

## Chapter 9

### Case Studies

# Case Studies of Inadvertent Interactions Between Polymers and Devices in Field Applications

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## INTRODUCTION

Polymeric compounds are selected for a wide range of applications by technical persons with a variety of backgrounds. Initial choices may be moderated by other specialists who are often unaware of the potential pitfalls and adverse interactions associated with the use of cost-effective or inappropriate alternate materials. Manufacturers who provide subcomponents may not be included in the design reviews of finished products into which their components are being used. Additionally, suppliers of commercial polymeric materials may be unaware of how their materials are being applied. As a result of these and other considerations, materials selections may be made based on a review limited to basic engineering properties. Considerations of long-term performance and response to specific operating conditions requires a degree of attention and insight that may be overlooked.

Several case histories are cited in which some aspect of materials selection and design were deficient in the application. A thermally activated electrical switch formerly made with a phenol formaldehyde thermoset resin was redesigned to include a thermoplastic resin. Localized heat associated with the arcing activity of the switch contacts caused thermal erosion of the housing, releasing reactive sulfur compounds which then reacted with the electrical contact faces, causing irregular performance and eventual contact welding. A pressure relief device in a consumer product was found to have highly variable performance as a result of extensive processing aid additions to the base polymer, selection of poor quality raw materials, and no attention to a root cause analysis with a review of the compound. Plasticizers released from PVC wire insulation at elevated operating temperatures wicked

along the conductor strands and onto relay contacts, resulting in a power plant shutdown. Components from a pharmaceutical product container were found to be exuding phthalate compounds which were not expected based on an initial review of the raw materials.

These cases are presented as constructive examples for those seeking to maximize the performance and useful life of devices making use of polymeric components through an integrated materials selection and design approach.

## DECOMPOSITION OF THERMAL SWITCH

A thermal limit switch used in a number of domestic and commercial appliances was historically manufactured with either a ceramic or a crosslinked phenol formaldehyde, providing many years of reliable service. A change in materials had been implemented to facilitate processing, resulting in a housing made with thermoplastic polyphenylene sulfide (PPS). The housing contained silver-laminated bronze electrical contacts, one of which was mounted on a bimetallic arm to provide thermally-controlled switching action. Failures of this switch were encountered wherein the contacts were found to weld together, resulting in a thermal runaway condition caused by a failure to interrupt current to the heater that the switch was intended to control. A forensic review of representative failed switches was undertaken.

Figure 1 presents a scanning electron micrograph of the surface of a contact removed from a failed switch. On the surface, many melted areas are clearly visible. Some of these are flat, showing the previously molten condition of the metal contacts. Metallographic cross-sections through such a contact showed severe localized melting. Elemental analysis of the contact surface indicated that silver sulfide was present. This compound produced an insulating layer on the surface of the contact, resulting in erratic current flow and localized heating due to limitation of the available contact surface area. Switches in various stages of degradation were operated with thermocouples

placed on the contacts and housing. Measurements indicated significant resistive heating, merely due to flow of the rated current. Chemical analysis of the polymer heated to the as-found level, using gas chromatography and mass spectrometry (GC/MS), confirmed formation of hydrogen sulfide, carbonyl sulfide, sulfur dioxide, hydrogen, and methane.

Examination of the switch housing interior surfaces surrounding the contacts revealed significant erosion of the polymer as shown by the light colored oval region in Figure 2. Closer examination

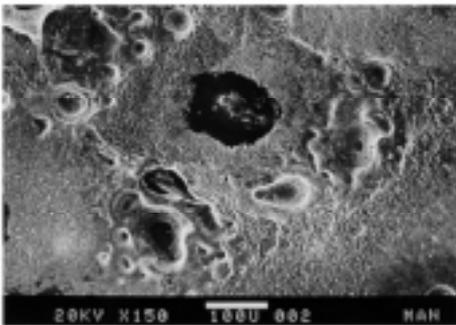


Figure 1. Surface of contact showing raised areas where welding occurred, 150x.

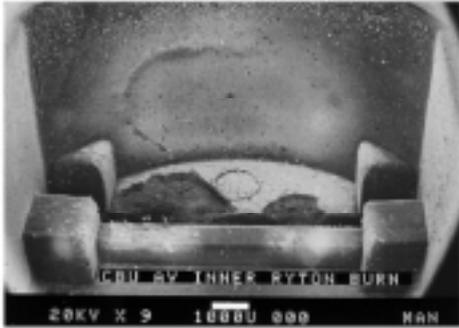


Figure 2. Interior surface of switch housing showing polymer erosion, 9x.



Figure 3. Top view of pressure relief device.

revealed the glass and mineral reinforcement particles within the PPS compound standing in relief, due to polymer pyrolysis. This damage was due to the intense localized heating produced by arcing as electrical contact between the switch contacts was established then broken during normal operation. The combined evidence of contact melting and PPS pyrolysis suggested short-term temperatures in excess of 600°C.

## INCONSISTENT PRESSURE RELIEF MEMBRANE

A pressure relief membrane used in a consumer product was found to exhibit erratic performance both in quality assurance testing and in the consumer market. The pressure relief device was a critical component and played an integral role in product function and safety.

The device was manufactured using a compounded thermoplastic polypropylene which was injection molded into the necessary form. As can be seen in Figure 3 the molded part is quite complex in design; consisting of numerous ribs, radial formations and most importantly, the thin membrane which acts as a pressure rupture diaphragm. The latter is coined in the injection molding process.

Investigation of the device revealed many areas of misapplied designs and a general focus on processing performance instead of functionality. The thermoplastic compound which was used to fabricate the units made use of a fairly complicated formulation. The original base resin was dropped from the supplier's product line and alternates were substituted. In conjunction with these changes, increased device anomalies and difficulties controlling the burst pressure range were experienced. After a preliminary materials investigation of the disclosed formulation, interactions of the materials being used were

identified as being inordinately complex and in some cases inappropriate for this application.

