

FRACTOGRAPHY OF METALS AND PLASTICS

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Abstract

Fractography is critical to failure analysis of metals and plastics. Fractography of plastics is a relatively new field with many similarities to metals. Utilizing case histories, various aspects of failure analysis and fractography are compared and contrasted.

Common failure modes include ductile overload, brittle fracture, impact and fatigue. Analogies can also be drawn between stress corrosion cracking (SCC)/stress cracking, corrosion/chemical aging, dealloying/scission, residual stress/frozen-in stress, and welds/knit lines. Stress raisers, microstructure, material defects, and thermo-mechanical history play important roles in both cases. Key fractographic features for metals and plastics are described.

Historical Perspective

Plastics have been in existence for approximately 130 years. John Hyatt patented nitrocellulose, the first commercial plastic, in 1869. However, full-scale development and use of plastics is only about 50 years old. In contrast, metals have been in use for many hundreds of years.

The application of engineering materials is unavoidably accompanied by the occurrence of failures, many of which have been catastrophic. The consequences of material failures; including deaths, financial losses and legal ramifications; have encouraged the development of effective failure analysis methods. Although the cost of failure analysis may exceed the value of the part, the cost of service failures usually far exceeds the cost of failure analysis. Many of the techniques utilized over the years for the evaluation of metals have been successfully applied to plastics with only minor modifications.

Fractography is arguably the most valuable tool available to the failure analyst. Fractography, a term coined in 1944 to describe the science of examining fracture surfaces, has actually been utilized for centuries as part of the field of metallurgy. Even before that, however, Stone Age man possessed a working knowledge of fracture. Archeological findings of lithic implements, weapons and tools shaped from stone by controlled fracture, indicate that prehistoric man knew how to: (1) select rocks with favorable fracture

behavior; (2) use thermal spalling to detach bedrock from the working core; and (3) shape stone by pressure flaking.

Fractography as we know it today, developed in the 16th century as a quality control practice employed for ferrous and nonferrous metal working. "De La Pirotechnia" published by Vannoccio Biringuccio (1) in 1540 is one of the first documents to detail fractographic techniques.

Invention of the optical microscope in 1600 provided a significant new tool for fractography. Yet it was not utilized extensively by metallurgists until the 18th century. In 1722, R.A. de Réaumur (2) published a book with engravings depicting macroscopic and microscopic fracture surfaces of iron and steel. Interestingly, the categories of macroscopic features developed by de Réaumur have remained essentially unchanged through the centuries.

Partly due to the development of metallographic techniques for examining cross sections of metals, interest in microfractography waned during the 19th century. Metal workers continued to utilize fractographic techniques for quality assurance purposes but, for the most part, researchers and publications ignored fractography.

Several technological developments in the 20th century revitalized interest in fractography. Carl A. Zapffe (3) developed and extensively utilized fractographic techniques to study the hydrogen embrittlement of steels. His work led to the discovery of techniques for photographing fracture surfaces at high magnifications. The first fractographs were published by Zapffe in 1943.

An even more revolutionary development was the invention of the scanning electron microscope (SEM). The first SEM appeared in 1943. Unlike the transmission electron microscope, developed a few years earlier, it could be used for fracture surface examination. An SEM with a guaranteed resolution of approximately 500 angstroms became commercially available in 1965. Compared to the optical microscope, the SEM expands resolution by more than one order of magnitude and increases the depth of focus by more than two orders of magnitude. The tools for modern fractography were essentially in place before plastics achieved widespread use.

Failure Analysis Overview

The general procedure for conducting a sound failure analysis is similar for metallic and nonmetallic materials. The steps include: (1) information gathering; (2) preliminary, visual examination; (3) nondestructive testing; (4) characterization of material properties through mechanical, chemical and thermal testing; (5) selection, preservation and cleaning of fracture surfaces; (6) macroscopic examination of fracture surfaces, secondary cracking and surface condition; (7) microscopic examination; (8) selection, preparation and examination of cross sections; (9) identification of failure mechanisms; (10) stress/fracture mechanics analysis; (11) testing to simulate failure; and (12) data review, formulation of conclusions and reporting.

