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Unraveling the Mystery: Using Microscopy in a Metallurgical Failure Investigation

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Conducting a root cause metallurgical failure analysis is similar to a forensic investigation by a detective. Any failure will leave clues as to the sequencing of events, and these must be pieced together to determine the root cause of the failure. Often this requires gathering and understanding the background information, which includes the material history, service process or operational history and design criteria. Obtaining and understanding such information is critical because a failure analysis inherently follows an order of increasing destructiveness of tests and sample removal. Even a non-destructive test such as with dye penetrant can contaminate a fracture surface. The overall objective of the investigation is to identify the root cause of the failure in order to prevent future occurrences.

Thus the most critical aspect of the failure analysis methodology is the planning to ensure that the investigation achieves the goal of determining the root cause. It is extremely easy to section a sample for optical or scanning electron microscopy incorrectly with the result of obtaining useless information. Unfortunately, when the failure is small or there is a single crack, any initial mistakes can greatly compromise the investigation. As an example of the appropriate approach for conducting a proper, thorough but efficient failure analysis, a failure from a pilot plant will be used as often times these failures tend to be very small and require significant planning before initiating the physical parts of the investigation.

The failure of interest occurred in a fitting located directly downstream of the pilot plant reactor that failed after 10 days of operation. Material of construction was confirmed to be type 316 stainless steel. Hydrocarbon feed during the run included low levels of H_2S. Cl level in the feed was ~1 wt ppm. The reactor temperature was varied between 315 and 400°C with a plant pressure of 640 psig.

A possible crack was noted on the outside diameter (OD) of the fitting and the tube section was slit to reveal the extent of the cracking on the inside diameter (ID) (Figure 1). The sliced tube section was cut again length wise so that half could be used for metallographic analysis at the presumed initiation site and the other half could be used for SEM fractography.

A branched, transgranular crack initiating from the ID was observed that propagated almost completely through the wall of the tube, as shown in the metallographic cross-section of Figure 2. Branching was not extensive, suggestive of high stress Cl stress corrosion cracking. Microstructure was typical of cold working and no sensitization could be found.

Most of the fracture surface consisted of transgranular shear (Figure 3) with only a small portion exhibiting a dimpled appearance typical of ductile overload. SEM/EDS chemical analysis at the crack initiation site identified the presence of both S and Cl; however, only S and no Cl was found a short distance (200 μm) from the ID. The ID of the tube also showed the presence of Cl and S but no Cl or S was detected at the crack tip. Cl was detected on other parts of the fitting with less noted for the outer block while the outer tube section had no detectable Cl or evidence of cracking.

The ultimate cause of the failure was the result of Cl stress corrosion cracking. Because of the design and assembly of the fitting, the line was under high torsional stress. Although the Cl was at extremely low concentration, the Cl concentrated at the failed location because of vapor/liquid condensation along with the sulfide corrosion scale providing a concentration site. Change to a fitting connection producing less stress and suitable insulation to prevent vapor/liquid concentration was recommended.