Organic chemical analyses of representative devices were conducted using GC/MS. This method was selected to confirm the identity of the organic ingredients and processing aids in the questionable formulation. GC/MS analysis of the seals revealed significant formulation variations between different lots of material. It was determined that the use of additives such as the antioxidants, antiblocking agents, internal lubricants, and other processing aids was inconsistent. The most significant variations were among materials not specified in the formulation. Processing aids such as silicones (used as internal lubricants to modify flow behavior), plasticizers (typically used for increasing impact resistance and adding flexibility), and waxes (used as lubricants and flow modifiers) were noted to be present in many of the device lots. These components appeared at random and were not used consistently. It was suspected that they were added as on-line processing aids to assist with mixing by the compounding operators and/or to achieve a target melt flow index.

The formulation suffered from years of incremental modification for performance and processing issues which often suppressed the symptoms but never addressed the root causes. For example, there were three agents listed in the formulation which served as antioxidants. Due to the nature of their chemical functionality, these materials did not enjoy a positive synergy. Instead they competed in the formulation causing none of these materials to offer as much protection to the resin and other organic components in combination as they would when used individually. The antioxidant package was further complicated when a review of their functional characteristics was completed. Originally, the molded pressure relief device suffered from a reaction with copper within the contacting unit surfaces. A metal deactivating antioxidant was added to the formulation to correct this problem. A review of the formulation clearly indicated that the original antioxidant was an amine (nitrogen-hydrogen) compound. This antioxidant sustained limited thermal decomposition during processing, leading to the production of amine compounds. These reacted with copper, leading to the formation of blue-colored copper compounds. While the addition of the metal-deactivator was successful in reducing this occurrence, the original antioxidant was left in place. The replacement and original antioxidants were not chemically compatible, nor was the amine antioxidant stable with respect to the antioxidant supplied in the base polypropylene resin. A third antioxidant was then added to improve oxidative stability.

A different problem was noted when a scanning electron microscope (SEM) was used to examine selected areas of representative seals. The high magnification of the SEM provided a view of the relative size of the individual filler particles and their alignment in key areas such as the diaphragm. Examination revealed that the filler materials had a tendency to agglomerate in this region and that the overall filler concentration in the diaphragm area

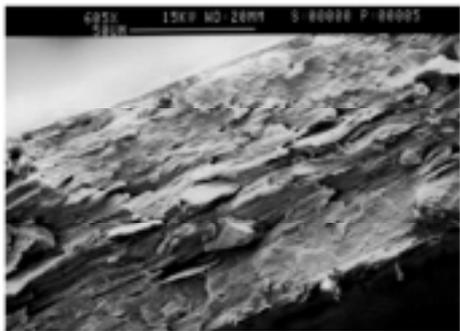


Figure 4. Micrograph of diaphragm cross-section, 605x.

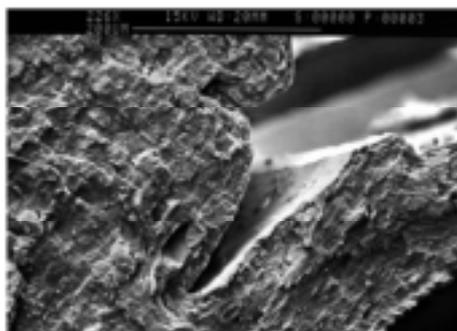


Figure 5. Micrograph of diaphragm corner, 226x.

was inconsistent throughout many devices. As shown in Figure 4, the talc particles were quite large when compared with the overall thickness of the diaphragm. As illustrated in this micrograph, the particles aligned in the plane of the membrane and created a stacking effect. In this case, the shape of the particles was inappropriate due to the flow mechanics in the mold cavity. Figure 5 shows the corner at the edge of a representative diaphragm. The filler particles in this area were also dramatically aligned along the curvature of the diaphragm. This suggested that the resin flow in this area during molding was restricted by the presence of the talc particles. This caused the residual stresses in the diaphragm area to be quite high and the particle size of the talc to vary depending on the level of flow restriction during injection.

The effect of the talc particle size variation on the inconsistent performance of the seals was significant. This characteristic mainly affected the flow rheology of the compound under high shear conditions during injection molding. The talc particle size, in comparison to the diaphragm thickness, also led to an erratic influence on the tear characteristics during product performance. Talc agglomeration and absence of bonding with the base polymer further contributed to poor performance.

Inconsistent diaphragm burst performance was caused by a combination of chemical, physical and rheological phenomena. The lots of devices which exhibited a particularly high burst pressure were the result of a very fine particle size talc in conjunction with a low concentration of processing aids. The increased strength of the base resin and lack of large talc particles for burst initiation necessitated high burst pressures. The devices which exhibited lower diaphragm burst pressures suffered from a combination of large talc particles and an absence of lower molecular weight polymer to assist with the flow and wetting of the filler. This resulted in high orientation effects which led to very high residual stresses causing premature failure. These anomalies illustrate the combined effects of the uncontrolled chemical

additives and random talc particle size on the consistent performance of the compound. In this formulation, even if extreme care were taken in manufacturing, the number of materials involved and the inherent variability and performance of the talc made it virtually impossible to produce a consistent product.

