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Using Fractography to Investigate Causes of Material Failure in Sulfone Polymers

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Fractography is defined as the study of the fractured surfaces of materials. It is routinely used to determine the cause of failure in engineering structures by studying the characteristics of a fractured surface. It can also be used in a more fundamental manner to develop and evaluate theoretical models of crack growth behavior.

Fractography can be used as a quick and simple procedure to determine the root cause of material failure in plastics. Solvay Specialty Polymers has used this technique to study its high-performance plastics, including its broad range of sulfone polymers:

- Udel® polysulfone (PSU)
- Veradel® polyethersulfone (PESU)
- Radel® polyphenylsulfone (PPSU)
- Acudel® modified PPSU

This document summarizes findings from an assortment of evaluations of Solvay's sulfone polymers conducted over several years. It also discusses typical surface patterns that could be observed in broken plastic parts as related to part loading type.

Fractography Description

One of the aims of fractographic examination is to determine the cause of failure. Different types of crack growth (e.g. fatigue, stress corrosion cracking, and excessive loading) produce characteristic features on the surface that can be used to help identify the failure mode. Most of the time, the overall cracking pattern is more important than a single crack.

Fractography methods

Initial fractographic examination is commonly carried out on a macro scale using low power optical microscopy and oblique lighting techniques to identify the extent of cracking, possible modes and likely origins. Optical microscopy is often sufficient to pinpoint the nature of the failure and the causes of crack initiation and growth.

In some cases, fractography requires examination at a finer scale. This is usually carried out using a Scanning Electron Microscope (SEM) because its resolution is much higher than that of an optical microscope. Samples are examined in a partial vacuum and color is absent.

The SEM is especially useful when combined with Energy Dispersive X-ray spectroscopy (EDX), which can be performed in the microscope, enabling very small areas of the sample to be analyzed for their elemental composition. One disadvantage of the SEM approach is related to its high resolution. Only a very small area can be analyzed and the fracture pattern can rapidly become very complex to interpret.

Methodology

To carry out an effective analysis, it is recommended to first perform a general analysis of the fracture surface in order to get a general overview. As described later in this document, a localized analysis that doesn't take into account the entire damaged zone can result in wrong conclusions. Optical microscopy is the most appropriate tool for the initial screening in order to determine the type of failure and to locate the initiation area. Increasing magnifications can be used to better identify and characterize the deformation zones.

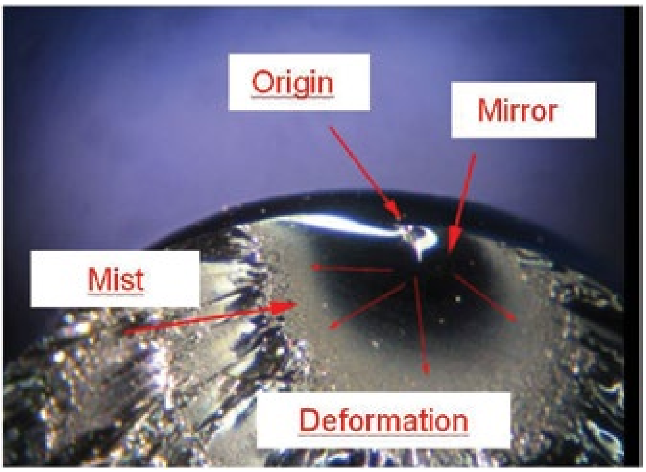
As a second step, SEM coupled with X-ray spectroscopy can be used to better analyze the initiation zone and characterize it. Examples provided in this document are restricted to optical microscopy analysis, since this technique can be rapidly implemented and used in a QC laboratory.