Although the basic steps of failure analysis are nearly identical, some differences exist between metals and plastics. Nondestructive testing of metals includes magnetic particle, eddy current and radiographic inspection methods that are not applicable to plastics for obvious reasons. However, ultrasonic and acoustic emission techniques find applications for both materials.

Likewise, different chemical test methods are necessary. Typical test methods for metals are optical emission spectrometry (OES), inductively coupled plasma (ICP) and combustion. Fourier transform infrared (FTIR) spectroscopy is extensively used to identify plastics by molecular bonding and thermal testing, differential scanning calorimetry (DSC) and thermo-gravimetric analysis (TGA), is also very important for polymer characterization. Energy dispersive X-ray spectroscopy (EDS), used in conjunction with the SEM, is a very practical tool for elemental chemical analysis of metals and plastics. Also noteworthy, different chemical solutions are required for metals and plastics to clean fracture surfaces and to etch cross sections to reveal microstructure.

Causes of Failure

Of course, the primary objective of a materials failure analysis is to determine the root cause of failure. Whether dealing with metallic or nonmetallic materials, the root cause can normally be assigned to one of four categories: design, manufacturing, service or material. Often times, several adverse conditions contribute to the part failure. Many of the potential root causes of failure are common to metallic and nonmetallic materials.

Improper material selection, overly high stresses, and stress concentrations are examples of design-related problems that can lead to premature failure. Material selection must take into account environmental sensitivities as well as requisite mechanical properties. Stress raisers are frequently a preferred site for fracture origin,

particularly in fatigue. These include thread roots (Figure 1), sharp radii of curvature, through holes, and surface discontinuities (e.g., gate marks in molded plastic parts).

Likewise, many manufacturing and material problems found in metals are also observed or have a corollary in plastics. Weldments are a trouble prone area for metals, as are weld lines or knit lines in molded plastics (Figure 2). High residual stresses can result from metal forming, heat treatment, welding and machining. Similarly, high frozen-in stresses in injection molded plastic parts often contribute to failure. Porosity and voids are common to metal castings and plastic molded parts (Figure 3). These serve as stress raisers and reduce load carrying capability. Other manufacturing- and material-related problems that may lead to failure include adverse thermo-mechanical history, poor microstructure, material defects and contamination.

Environmental degradation is one of the most important service-related causes of failure for metals and plastics. Others include excessive wear, impact, overloading, and electrical discharge.

Failure Mechanisms

Another key objective of failure analysis is to identify the failure mechanism(s). Once again, some failure modes are identical for metals and plastics. These include ductile overload, brittle fracture, impact, fatigue, wear and erosion.

Analogies can also be drawn between metals and plastics with regards to environmental degradation. Whereas metals corrode by an electrochemical process, plastics are vulnerable to chemical changes from aging or weathering. Stress corrosion cracking, a specific form of metallic corrosion, is similar in many ways to stress cracking of plastics. Both result in brittle fracture due to the combined effects of tensile stress and a material specific aggressive environment. Likewise, dealloying or selective leaching in metals (Figure 4), the preferential removal of one element from an alloy by corrosion, is somewhat similar to scission of polymers (Figure 5), a form of aging which can cause chemical changes by selectively cutting molecular bonds. Analogies can also be drawn between metals and another type of polymer, rubber. The precipitation of internal hydrogen in steels can lead to hydrogen damage, which is characterized by localized brittle areas of high reflectivity known as flakes or fisheyes on otherwise ductile fracture surfaces (Figure 6). Similarly, explosive decompression in rubber O-rings produces fisheye-like oval patterns on the fracture surfaces (Figure 7). Explosive decompression is the formation of small ruptures or embolisms when an elastomeric seal, saturated with high pressure gas, experiences an abrupt pressure reduction. This failure mechanism is analogous to the "bends" that afflicts divers that surface too quickly.

Fractography

When material failure involves actual breakage, fractography can be employed to identify the fracture origin, direction of crack propagation, failure mechanism, material defects, environmental interaction, and the nature of stresses.