### **PLASTICIZER BLOOM FROM PVC CABLE JACKETS**

During inspections at a nuclear power plant, green liquid deposits were found concentrated on the surface of selected low voltage cables, at their terminations as well as in the instrument panel in which these cables ended at connections. The cables were rated at 600 volts and incorporated a cross-linked polyethylene (XLPE) insulation with a polyvinyl chloride (PVC) jacket. The estimated age of the cables was 20 years. The green liquid deposits were determined to be non-drying, with a high viscosity, and good lubricity. Analysis of this liquid by Fourier Transform Infra-Red Spectroscopy (FTIR) confirmed that it was mostly adipic acid diethyl ester. This compound is a common plasticizer for PVC and is typically yellowish in color. An FTIR absorption peak unaccounted for by adipic acid diethyl ester was assigned to a silicone fluid (diphenylsilane). This may be attributed to a second plasticizer used in these cable jackets. Samples of the liquid were pyrolyzed and the residue was analyzed with energy dispersive X-Ray analysis (EDX). This revealed the presence of copper with traces of aluminum, silicon, calcium, iron, and lead. The presence of copper salts in the fluid was responsible for the noted green color.

The presence of these green fluid deposits closely followed a record 'heat wave' in this particular region. It was deduced that this elevated regional temperature caused the sudden appearance of these exuding plasticizer compounds from the PVC cable jackets. These compounds can cause severe consequences in electrical systems due to their insulating properties. If these compounds were allowed to migrate into electrical switches, relays, or meters they would inhibit proper performance. In this particular case, the plasticizer impinged on the jackets of adjacent cables, causing them to swell then split. In another identical occurrence, a plant shutdown resulted when plasticizer crept onto the surface of electrical contacts used for a pump motor relay.

### **EXTRACTS FOUND IN PHARMACEUTICALS**

The presence of two plasticizers, dioctyl phthalate (DOP) and diisooctyl phthalate (DIOP), in a drug formulation caused significant concern to the pharmaceutical companies since aromatics of this type are under regulatory scrutiny. Investigation into the origins of these contaminants led to analytical review of elastomeric components of the product container. Extensive GC/MS analysis isolated the source of the DIOP as being the elastomer raw mate-

rial. Further research indicated that the supplier of this elastomer was adding DIOP during manufacture to act as a melt-flow modifier to control the Mooney Index of the final product.

The DOP, however, was traced to contamination from the polymer compounding equipment. Frequently, oils used to lubricate mixing equipment exude into the compound being produced through dust seals, for example. Knowing this, manufacturers will often utilize lubrication products which are compatible with the polymer products that are produced. In this case, however, the oil utilized contained DOP which would be acceptable in many thermoplastic compounding applications, not slated for medicinal use. The resultant extraction of DOP from components of the product container, however, was not acceptable.

## DISCUSSION

Development of thermoplastic and thermoset polymer compounds is a mature science that continues to grow with the development of new types of additives, changing regulatory requirements, and proprietary considerations. The selection of all materials that are incorporated into a compound may follow lines that are not always clear. Some ingredients may be outdated. Others may have been added for a customer-specific end use and the compound later became available for the general market. A very wide range of off-the-shelf compounds are available for engineering applications. Many will fit into the existing requirements or designs and/or processes may be altered to accommodate the compound that best fits the needs. These choices, though, are often limited to the general engineering/technical properties without sufficient detailed consideration of the materials in context of the application.

An ideal situation is one in which the end-use manufacturer has available the equipment necessary to develop a polymer compound specifically suited to an individual application. In this clean sheet approach, each ingredient may be carefully considered in context of the application, aging characteristics, processing effects, and synergy with other formulation components. Compounding facilities need not be directly available; contract organizations are available and many of the commercial polymer compound suppliers offer custom compounding services.

Analyses of plastics failures and contamination issues often indicate that it is necessary to return to the basics and re-examine the material in context of the application. With this approach, a polymer would be formulated using a minimum number of ingredients, each of which would be the most appropriate and efficient for the end use. By reducing the number of ingredients, the controls necessary for each supplier are greatly simplified and the potential for adverse interactions reduced. Many raw materials are more complex than may be apparent and, in some cases, the 'hidden' ingredients may be detrimental to an application. Virtually all commercial elastomers are supplied with an antioxidant already included and

the type may change periodically. Master-batching agents and processing aids, such as calcium stearate, may be used when adding antioxidants to a raw polymer. Crosslinking additives and their synergists are another source of antioxidants and other compounds. Crosslinking is a chemically challenging process in which thermal decomposition of a reactive peroxide is typically used to provide free radicals. This requires an additional antioxidant to protect the polymer, while reaction products including acetophenone, cumyl alcohol, and acetic acid become available to interact with the other raw materials or additives.

In the case of the thermal limit switch, the choice of materials for the housing inherently led to a reduction in the useful life of the device. The stability and useful life of the switch could be readily enhanced through the use of a polymeric housing that does not produce reactive gaseous products. Many thermoset materials are available, as are ceramics. While the near-term economy of using a thermoplastic material may have appeared attractive, the long-term effect on performance may not have been readily apparent when a material substitution was made.

In the second situation corrective measures were implemented so that predictable and consistent performance of the pressure relief device could be attained. Compound reformulation took place which included the careful selection of a clean homopolymer base resin, a specially designed and compounded antioxidant and a low aspect ratio, small particle size reinforcement. The compound simplification, in combination with highly functional components, allowed for exceptional performance and reliability.

In the example of plasticizer bloom from a set of cables, it is interesting that the simple loss of a compounding ingredient could lead to such indirect, but major consequences. In this case, exposures to long-term conditions of elevated temperature could be surmised, based on the application and service environment. Grafted plasticizers are available. Alternatively, though, a complete reconsideration of the material in this environment would have been beneficial. A polymer compound that is inherently flexible would not involve a plasticizer and the potential adverse effects of its loss. Finally for the pharmaceutical container component example, reformulation of the raw polymer compound, as well as substitution of machine lubricant with a food-grade aliphatic mineral oil was necessary, followed by substitution of increased purity raw materials, before use of this material could be continued.

## **CONCLUSIONS**

It is important that the total life cycle of polymeric compounds be considered in context of the end-use application. Some basic guidelines can be developed from a review of situations in which the process was not optimized.