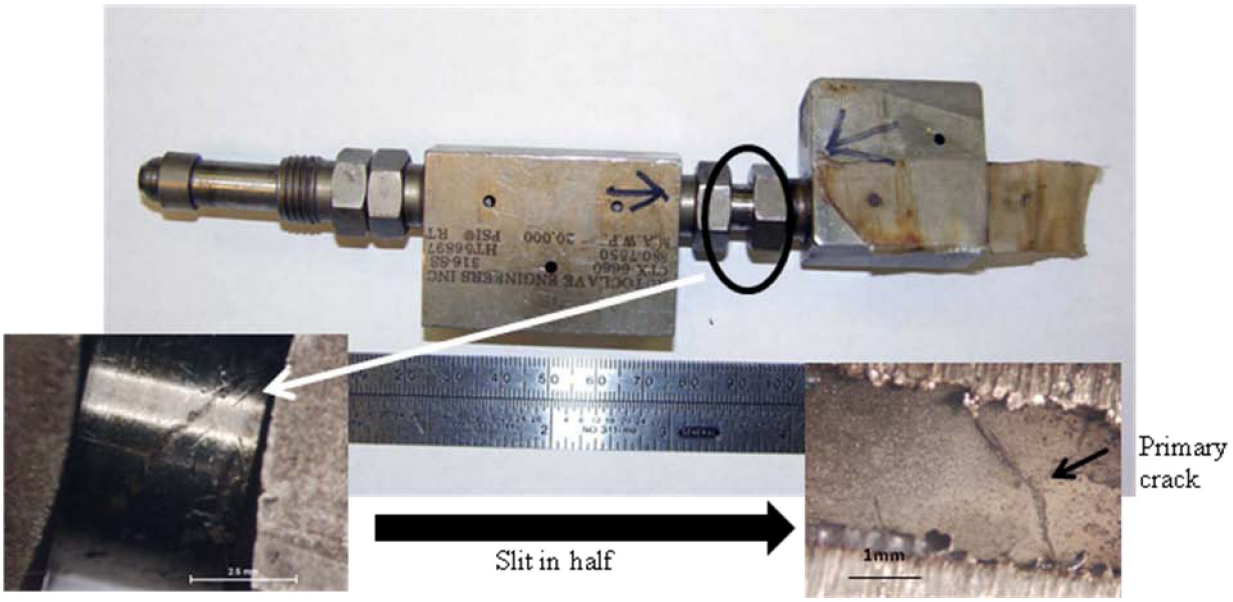

Figure 1. Observed crack on OD and sectioned for viewing on ID; inlet is on right.

Figure 2. Branched transgranular cracking initiating from ID.

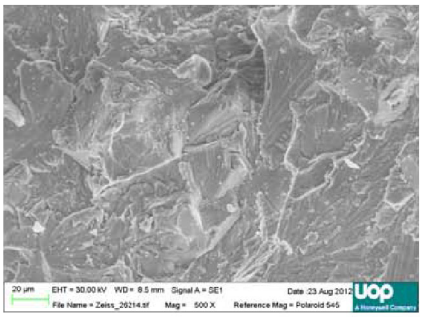

Figure 3. Transgranular fracture surface.

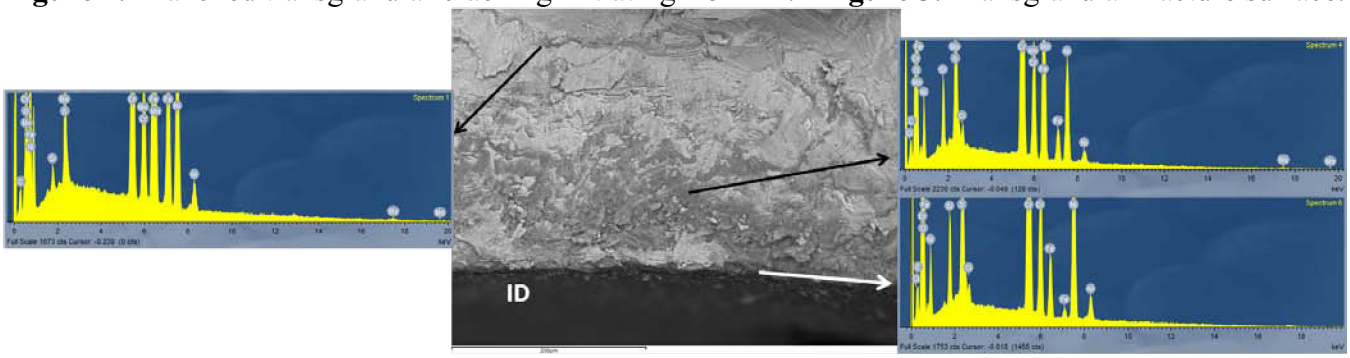

Figure 4. At ID of fracture surface Cl is detected and has concentrated in the sulfide scale.

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