Some of the macroscopic and microscopic features employed by the failure analyst to evaluate fracture surfaces of metals and plastics are described below. Note, however, that many of the fractographic features described for plastics are not observable for reinforced plastics and plastics containing high filler content.

Macroscopically Visible Fractographic Features

On a macroscopic scale, all fractures (metals and plastics) fall into one of two categories: ductile and brittle. Ductile fractures are characterized by material tearing and exhibit gross plastic deformation. Brittle fractures display little or no macroscopically visible plastic deformation and require less energy to form. Ductile fractures occur as the result of applied stresses exceeding the material yield or flow stress. Brittle fractures generally occur well below the material yield stress. In practice, ductile fractures occur due to overloading or under-designing. They are rarely the subject of a failure analysis. Fracture analysis usually involves the unexpected brittle failure of normally ductile materials.

Many macroscopically visible fractographic features serve to identify the fracture origin(s) and direction of crack propagation. Fractographic features common to metals and plastics are radial marks and chevron patterns. Radial marks (Figure 8) are lines on a fracture surface that radiate outward from the origin and are formed by the intersection of brittle fractures propagating at different levels. Chevron patterns or herringbone patterns are actually radial marks resembling nested letters “V” and pointing towards the origin.

Fatigue failures in metals display beach marks and ratchet marks that serve to identify the origin and the failure mode. Beach marks (Figure 8) are macroscopically visible semi-elliptical lines running perpendicular to the overall direction of fatigue crack propagation and marking successive positions of the advancing crack front. Ratchet marks are macroscopically visible lines running parallel to the overall direction of crack propagation and formed by the intersection of fatigue cracks propagating from multiple origins.

Brittle fractures in plastics exhibit characteristic features, several of which are macroscopically visible (Figure 9). These may include a mirror zone at the origin, mist region, and rib marks. The mirror zone is a flat, featureless region

surrounding the origin and associated with the slow crack growth phase of fracture. The mist region is located immediately adjacent to the mirror zone and displays a misty appearance. This is a transition zone from slow to fast crack growth. Rib marks are semi-elliptical lines resembling beach marks in metallic fatigue fractures.

Microscopically Visible Fractographic Features

On a microscopic scale, ductile fracture in metals (Figure 10) displays a dimpled surface appearance created by microvoid coalescence. Ductile fracture in plastics (Figure 11) is characterized by material stretching related to the fibrillar nature of the polymers response to stress. Although a part may fail in a brittle manner, ductile fracture morphology is frequently observed away from the origin, if the final fast fracture occurred by ductile overload (e.g., the “shear lip” in metal failures). The extent of this overload region is an indication of the stress level.

Brittle fracture of metallic materials may result from numerous failure mechanisms, but there are only a few basic micro-fractographic features that clearly indicate the failure mechanism: (1) cleavage facets (Figure 12); (2) intergranular facets (Figure 13); and (3) striations (Figure 14). Cleavage facets form in body-centered cubic (BCC) and hexagonal close-packed (HCP) metals when the crack path follows a well defined transgranular crystallographic plane (e.g., the {100} planes in BCC metals). Cleavage is characteristic of transgranular brittle fracture. Intergranular fracture, recognizable by its “rock candy” appearance, occurs when the crack path follows grain boundaries. Intergranular fracture is typical of many forms of SCC, hydrogen embrittlement and temper-embrittled steel. Fatigue failures of many metals exhibit striations at high magnifications (normally magnifications of 500 to 2,500X are required). Striations are semi-elliptical lines on a fatigue fracture surface that emanate outward from the origin and mark the crack front position with each successive stress cycle. The spacing of fatigue striations is usually very uniform and can be used to calculate the crack growth rate if the cyclic stress frequency is known. Striations are discriminated from striation like artifacts on the fracture surface in that true fatigue striations never cross or intersect one another.

Plastics do not display cleavage and intergranular fracture. However, like metals, striations are found on fatigue fracture surfaces (Figures 15 and 16). Striations in plastics are typically observable at much lower magnifications (50-200X). However, local softening and melting due to hysteretic heating can obliterate fatigue striations in less rigid plastics.