The application should be well understood in terms of stresses (thermal, chemical, physical, radiation, etc.). Near- and long-term exposures must be considered.

Review the candidate or existing material with a fresh perspective and careful attention to all raw materials, their quality, and roles.

Simplicity of design facilitates processing, cost control, longevity, and quality assurance. This requires that raw materials be inherently suited for the compound, rather than placing a strong reliance on complex additive packages.

The performance of a compound cannot be limited by processing or post-processing handling. Attention to detail is necessary to assure that the intended and realized formulations are identical.

While some of these suggestions may seem tedious, it is often the case where short-term economy and lack of application-specific insight may lead to significant losses when a poorly selected material fails in service.

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# Case Studies of Plastics Failure Related to Improper Formulation

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## INTRODUCTION

Plastics failures can usually be ascribed to the material, design or processing, or combinations thereof.<sup>1</sup> Previous ANTEC papers by the authors have dealt with all of these factors. In this paper we focus on the formulation or composition aspect of the material in failure. Three of five cases cited involve adhesive failure. In all cases chemical analysis was required to determine if the formulation was a major contributing factor to failure.

## CASE STUDIES

### 1. ADHESIVE FAILURE OF A MULTILAYER FILM

The formulation called for a certain order of films and surfaces for good bonding. Failure to bond well at certain layers was found to be due to improper order of surfaces, so that two in contact would not bond. Simple reversal of one layer solved the problem. The error was found by infrared spectroscopy of surface composition.

### 2. FAILURE OF EPOXY RESIN TO HARDEN

An adhesive using a two-part epoxy resin failed to harden in the field. An analysis showed that the hardener had been omitted, thus absolving the supplier of the adhesive from responsibility. The method of analysis was thermal desorption gas chromatography/mass spectroscopy (TD/GC/MS)<sup>2,3</sup> using the Direct Dynamic Thermal Desorption Device developed at the University of Connecticut by Gary Lavigne. Sample contained in a glass tube, held in place by glass wool, is devolatilized in the heated GC injection port. Typical time and temperature are two minutes at 300°C, after which the sample tube is withdrawn using gas pressure. The tube, held upside down, is in the closest possible direct contact with the GC column, thus eliminating transfer lines or paths. The dynamic feature is that continuous helium gas flow carries off volatiles as they are produced, transferring them to the head of the column at low temperature prior to the usual temperature programming of the GC. The

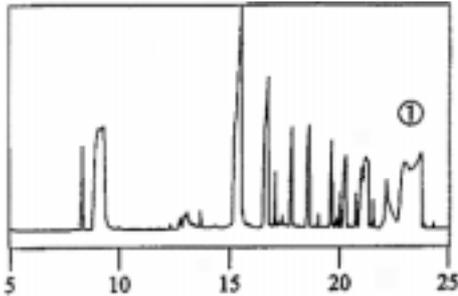


Figure 1. Thermal desorption GC/MS chromatogram of epoxy resin that did not harden. Y axis - ion count; X axis - retention time in minutes. Peak no. (1) - high content of unreacted bisphenol A - diglycidyl ether.

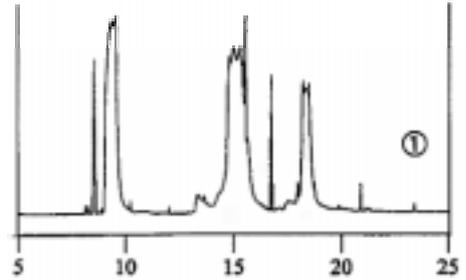


Figure 2. Thermal desorption GC/MS chromatogram of normal two-part epoxy resin that hardened. Y axis - ion count; X axis - retention time in minutes. Peak no. (1) very low content of bisphenol A - diglycidyl ether.

GC/MS used is the Hewlett/Packard 6980, the GC column was SGE PBX5 0.22 mm ID x 25 meters 0.1 micron film. Temperature program was 150°C/minute to 300°C.

Figure 1 is the chromatogram of adhesive that did not harden. It contains a high content of unreacted bisphenol A diglycidyl ether. Figure 2 shows normal two part resin. The diglycidyl ether (unreacted epoxy resin) is gone and other peaks from the hardener are seen. This analysis provided unequivocal proof that the hardener had been omitted by the customer in the field.

### 3. DELAMINATION OF TIN COATING FROM COPPER SHIELD OF AN ELECTRICAL POWER CABLE

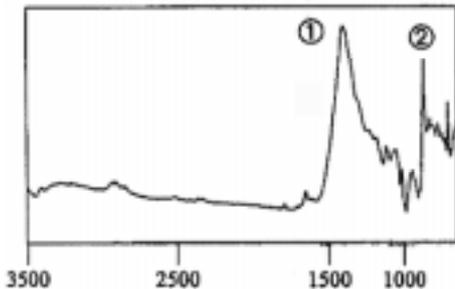


Figure 3. Subtracted IR spectrum of problem tape minus normal tape. Y axis - absorbance; X axis - wavenumbers in  $\text{cm}^{-1}$ . Peaks (1), (2)-calcium carbonate.

Tin coated copper shield over a power cable was held in place by an impregnated fabric tape wrap. Removal of the tape during cable installation caused tin to be removed from the copper shield. Infrared analysis detected calcium carbonate in quantity on both surfaces of the tape, i.e., throughout the tape. Normal tape that did not cause tin to be removed did not contain calcium carbonate. While it is not clear why calcium carbonate would cause the problem, there was no question that it was associated with improper formulation of the tape. Figure 3 was obtained by subtracting the spectrum of normal tape from that of the problem tape. The peaks near 1400 and 900  $\text{cm}^{-1}$  are definitive for the carbonate.