Fracture Pattern in Polymers

A typical fracture pattern for polymers is shown in Figure 1 and includes the following zones:

- Mirror zone: smooth and flat surface made of very small crazes very localized around the initiation point. This region is characterized by a slow crack growth velocity and its size is inversely proportional to the square of the stress at fracture.
- Mist zone: flat smooth area surrounding the mirror region that shows a slight change in surface texture. It is a transition zone from slow to fast crack growth.
- Deformation zone: texture of this area is directly related to the type of loading and the applied stress. It can include hackles, striations and beach marks. The analysis of this region is critical for determining causes of failure.

Figure 1: Typical fracture patterns in polymers



Applications to Polysulfones

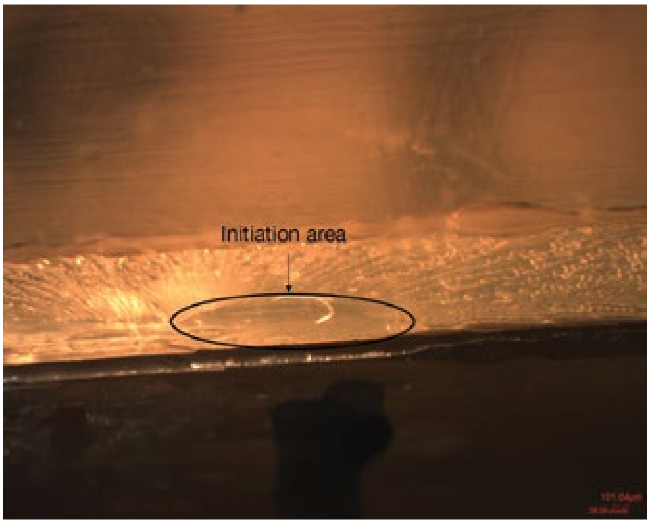
Because sulfone polymers are amorphous materials, they can exhibit three different surface fracture patterns:

- Static mechanical failure
- Dynamic mechanical failure
- Environmental and stress cracking

Static Mechanical Failure

Static mechanical failure consists of the rapid propagation of a crack through the material which is under an external load. The fractured surface pattern is presented in Figure 2.

Figure 2: Surface fracture pattern typical of static mechanical failure in sulfone polymers



In static mechanical failure, the mirror zone is surrounded by hackles, which are divergent lines radiating outward from the fracture origin and pointing along the crack propagation direction. Hackles correspond to a more violent fracture such as a large absorption of strain energy through important plastic deformation (crazing or shear yielding). They result from crack branching when the propagation rate of the initial crack becomes too high. Hackles are generally located in areas where the stress field is changing rapidly in either direction or magnitude.

Static mechanical failure is typical of a rapid or instantaneous rupture, including crack formation and its rapid propagation.

The initiation point of the failure corresponds to the area from where the propagation lines emerge. In most cases, failures are initiated from high stress areas such as corners, ribs, defects and foreign particles. Crack formation is not related to a repetitive process but rather results from a constant excessive loading such as tension, bending, shear or torsion. Such excessive loading can have different causes such as:

- Excessive stress applied on the part during use or assembly
- Wrong design
- Presence of defects or contaminants in the polymer acting as stress concentrators

The initiation zone can vary in size depending on its nature. In case of local defects or polymer contamination by foreign particles, the initiation point is generally very small (1 µm to 100 µm). In contrast, the initiation area can spread over a few millimeters when the static mechanical failure occurs subsequently to another failure process (Figure 6a and 6b).

Depending on the stress level applied to the parts, the rate of crack propagation can be more or less elevated. In the case of very high propagation rates, the fracture surface pattern is often more complex and a crocodile-skin pattern can be observed (Figures 3 and 4).

This pattern results from profuse crack branching during crack propagation. At sufficiently high crack velocity, the single crack front can split up into very small cracks that continue to propagate with slightly different crack angles.