In addition to mirror zones, mist regions and rib marks, which are normally visible without the aid of a microscope, brittle fracture of plastics may display hackles, Wallner

lines and conic marks. Hackles (Figure 9) are divergent lines radiating outward from the fracture origin. They resemble river patterns observed on the cleavage facets of transgranular brittle fractures of metals. Wallner lines are faint striation-like markings formed by the interaction of stress waves reflected from physical boundaries with the advancing crack front. Conic marks are parabolic-shaped lines pointing back towards the origin. Hackles and Wallner lines may or may not be visible without the aid of a microscope.

- (3) Brunet, Paris, 1722; see translation by A.G. Sisco, "Réaumur's Memoirs on Steel and Iron", University of Chicago Press, 1956.
Zapffe, C.A. and Moore, G.A., *Trans AIME*, 154, 335-359 (1943).

Closing Remarks

Fractographic techniques developed and applied to metal failures for centuries have been readily adapted to the fracture analysis of plastics since their emergence as a key engineering material over the last 50 years. However, more work remains to be done to advance fractography of plastics. One notable area for research is fracture analysis of composites, reinforced plastics, and plastics containing high filler content. Fractures of these materials are too often dismissed as inherently lacking meaningful fractographic features. Finally, there is a definite need for an authoritative publication on fracture in plastics.

Acknowledgements

The author gratefully acknowledges the contributions of Dave Christie and Steve Ruoff of IMR Test Labs.

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- (2) Réaumur de, R.A., "L'Art de Convertir le Fer Forgé en Acier, et L'Art d'Adocir le Fer Fondu", ("The Art of Converting Wrought Iron to Steel and the Art of Softening Cast Iron"), Michel

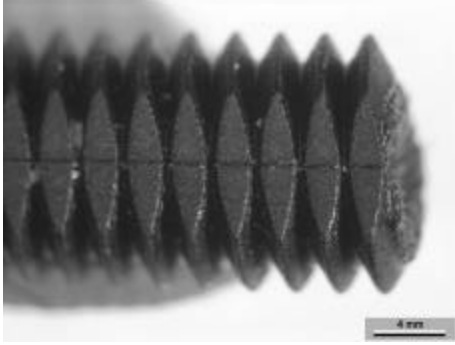


Figure 1 - Fracture of a glass-filled polyamide threaded part due to stress concentration.

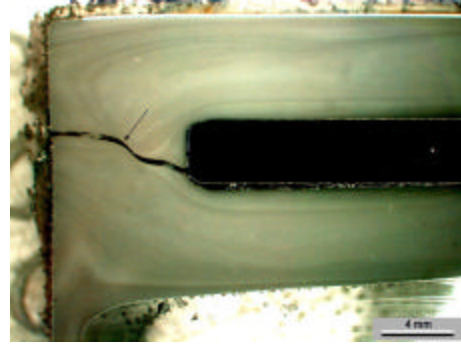


Figure 2 - Cross section showing fracture along the knit line of a perfluoralkoxyethylene lined impeller.

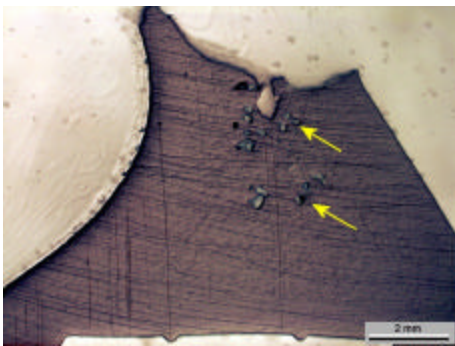


Figure 3 - Cross section of a polyacetal hinge that fractured (arrows) through an area of porosity.