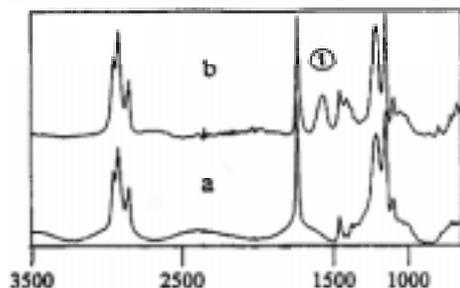


Figure 4. IR spectrum of unused grease (4a) and used degraded grease (4b). Y axis - absorbance; X axis wave-numbers in  $\text{cm}^{-1}$ . Peak no. (1) - new peak in used degraded grease.

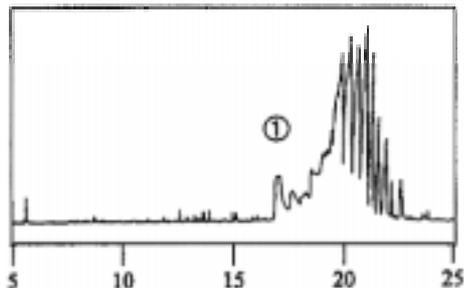


Figure 5. Thermal desorption GC/MS chromatogram of unused grease. Y axis - ion count; X axis - retention time in minutes. Peak no. (1) - triphenyl phosphate (overload).

#### 4. STIFFENING OF GREASE LUBRICATING A MOVEABLE SCREW IN A SERVO MOTOR

This failure occurred during proof testing of a servo motor in development, evaluating the ability of the grease on a moveable screw in the part to provide smooth movement of the screw back and forth. The grease lost its lubricating quality, causing the motor to bind. The grease contained a fluoropolymer, a polyol ester fluid and triphenylphosphate. Infrared analysis and TD/GC/MS revealed that the phosphate content was greatly reduced in degraded grease and carboxylic acids had been hydrolyzed from the polyol ester. The reaction product of hydrolysis of triphenylphosphate is phosphoric acid, the likely cause of hydrolysis of polyol ester. It also contributes to corrosive attack of the metal screw. Elemental analysis of grease showed metal content from the screw.

Figure 4a is the IR spectrum of unused grease, and Figure 4b is for degraded grease. In 4b, the ester band at  $1740\text{ cm}^{-1}$  is reduced relative to the carbon-hydrogen bands near  $3000\text{ cm}^{-1}$ . A new band formed at approximately  $1650\text{ cm}^{-1}$  due to COOH of carboxylic acid. TD/GC/MS provided further insight into chemical changes that had occurred. Figure 5 is for unused grease. Major peaks are for triphenylphosphate (overload) and a mixture of polyol esters. Figure 6 is for failed grease, showing a much lower content of phosphate and new peaks for heptanoic, octanoic and decanoic acids. The latter formed from partial hydrolysis of the polyol ester. The same GC column and thermal desorption conditions were used as in case 2 above. Figure 7 shows TGA thermograms of unused and degraded grease. The weight loss at  $500\text{--}600^\circ\text{C}$  in 7a is the fluoropolymer. Inorganic ash content was 2% for unused, and 15% for used grease. The higher ash content was shown to contain elements from the screw.

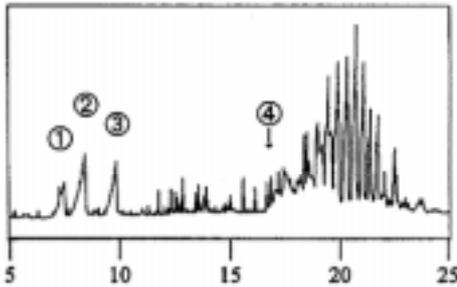


Figure 6. Thermal desorption GC/MS chromatogram of used degraded grease. Y axis - ion count; X axis - retention time in minutes. Peaks (1), (2), (3) - heptanoic, octanoic, decanoic acids; peak (4) - greatly reduced content of triphenylphosphate.

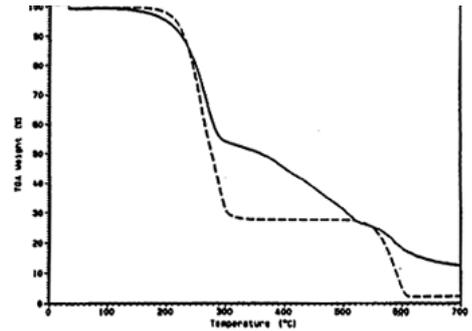


Figure 7. Thermogravimetric analysis of unused grease (---) and used degraded grease (—).

This case illustrates how an additive (triphenylphosphate) can change in service giving a new material (phosphoric acid) which can drastically affect performance. IR and GC/MS were essential in revealing the chemical changes that are responsible for this failure.

## 5. UNINTENTIONAL COLOR IN A PORTION OF AN ELECTRICAL CABLE

PE coaxial cable had unintentional color which faded out after several feet. Concern about possible effect on electrical performance led to attempts to determine the source or cause of the color. Contamination with regrind PE from another product was suspected.

Infrared showed no difference between colored and normal PE. Thermal desorption GC/MS at 300°C also showed no difference. These analyses indicated that the material responsible for color was probably present at very low level. While that provided some assurance that the effect on electrical properties would probably be low, a more positive identification of the contaminant was needed to satisfy the customer and the manufacturer.

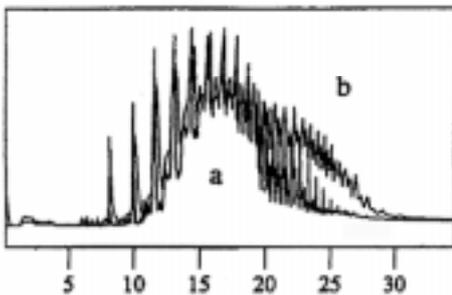


Figure 8. Thermal desorption GC/MS chromatogram of supercritical fluid extract of normal PE (8a) and colored PE (8b). Y axis - ion count; X axis - retention time in minutes.