Figure 3: Complex crocodile-skin patterns can be observed at very high propagation rates

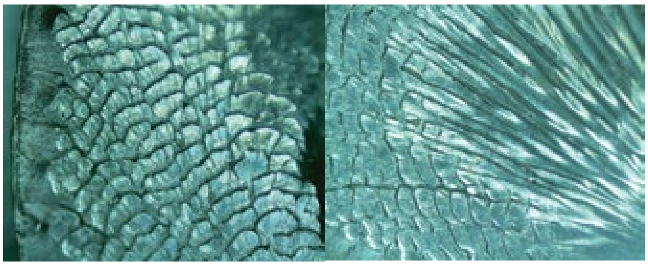


Figure 4: Crocodile-skin pattern subsequent to a mechanical failure as the propagation rate increased

Dynamic Mechanical Failure

Dynamic mechanical failure is also called fatigue failure. In this case, the part break results from successive loading applied to the part. This repetitive process can have several origins such as thermal cycles, pressure variation and repetitive movement.

The pattern representative of dynamic mechanical failure is shown in Figures 5a and 5b. The deformation zone consists of successive striations (semi-elliptical lines) that emanate outward from the initiation zone. Each striation represents a true crack arrest marking and corresponds to crack-front position at each successive stress cycle.

The space between the striations, which is generally uniform, depends on the stress applied locally as well as on external conditions such as material temperature.

The initiation point corresponds to the area from where the striations emanate (Figures 5a and 5b). As for static mechanical failure, this zone is typically located in high stress areas such as corners, ribs, defects and foreign particles.

Dynamic mechanical failure is representative of a progressive part break. In the first step, a crack is initiated in the polymer matrix and grows slowly through the material under the effect of cyclic loading. As long as the material surrounding the crack can sustain the applied external stress, the part will not break. As a result, striations can be observed over a wide area around the initiation point depending on the type of applied stress loading as well as on the part design.

As soon as the stress can no longer be supported by the remaining material, a rapid failure occurs following a process similar to the static mechanical failure. In those cases, both types of patterns can be observed on the same fracture surface. This coexistence is quite frequent in case of failures resulting from dynamic mechanical loading (Figures 6a and 6b).

Figure 5a: Typical fracture pattern of dynamic mechanical or fatigue failure in sulfone polymers

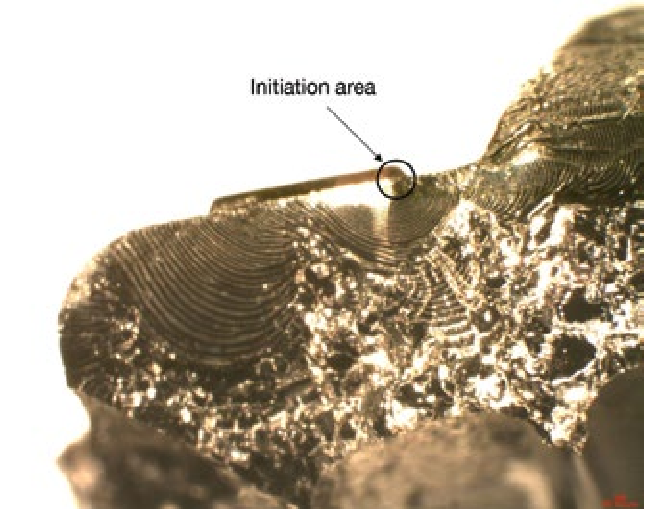


Figure 5b: Typical fracture pattern of dynamic mechanical or fatigue failure in sulfone polymers

