C20 The Application of Scanning Electron Microscopy and Energy Dispersive Spectroscopy for Forensic and Failure Investigations

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After attending this presentation, attendees will have a better understanding of the use and value of using SEM with EDS in investigating component and industrial and chemical process failures.

This presentation will impact the forensic science community by providing examples of approaches to use in failure investigations in a timely manner.

Scanning Electron Microscopy (SEM) with associated Energy Dispersive Spectroscopy (EDS) has been used extensively for industrial forensic applications at Materials and Chemistry Laboratory, Inc. (MCLinc), particularly to evaluate causes of failure in manufacturing and chemical processes. Failure investigations are typically initiated with a client interview to establish alloy type of the failed component, operating conditions, process excursions, and other historical data. The region of failure is then visual inspected. Proper collection and conditioning of samples for shipping has been found to be essential for preserving sample integrity prior to analysis. After visual inspection and photography, coupon sections are mapped out and separated to allow for comparison of sections near the region of failure and those isolated from the region of failure. The coupons are then embedded in epoxy in a 1.25 inch diameter mold, cured, and polished to 0.3µm. The mounts are then examined by Reflective Optical Microscopy and SEM. The polished cross sections are typically etched during the examinations to expose the grain structure of the alloys. Optical Microscopy is conducted to determine the general grain structure and/or inclusions while Electron Microscopy is used to examine fine details at higher magnification and obtain general chemistry and chemistry of features and/or precipitates within the grain boundaries. The grain structure observed in the failure region provides insight into the failure mechanism. Observed failures include thermal granular growth, intergranular attack, and general corrosive attack.

A recent investigation included the examination of a 3/8 inch Inconel 600 vessel which failed in hydrogen fluoride service. The results of the investigation demonstrated that sulfidation was the cause of the failure. It was determined that a process variable caused the sulfur concentration in the feed stream to be higher than typical. Moreover, a mechanical issue involving a defective heater element caused elevated temperature at the failure region, hence, accelerating the rate of sulfidation. The low oxygen content of the process also contributed to the accelerated sulfidation. Photomicrographs and EDS spectra revealed a breakdown of the Cr_2O_3 layer with the resulting transport of sulfur within the bulk alloy. Ni-S, Cr-S, and Cr depleted regions were also observed. This observation was consistent with published literature on sulfidation. Results of this investigation provided a mechanism for failure that is consistent with the operating conditions.

Failure Investigations, SEM-EDS, Chemical Processes

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