Supercritical fluid extraction was performed to help isolate from the PE whatever was causing the color. A simple device made here was pressurized to 6000 psi with carbon dioxide at room temperature. 77 mg of PE, first ground to small particle size, was extracted. The CO<sub>2</sub> extract was deposited directly in thermal desorption glass tubes. TD/GC/MS at 350°C gave the chromatograms in Figure 8. Figure 8a (colored PE) shows material at 20-30 minutes GC retention time which

is not present in normal PE. Mass spectral analysis found diglyceride of palmitic acid, and other fatty esters, in the colored PE extract.

The weight percent of material from the contaminated colored PE was so low that only by concentrating it by supercritical extraction was it possible to detect it by GC/MS. Subsequent analysis of candidate recycled PE containing the fatty esters found in the cable in question provided a means of locating the source.

If the contaminant PE had not been colored, its presence would not have been known. While the chemical nature of the contaminant proved to be no problem to electrical properties, the manufacturer had no choice but to try to locate the source of the contaminant, be it serious or not.

## CONCLUSIONS

Infrared spectroscopy has been used for many years as a key method of analysis to aid in determining causes of failure and variable performance. This paper gives examples of the utility of IR. It gives spectra or fingerprints of the whole material. Without separation of components, so that each can be identified, IR is limited in the information it can furnish. TD/GC/MS has become as important, if not more important, than IR. By separating a mixture into components, and providing mass spectra of each, details of composition are learned that IR alone cannot provide.

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# Translating Failure Into Success – Lessons Learned From Product Failure Analysis

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## INTRODUCTION

Given the large number of plastic product failures that have occurred over the years, one would expect that, at least among products of similar complexity, the number of failures should be reduced with time simply from gaining experience with them. Although this may be true for a single manufacturer, it does not seem to translate across the industry as a whole; certain failure modes tend to repeat themselves with undesirable frequencies. From the academic standpoint, many researchers have developed tools in the areas of fracture mechanics, stress relaxation, physical aging, fatigue, and friction. Unfortunately, the average plastics designer does not have access to these sophisticated and sometimes cumbersome techniques. The objective of this paper is to describe some common failure modes and

present them in a simple fashion that should enable the reader to learn from others' mistakes. The reader should note that, in the interest of privacy, some of the case studies have been made generic.

## THE DESIGN PROCESS

The design process is sometimes illustrated as a circular one (Figure 1) which involves conceptualization of a product, construction of a prototype, evaluation and testing of the prototype, and return to the conceptualization/design phase to correct any deficiencies discovered during testing. Clearly, this is a great simplification, with each one of these phases representing multiple steps.

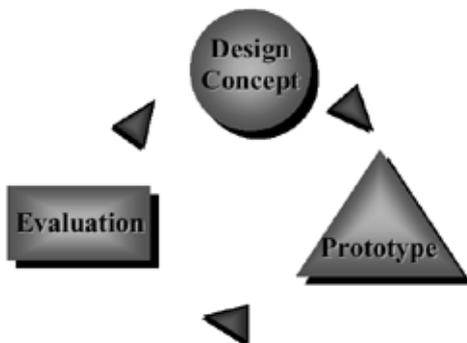


Figure 1. Schematic of a typical process used in design of plastic components. The product is conceptualized, analyzed, sized and then fashioned into a prototype. The prototype is tested and evaluated and any anomalies revealed are corrected such that the process continues until the final commercial product is realized. Re-design and improvement may continue through the product cycle as appropriate.

For example, in order to design and build a prototype, one must come up with dimensions that satisfy the relevant design criteria.

A common basis for sizing a part involves its capability to support stress. When considering the stresses on a part, one must evaluate the residual or built-in stresses from processing, as well as external or applied stresses. The sum of these stresses (the total stress) should be kept less than the working stress, a value often supplied by a resin supplier which is less than the strength of the material and considers the operating environment and type of load. By way of example, a certain resin may have a strength of 70 MPa under ambient conditions. Under elevated temperatures and continuous application of load, the supplier may recommend limiting the working stress to 17.5 MPa. A common error in this early design phase is to size the part based on external loads only and limit the applied stresses to the allowable working stress; ignoring the contribution of residual stress can potentially have catastrophic effects. The designer is left little recourse in the event of a product failure, as the material is now being used in a manner inconsistent with the supplier's recommendations; allowable working stresses have been exceeded.

Evaluation of stress in and of itself can also lead to problems in finished products. With the advent of fast personal computers and inexpensive finite element analysis (FEA) codes, designers can be presented with tools that can, in the absence of accurate inputs, give misleading results. A common mistake is to use published mechanical property data as input to the FEA. The published data may be fine for a comparative analysis between different resins or grades, or perhaps for examining the relative effects of a geometry change. However, accurate results are more readily attained by measuring the mechanical properties of coupons excised from the part itself; it is well known that processing can affect material behavior. A proper FEA model will also consider the anisotropy in mechanical properties which can develop in certain types of processing. All too often, this variation is ignored and unexpected failures result.

The testing phase of the design process is also one that must be carefully executed. The type of loads which a component may experience (i.e., impact, fatigue, thermal, etc.) must be considered. Although it is impossible to envision every loading scenario, those which are reasonably expected in the service environment should be evaluated. This type of testing is sometimes called end-use testing, and involves subjecting a component to the loads, load cycles, temperatures, and environments (i.e., chemical exposure or UV) that it may experience during its life. Often one or more of these variables is changed to accelerate the time of which the end-use test is conducted. We have examined many failures which could have been identified and eliminated through proper end-use testing.