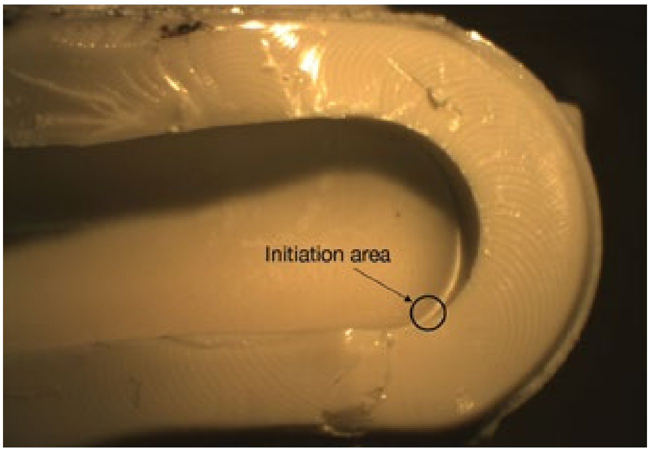


Figure 6a: Coexistence of two different failure processes, which switches from original dynamic mechanical failure into a rapid propagation (static mechanical failure)

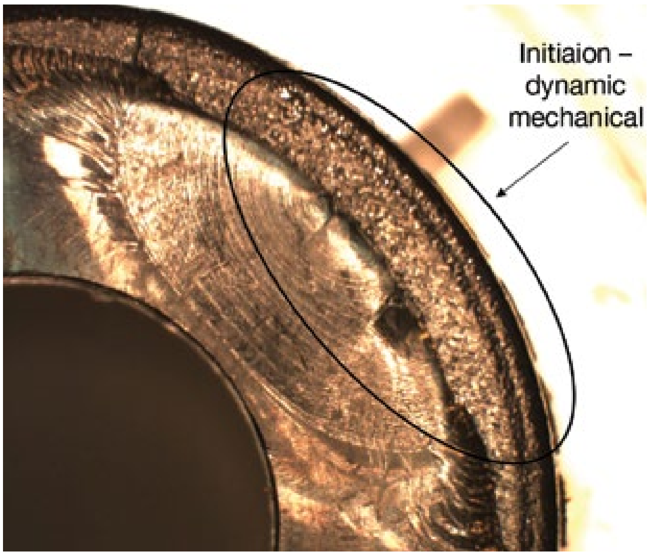


Figure 6b: Coexistence of two different failure processes, which switches from original dynamic mechanical failure into a rapid propagation (static mechanical failure)

