is shown exhibiting features characteristic of ESC.

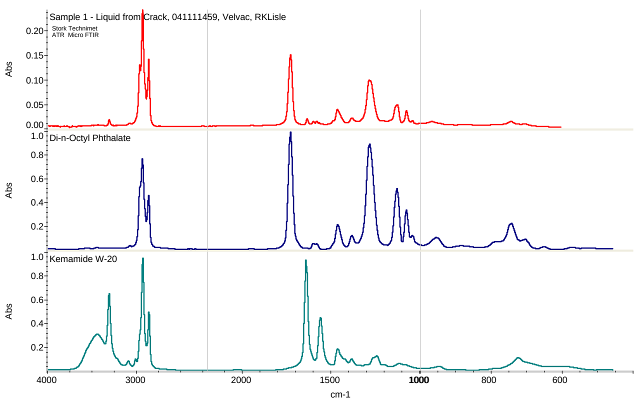

Fig. 2 - The FTIR spectrum obtained on the residue from the fracture contained absorption bands indicative of a phthalate based ester.

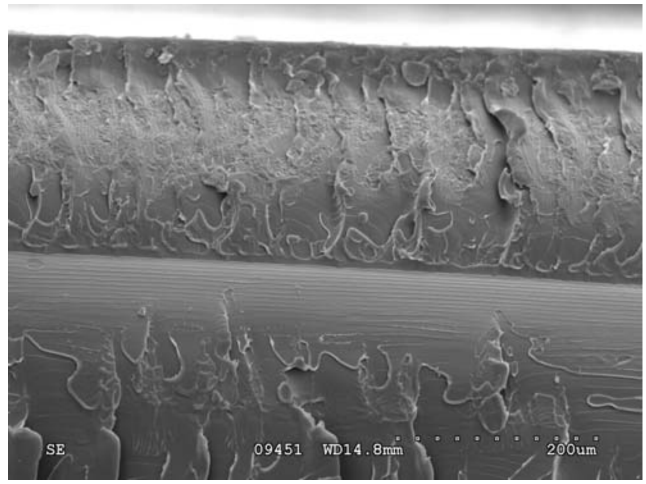

Fig. 3 - An intersecting network of cracks was present on the headlight lens.

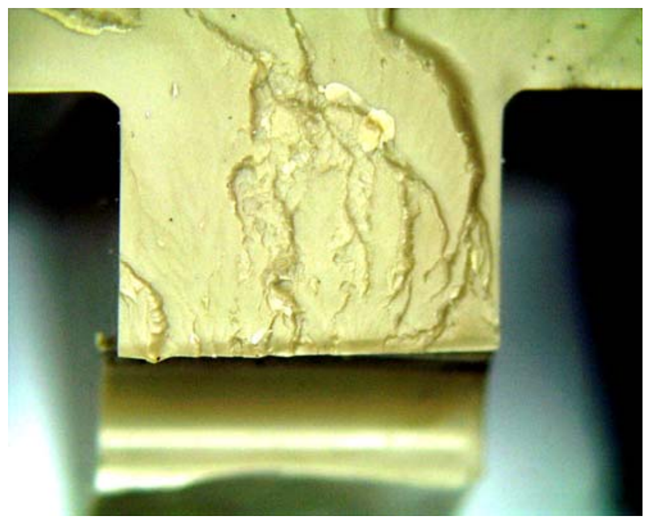

Fig. 4 - Scanning electron micrograph showing the cracking within the lens hard coat and the lens base material. (SEM 150X)

Fig. 5 - A typical latch handle fracture surface is shown displaying features associated with multiple crack initiation sites.

Fig. 6 - A high concentration of oil was present on the

hinge roll pin.

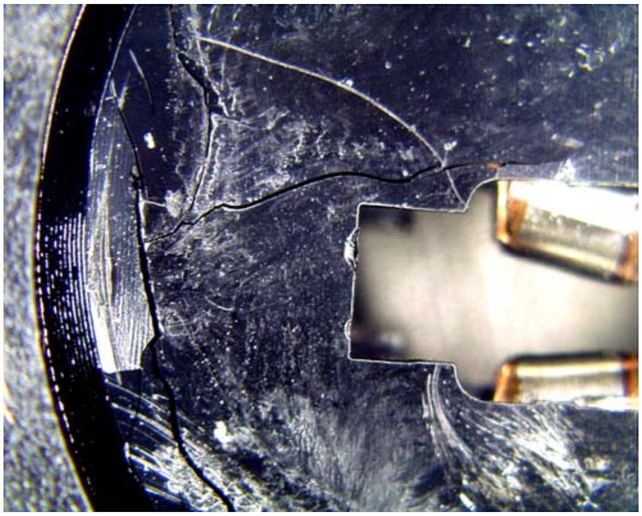

Fig. 7 - A substantial level of crack was apparent on the ignition switch base. Silver streaking associated with splay was also evident.

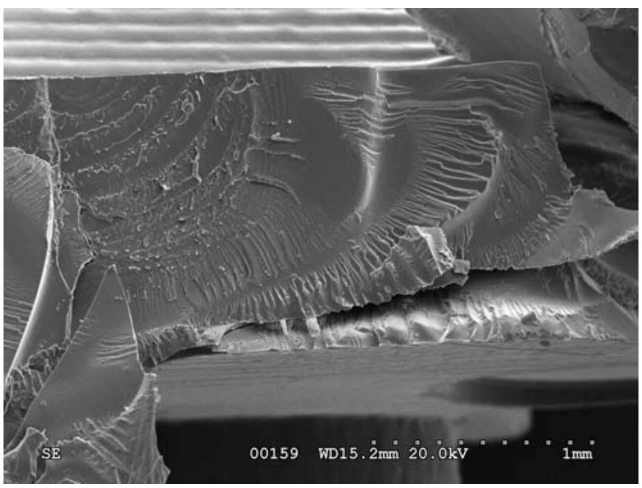

Fig. 8 - Scanning electron micrograph showing the ignition base fracture surface features characteristic of ESC. (SEM 40X)