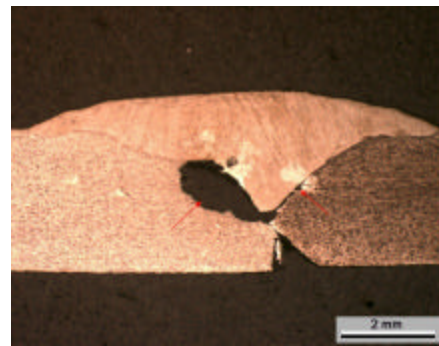


Figure 4 - Microbiologically induced corrosion of a 304 SST vessel weld characterized by pitting and selective leaching (arrows).

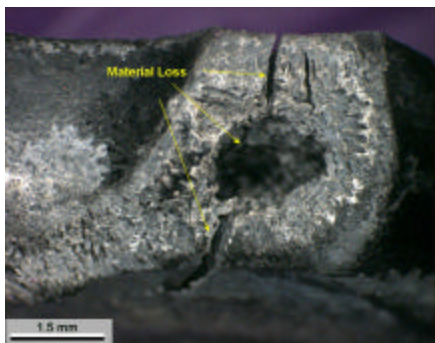


Figure 5 - Hollowing out of a polyacetal hinge due to acid catalyzed hydrolysis.

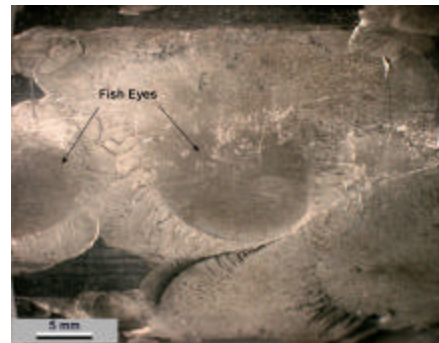


Figure 6 - Hydrogen damage of an induction hardened steel piston rod displays "fisheyes".

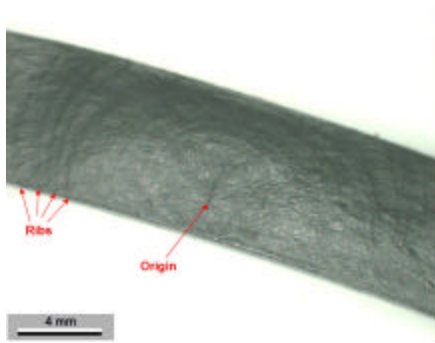


Figure 7 - Explosive decompression fractures of rubber O-rings are characterized by fisheye-like patterns.

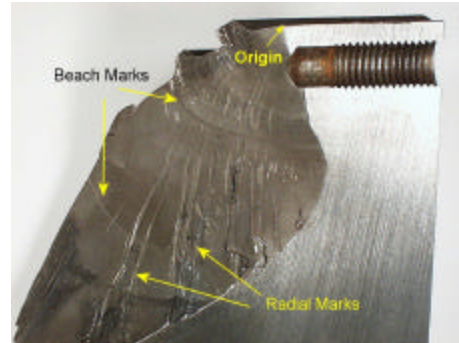


Figure 8 - Beach and radial marks are visible on this torsional fatigue fracture of a 6" dia. 4340 shaft.

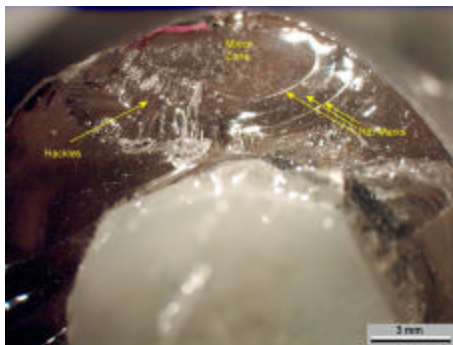


Figure 9 - Brittle fracture of an epoxy layer displays a mirror zone, rib marks and hackles.

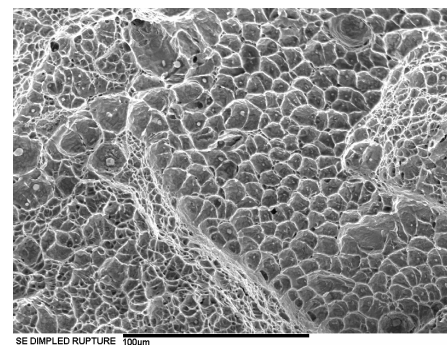


Figure 10 - Dimpled appearance typical of ductile fracture of metallic materials.