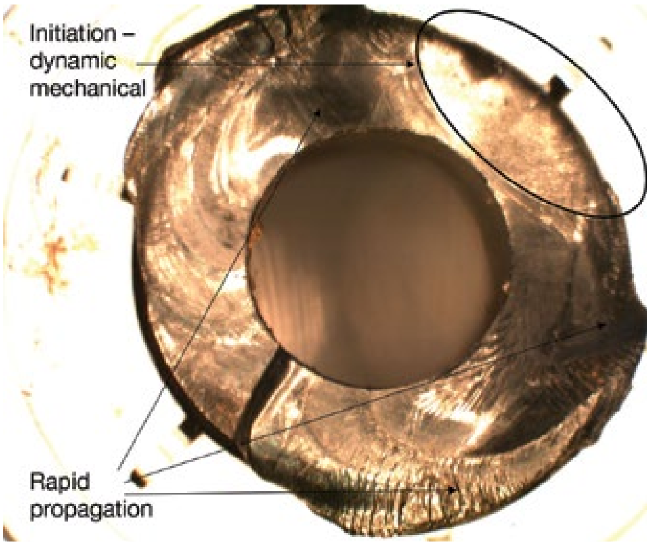


Figure 7: Pattern representative of moderate chemical attack in sulfone polymers

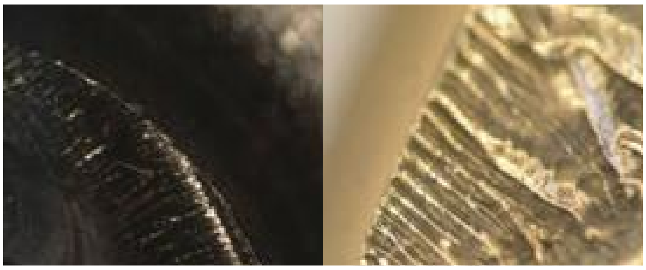
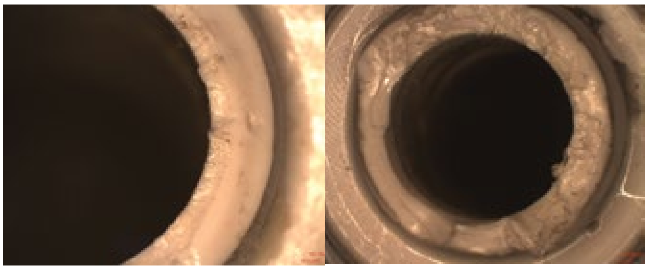


Figure 8: Pattern representative of severe chemical attack in sulfone polymers



Environmental Stress Cracking

The fracture pattern representative of environmental stress cracking can be very complex and can exhibit different textures depending on the severity of the chemical attack. In case of a moderate attack, parallel crazes perpendicular to the part surface are observed in the mirror zone (Figure 7). In contrast, a severe attack can result in a smooth and shiny extended mirror zone. A very rough mirror zone can also be observed in the latter case (Figure 8).

Crazes and deformation zone locations are directly related to the contact position between the sulfone polymer and the chemical. In contrast with the other type of failure, the initiation point is not typically located in high stress areas, but more often located in the area that is in contact with the chemical; therefore, crazes can grow from the inner surface, the outer surface or from both.

In the case of chemical attack, an additional analysis of the surface by infra red or X-ray spectroscopy is often performed in order to collect information about the structure and to identify the composition of the corrosive species. Please contact your Solvay representative for assistance in identifying chemicals that are not compatible with sulfone polymers.

A preliminary general analysis of the fractured surface is very important in these cases. Indeed, as shown in Figures 9 and 10, a fracture pattern representative of a chemical attack with crazes parallel to the part surface can be easily confused with a fracture pattern representative of mechanical failure only if a localized analysis at high magnification is performed. Effective fractography must therefore always include a global analysis of the fracture surface.

Figure 9: Fractured surface in Radel® PPSU part at high magnification. Pattern seems to be representative of chemical attack.

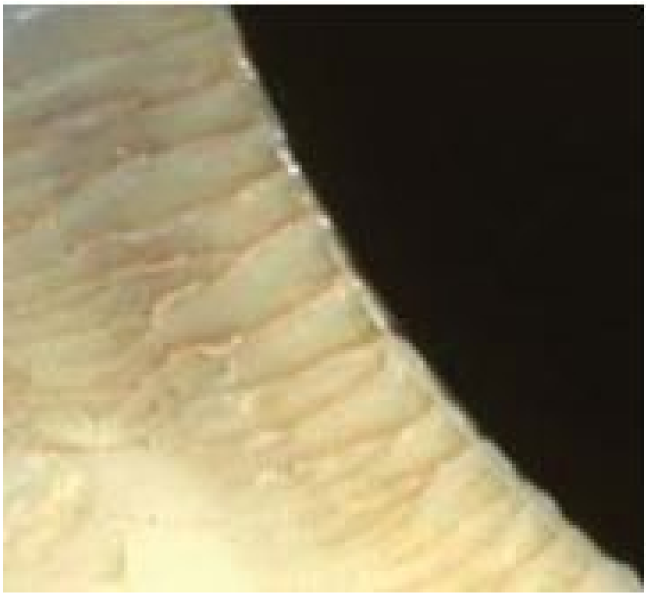


Figure 10: Fracture surface in the same part at low magnification. Pattern is clearly related to mechanical failure.

Conclusions

Fractography is a powerful technique used to determine the cause of failure in plastic parts made of sulfone polymers. The different surface fracture patterns related to these polymers have been presented and discussed allowing a rapid analysis of broken parts. The three main identified failure processes, associated to their fracture patterns, are static mechanical failure, dynamic mechanical failure, and environmental and stress cracking.

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