Fracture surfaces exposed to various environments generally contain surface debris, corrosion, or oxidation products that must be removed before meaningful analysis is possible. Before any cleaning procedures begin, the fracture surface should be surveyed with an optical stereo microscope, and the surface should be documented with appropriate optical photographs. This low-power microscope should be used to ascertain the severity of the surface contamination and to monitor the effectiveness of each subsequent cleaning step.

Sometimes the debris and deposits on the fracture surface can contain information that is vital to understanding the cause of failure, so some investigation and consideration of these surface characteristics are useful prior to fracture surface cleaning. Often, knowing the nature of the surface debris and deposits, even when unessential to the fracture analysis, will be useful in determining the optimum cleaning technique.

This overview article discusses fractographic features, fracture surface cleaning methods, and analysis of fracture surfaces. It also briefly overviews an investigation of a series of test exposures on a fractured Charpy impact specimen of H13 die steel. Further details of this investigation are available from the authors and have been accepted for publication to the ASM Journal of Failure Analysis and Prevention.

**Fractographic features**

Failure analysts usually investigate the fracture path to determine the fracture initiation and termination sites, as well as other fractographic features. Based on this information, the analyst can also identify various types of monotonic (single cycle) overload, fatigue (multiple cycle) cracking, and time-dependent (creep or corrosion) failure, or combinations thereof.

Fractographic features help determine how a part fractured. Table 1 shows various types of service conditions and fracture processes that can be identified from fracture surface examinations. Table 2 shows examples of what fracture surface markings indicate about a component’s service history and even its material properties.

Characterizing fracture surfaces and deter-
mining how a component fractured is sufficient to solve some problems for component designers. For example, analyzing fractures that develop during prototype testing is useful to identify designs with inadequate section size, unpredicted response to service loads, and severe stress concentration.

However, the purpose of failure analysis is usually to determine why a part fractured or degraded during service. This knowledge can help prevent personal injury and property damage, improve part quality by reducing material and manufacturing defects, and help resolve legal disputes between product manufacturers, suppliers and/or customers. To determine failure causes in these scenarios, other laboratory test results should be combined with the fracture surface observations.

Fracture surface cleaning

Since fracture surface cleaning invariably affects the fracture surface chemistry, localized elemental analysis (if required) is recommended prior to cleaning. Therefore, energy dispersive spectroscopy (EDS) analysis on a scanning electron microscope (SEM) should be conducted prior to fracture surface cleaning. Although SEM fractographic examination prior to cleaning is rarely helpful and is usually more effective after cleaning, the opposite is true of EDS analysis.

To make important fracture surface features discernible, the fracture surface may require cleaning prior to fractographic inspection. Cleaning can involve removal of lint, loose debris, oil and rust, or intentionally applied preservative coatings. Recommended procedures for cleaning, in order of increasing severity, include air blast, replica stripping, organic solvents, water-based detergents, cathodic cleaning, and chemical etching. All cleaning methods are intended to remove extraneous material from the fracture surface and leave the features of the fracture surface unaltered.

The method selected for cleaning a particular fracture depends upon the nature of the material to be removed and the nature of inspection required. For example, oil might be removed from a fracture surface with organic solvents or water-based detergents. Stork CRS employs RBS-35 (Chemical Products R. Borghgraef), but another typical solution is boiling Alconox (Alconox, Inc.) or 5 to 15% ethylenediamine tetraacetic acid (EDTA), an antioxidant, in deionized water. Lint and lightly adhered materials might be removed with an air blast or soft brush.

The method of cleaning also depends on the nature of the required inspection. If only macroscopic features must be observed, milder methods of cleaning are sufficient. However, microscopic inspection of the fracture features requires a method that thoroughly cleans the fracture surface.

Rust is a common contaminant of steel fracture surfaces. Superficial rust is sometimes removed with ultrasonic cleaning in detergent. More tightly adhered rust requires treatment with inhibited acid. “Inhibited” means that the acid contains a chemical (inhibitor) that retards the dissolution of metal without hindering the removal of metal oxides.

Stork CRS uses concentrated hydrochloric acid (HCl) and hexamethylene tetramine as recommended by the ASM Handbook. However, other investigators recommend 10 g/liter 1,3-Di-n-buty1-2 thiourea, with a 50:50 dilution or 3 grams of “2-Butyne-1,4Diol”, 5 ml HCl, per 100 ml water.

Surface effects

Interpreting the fracture surface after the removal of rust is complicated by the aggressive nature of the acid. The process of rusting alters the fracture surface by creating pits and destroying fine features of the fracture surface. The acid removes the rust, but after a period of time will itself create pits and destroy fine features of the fracture surface. Valid fracture surface interpretation requires preserving the fracture features as much as possible. Therefore, acid cleaning requires careful control so that fracture surface damage is prevented or minimized.

Some rust is very tightly adhered and requires a long exposure to acid for complete removal. Other lightly rusted pieces come clean with just a brief dip in the acid.

Because acid cleaning of rusted fracture surfaces is so common, we designed a study to ascertain the amount of damage that results from various durations of acid exposure. The purpose of the investigation was to answer the following questions:

- If rust is removed in a few seconds, is the fracture surface preserved during the treatment, or has it already been damaged?
- Will additional treatment remove additional rust, or will the extended exposure increasingly damage the surface?
The photomicrographs on the first page show a service fracture with various attributes visible as a result of fracture surface cleaning. The overload zone, ratchet marks, beach marks, corroded pearlite, and nonmetallic particles can be clearly seen.

**Experimental investigation**

Anecdotal stories and industry lore abound, sometimes indicating that the surface is not degraded at all. The goal of this investigation was to acquire objective data with systematic testing to evaluate the effects of inhibited acid cleaning of fracture surfaces for fractography.

To determine the effect of inhibited hydrochloric acid upon fracture surfaces, a Charpy impact specimen of H13 die steel was fractured at room temperature and a particular region of the fracture surface was photographed in a scanning electron microscope at various magnifications. The ground faces of the Charpy specimen provided accurate registration of the sample in the SEM so that the same fracture region could be repeatedly located after multiple acid exposure and examination cycles.

The fracture surface was then covered with a drop of water and allowed to rust 24 hours. Several different methods of inducing rust were tried, but this one proved the best at producing a continuous rust coating. The same region of the Charpy specimen was photographed using the SEM.

Subsequently, the fracture surface was immersed in inhibited hydrochloric acid consisting of 6N (normal) reagent grade, concentrated hydrochloric acid in water with 2g/L of hexamethylene tetramine added. This is the composition recommended in the ASM Handbook. The fracture surface was immersed for a period of 15 seconds; it was then removed and ultrasonically rinsed in methanol.

The same specimen was repeatedly exposed to inhibited hydrochloric acid and then photographed with the SEM. The cumulative time in the acid increased from 15 seconds to 30, 60, 120, 240, 480, 600, and 1200 seconds.

Results showed that when the fracture surface was cleaned in inhibited hydrochloric acid and viewed at lower magnifications, the large fracture surface features were still visible after long exposure times. However, when the authors viewed the cleaned surface at high magnifications, minute fracture surface features deteriorated rapidly with increased exposure to the cleaning acid. Therefore, the acid exposure time should be limited as much as possible for high magnification examinations, whereas exposure time is less critical for low magnification study of fracture surfaces. For high magnification work, exposures less than 15 seconds are recommended.

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