Accurate definition of the environment in which the component is end-use tested is also important. It is simple to say that a part will be used outside and therefore should be

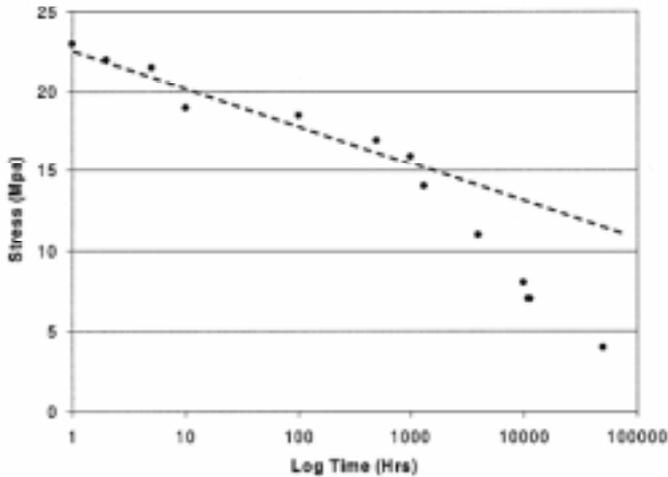


Figure 2. Schematic of curve used to evaluate strength of a product later in its service life. If product is only tested to 1,000 hours and then extrapolated to 100,000 hours as shown, the knee in the curve is missed and strength is overestimated.

time to failure. Higher stress levels fail at lower times and, after application of a safety factor, an extrapolated time is used to define a limiting stress level. More than one pipe has failed because the tester did not consider the change in material behavior, and has extrapolated data erroneously as shown in Figure 2. The standard test methods attempt to limit this kind of problem by forcing the experimenter to have sufficient data for a statistically valid extrapolation.

One approach to defining a testing program is to evaluate the relative risk of different failure scenarios using a failure modes and effects analysis (FMEA). The product of the FMEA is a table which quantifies the relative risk of different scenarios and aids the designer in choosing which modes to examine.

## LEARNING FROM FAILURES

Even the most thoroughly designed and evaluated products can experience failure. Proper analysis and examination of field failures can allow the designer to determine if the failure was from a one-of-a-kind unexpected event, or perhaps a load case that was not considered and should have been. If a program is initiated which tracks incoming field returns, repetitive failures can be discovered and remediated. In products that are related to consumer safety, such programs are a necessity; if the data are not acquired gradually over time, gov-

tested with UV exposure, but what about more abstract issues such as the material with which the part will be cleaned? Could it be exposed to adverse environments during shipping or storage? If the part is available in different colors, could color be a factor in the temperature the part experiences in service?

Additionally, what type of testing should be done to determine the useful life of a product? One method employed in the plastic pipe industry, and described in standard test methods, is to test the part at different stress levels and measure the

ernmental agencies will likely require compilation in a rapid, crisis type mode in the event of a recall.

Tracking programs can vary from simple to complex, but at a minimum should attempt to capture demographics, the user's description of the failure, actual versus intended use (in terms of environment and loading), and the manufacturer's assessment of the cause of failure. If these failure modes are ones captured in an FMEA, the results of that analysis can be modified to reflect new failure frequency data, and any remedial measures can be imposed to aid in overall risk reduction.

Although we do not formally track all of the plastics failures we have examined, we have seen some repetitive themes. In terms of load cases that are not properly considered by the designer, one of the most common theme that appears in our failure analysis activities is thermal expansion. When selecting plastics for replacement of metals, the fact that plastics typically move an order of magnitude more than metals must be considered. This also becomes an issue when plastics are rigidly attached to metals; if one restricts the contraction that plastics undergo with cooling, tensile stresses and cracks may develop. Restriction of movement during heating often causes plastic components to buckle. These general rules can be reversed, however, with composite materials, which can have small or even negative thermal expansion coefficients.

The issue of flame retardants in plastics is also one that seems to be prevalent today. Flame retardants are essential in certain applications, but are not universally appropriate in all plastics. For example, polyolefins are known for their propensity to burn once ignited -a property that can be greatly reduced by the addition of a flame retardant package. In some cases, though, polyolefins are best used without flame retardants even in applications with known risk of ignition. These include conditions where the retardants can compromise key properties, or where the product is protected from burning by monitoring or fire-suppression equipment.

Flame retardants also do not prevent combustion in all cases. Flame retardant polymers can burn readily if the geometry of the part is suitable for combustion; end-use evaluation on the final component rather than on test coupons can be especially relevant in burning characteristic studies.

## **CASE STUDIES**

### **COMPONENT HOUSING**

An ABS component housing was found to be cracking with a failure rate of about ten percent. Fracture surface analysis showed that the failures initiated on the inside of the part, thus reducing the concern for external environmental effects. When the interior components were examined, it was found that some of them were metallic and rigidly attached to the

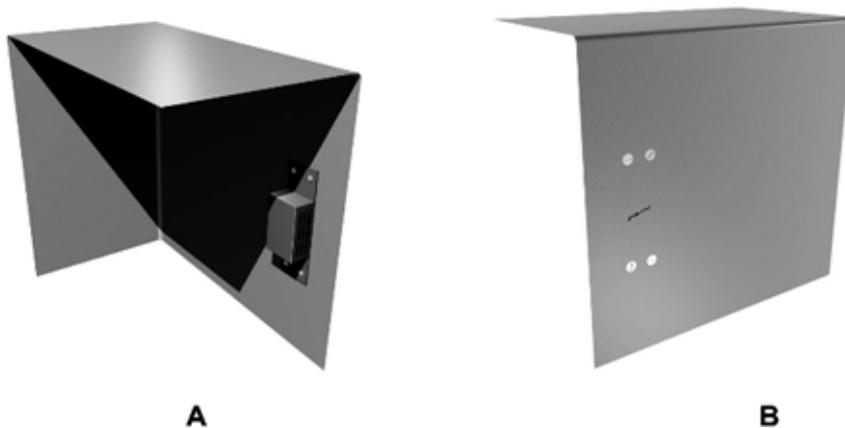


Figure 3. Graphic illustrating failure in a component housing. The metal box, shown in the right side of 3A, was rigidly attached to the housing with 4 screws as seen in 5B. When the part was cooled, restricted contraction of the ABS housing caused tensile stresses to develop that lead to subsequent cracking.

housing. Subsequent experiments revealed that rapid cooling of the housing could reproduce the observed cracking; the metal was restraining thermal contraction of the plastic, causing elevated tensile stresses and cracking (Figure 3). Proper consideration of thermal stresses and end-use testing would have identified and eliminated this failure mode.

