Some Failure Analysis Case Histories in Galvanized Steel Products

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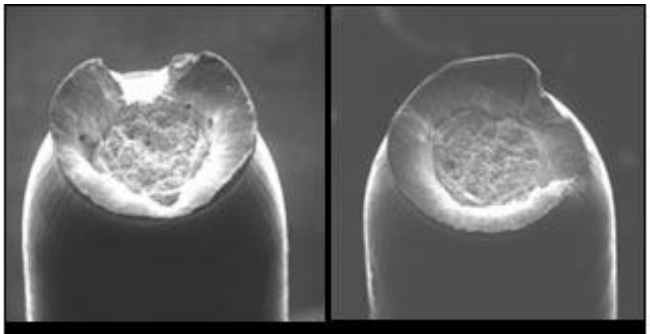
Materials analysis involves the determination of the physical and chemical properties of all sorts of materials, solids, liquids and gases, of any composition at all. Out of the effectively infinite number of properties that could be measured or determined, the analyst has the interesting job of providing for the client just those that matter for their job. As Dragnet's Sergeant Friday used to say: "Just the facts, M'am," and to that we add, "Leave out anything that doesn't matter for our case!"

While it is a serious error not to start any analysis at the macro or visible level, it's just in the nature of things that very soon we almost always begin to want to know more about the micro-level. There are two kinds of "modern" microscopies: those that use light to form the image and those that use electrons. Light microscopes have a history longer than 300 years, but they continue to improve steadily. Lens design has virtually eliminated serious aberrations, which for most of the instrument's history had kept their performance less than satisfactory. There are now quite literally dozens of important and useful ways to obtain light microscope images, depending on what properties you are interested in investigating.

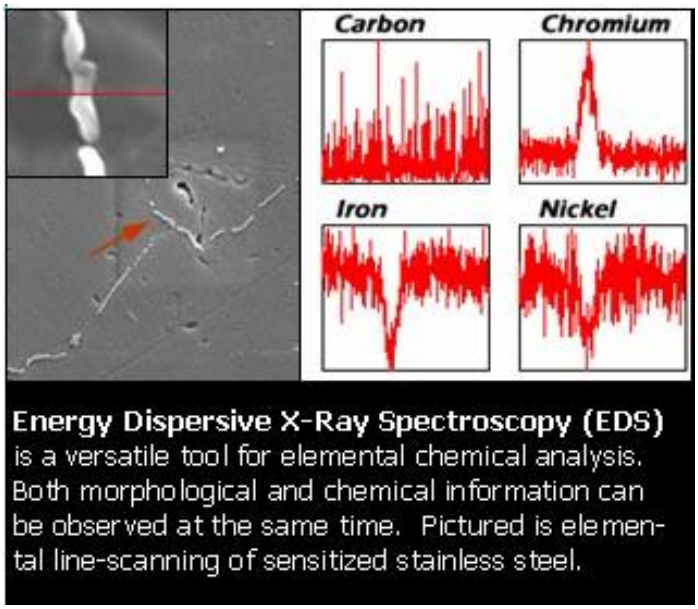
The primary introductory tool is stereo light microscopy, generally used at magnifications between 10X and 45X, taking you from the realm of the familiar into the unknown. Things look very different at 45X than they do with the eye.

At higher magnifications reflected light, often called "metallurgical" microscopy, is the most common technique. For this a sample is polished to create a perfectly planar cross section through it. Frequently important information can be obtained directly from this approach, but often it is valuable also to etch the sample surface chemically, bringing out substructures that help to explain materials properties, for example, solder and weld joints or heat treatment effects. Direct magnifications to 1000X are common using this technique. Since the depth of field of the microscope lenses is very small at these magnifications, samples which are not polished usually don't yield much information. It's mostly out of focus.



Scanning Electron Microscope (SEM) eliminated the depth of field problem in light microscopes. Pictured are classic Cup-and-Cone tensile fractures in steel wire. Note the very huge depth of field, with the sample surface in focus throughout the image.

This depth of field problem was first pushed back in the 1960's when scanning electron microscopy (SEM) came on the scene. This approach, using electrons instead of light to illuminate the sample and form the image, virtually eliminated the depth of field problem, producing images which seemed interpretable almost intuitively. The information content went up exponentially. Suddenly, with very little sample preparation, it became possible to see amazing details of fracture surfaces, crystal growth phenomena, even biological samples such as wood or the favorite, insect eyes! Magnifications up to 20,000X became commonplace.



Energy Dispersive X-Ray Spectroscopy (EDS) is a versatile tool for elemental chemical analysis. Both morphological and chemical information can be observed at the same time. Pictured is elemental line-scanning of sensitized stainless steel.

The key development that made SEM even more useful was the parallel development of energy-dispersive x-ray spectroscopy, a versatile tool for elemental chemical analysis. Now you could obtain both morphological and chemical information at the same time. Although serious quantitative analysis must still be done on polished samples rather than on rough surfaces, qualitative identification of composition can be done now on virtually anything. The subject of sensitivity limits is complex, but point analyses can detect remarkably small amounts of material. The beam actually penetrates several micrometers into most materials, so the activated volume contributing to a spectrum is not truly at the sample surface. But even so, given an understanding of the limitations, a remarkably good job can be done with EDS, even in surface analysis.

One of the most interesting new techniques for SEM work is that many new microscopes can be operated at deliberately poor vacuums, with water vapor or some other gas in the chamber eliminating the need for coating the sample to provide conductivity. While the imaging results may not quite equal those of high-vacuum scopes, the simplification of sample preparation is often worth the slightly lower quality. One problem in the past was that wet samples would collapse when dried in the microscope vacuum. That needn't happen now.

If higher magnifications are needed, or if atomic-level details are important, several techniques of transmission electron microscopy (TEM) are also available. In fact, fracture surface studies were first done by replica techniques, providing even greater levels of detail than are available to most SEMs today. Unfortunately sample preparation and sample size create some barriers to application of this powerful old-fashioned technique.

Most samples for TEM must be a maximum of 3mm in diameter and thin enough to transmit electrons (usually in the neighborhood of 1000 Angstroms thick). Fortunately simply grinding most powders in an agate mortar and pestle will produce edges that are sufficiently thin for useful TEM work. A uniquely powerful TEM tool is selected area diffraction, producing structural information to go along with EDS chemical information, completely characterizing crystalline materials. And powerful techniques are now available for producing routine ion-beam-thinned sections, from which grain boundaries, dislocations and many other ultra-structural properties can be characterized by TEM.

There are many other light and electron based techniques in the modern microscopist's arsenal. These include confocal laser-scanning light microscopy, in which successive optical slices up through the sample are stacked by computer to give transmission images of much greater effective depth of field than possible by any other technique. This technique has been used more by biological than materials microscopists. And the true surface techniques, such as x-ray photoelectron spectroscopy and Auger spectroscopy add the possibility of seeing the chemistry of the absolute contact region between two materials.

There is one other form of microscopy that uses neither light nor electrons, the so-called scanning probe techniques for which the Nobel Prize in Physics was awarded to Gerd Binnig and Heinrich Rohrer in 1986. Scanning tunneling microscopy was the first of these, but it has been followed by a veritable flood of other imaging modes. All of these have in common the fact that they are capable of detecting atom-to-atom relationships. These include electrochemical phenomena of many kinds. The drawback of these for most industrial users is that industrial samples are usually not "ideal" and thus are extremely complicated if viewed at the atomic level. The noise level usually simply swamps the signal. Industrial applications of scanned-tip microscopy will require extremely careful planning of experiments to isolate the phenomenon of interest.

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