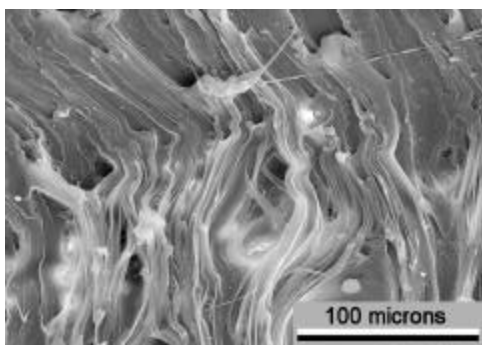


Figure 11 - Fracture of a polyethylene tensile test specimen exhibits material stretching.

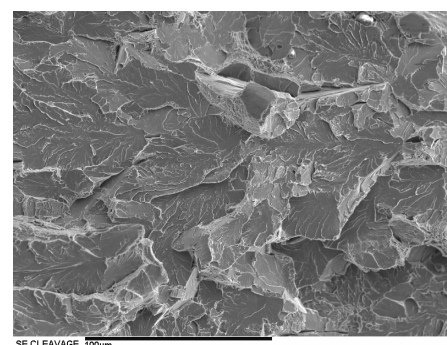


Figure 12 - Brittle fracture of a FC-0205 powder metal control rod displays cleavage facets.

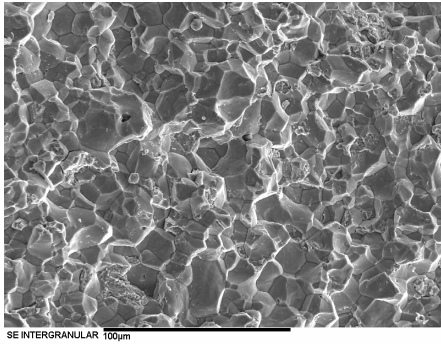


Figure 13 - Intergranular fracture of an embrittled cast steel pneumatic wrench.

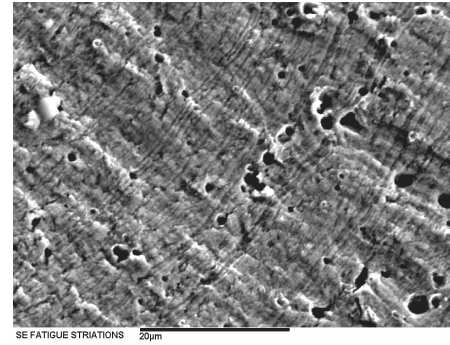


Figure 14 - Fatigue striations are visible on this Type 302 stainless steel spring fracture.

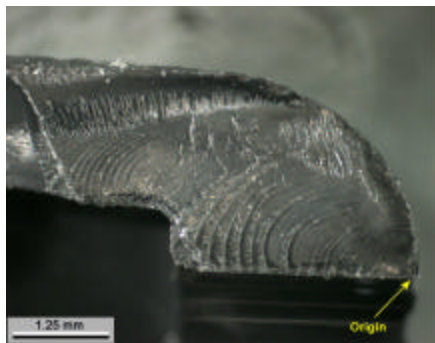


Figure 15 - Fatigue striations emanate from the fracture origin of this polycarbonate latch handle.

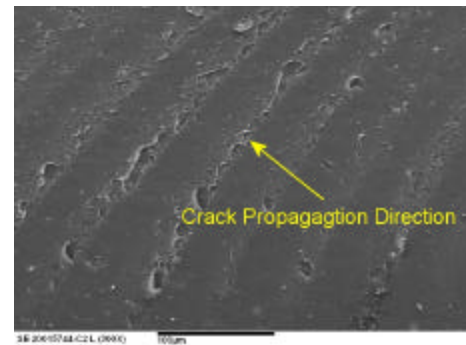


Figure 16 - SEM photomicrograph of the fatigue striations shown in the previous figure.