### BRAKE CUP

A rubber cup used as the primary seal in a truck master cylinder was found to be torn after inspection following a severe accident. It was contended that the cup was defective and contained manufacturing defects. Fracture surface analysis revealed a rapid fracture and severe component wear. A key tool in successfully the cup's performance was an FEA that showed the stresses in the cup under use were inconsistent with failure scenarios proffered by others. Even though the cup was quite small, coupons were excised from a similar one and tested for mechanical properties that were input into the FEA. The actual, measured properties were different than the published ones and thus were critical to developing an accurate and representative model.

### COMPOSITE PRESSURE VESSEL

A filament wound fiberglass self contained breathing apparatus (SCBA), with a service pressure of about 31 MPa, ruptured during storage in a fire truck. Inspection of the vessel revealed a regular cracking pattern consistent with stress corrosion behavior (Figure 4). Chemical analysis of the surface showed the presence of acidic (low pH) components found

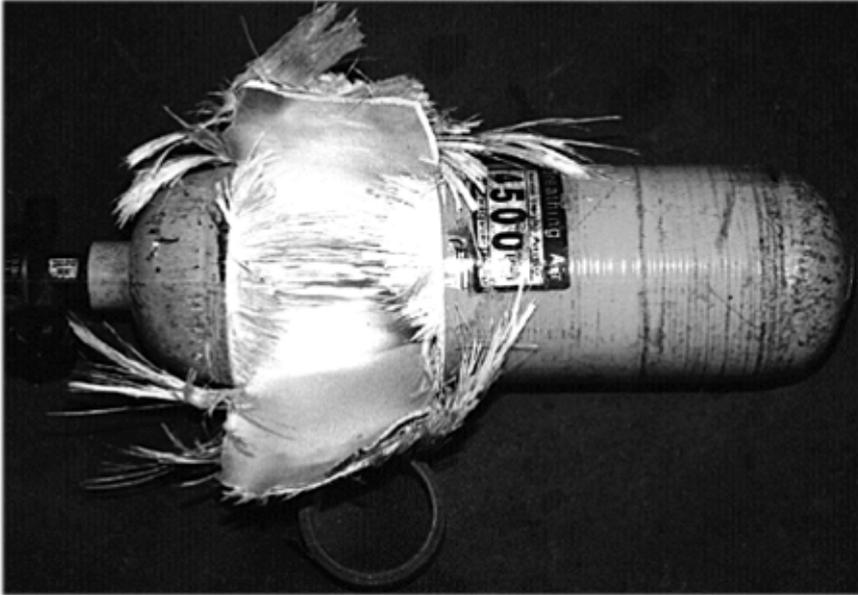


Figure 4. Photograph of stress corrosion cracking in filament wound fiberglass pressure vessel with an aluminum liner. The fiberglass overwrap failed from stress corrosion cracking after exposure to an acidic substance. Many designers are unaware of the susceptibility of fiberglass to acidic environments.

in aluminum rim cleaners. These compounds can severely stress corrode glass fibers under stress. Many designers are not aware of such stress corrosion phenomenon; we have seen this type of failure several times in the last few years.

## SWAMP COOLER

A plastic swamp cooler was designed and constructed by a manufacturer who sought market differentiation from metallic coolers; the plastic device would not rust. The cooler was made from polypropylene and contained an internal pump that was known to fail with regular frequency. In one case, this pump failed and caused the cooler to ignite and initiate a subsequent house fire (Figure 5). This cooler was unmonitored on the roof of a house and contained a known ignition source. It thus represented a perfect candidate for addition of flame retardants; end-use testing would have likely revealed this to the manufacturer.



Figure 5. Polypropylene swamp cooler 1 minute and 44 seconds after ignition from an external flame source (A). The lack of flame retardant in this product lead to the house fire shown in B.

## SUMMARY

Examination of many different plastic component failures has revealed some recurring themes. As a simple tool, we present the pneumatic REDUCE to remind designers of some actions that may help reduce failures in their components:

**Re-Evaluate** – If failures come in from the field, determine the cause, track the frequency, and make changes if necessary. Learn from your mistakes and those of others.

**End-Use Test** – Prior to commercialization, evaluate the product under expected service conditions. Do not use the consumer as the test bed.

**Design** – Invoke a cyclic design process that allows for product improvement before commercialization. Establish proper design parameters when sizing a part. For example, don't size the wall of a component solely on flame resistance if stress is the driving force.

**Understand** – Know how your finished product will be used in service. Is it a component of complete unit? Will it be attached to something that may restrict its movement? Will it be subjected to cyclic (fatigue) loads?

**Calculate** – What are the stresses on your part? Consider residual as well as applied stresses. If you don't have the tools to calculate stresses, try to measure them. Immersion of molded parts into appropriate solvents can reveal areas of high residual stress. End-use testing can address applied stress concerns.

**Environment** – What kind of conditions will the part be exposed to? Don't forget to consider thermal as well as chemical effects.