Medical device materials are analyzed at all stages of design and service, from initial fabrication and development of a prototype, through examination of the device or surrounding tissues following removal from the patient. The resulting understanding of materials properties and related performance can prevent device failure, ensure patient safety, and drive the next innovations in materials and device design.

To acquire this understanding, microstructural characteristics such as crystalline phase, presence of secondary phases, and uniformity of elemental distribution must be assessed and combined with results from performance testing to determine whether the alloy is suitable for the intended application.

Transmission electron microscopy (TEM) is an ideal technique for analyzing metals and other materials to gain an understanding of their structural and elemental properties on the sub-micrometer to atomic scale. The method can be applied to materials at various stages of processing, to finished products, and even to wear debris, such as sub-micrometer particles removed from device surfaces or patient tissues.

Analysis of Medical Devices

The JEOL JEM-3010 transmission electron microscope (Fig. 1) at McCrone Associates is configured for materials analysis. It is capable of 1.2 million times magnification, and can resolve features as small as 0.14 nanometer. Along with this high-resolution imaging capability, electron diffraction analysis allows for crystalline phase identification. Additionally, an energy dispersive X-ray spectrometry (EDS) system attached to the microscope provides elemental identification of features or areas as small as three nanometers.

This combination of capabilities makes the TEM an extremely powerful tool for materials characterization, providing information that may not be obtainable by other methods. Techniques such as X-ray diffraction and scanning electron microscopy sample larger amounts of material and provide results that complement those from TEM. A multi-technique approach to materials analysis is recommended, but TEM is often the best method by which to gain the nanometer-scale understanding necessary for development of today’s high performance materials.

McCrone Associates’ applications of TEM analysis to medical devices have included imaging of a porous metal oxide anode device, identification of metal and polymeric wear particles isolated from devices and tissues, and characterization of a prototype austenitic stainless steel alloy for a vascular stent.

Sample Preparation

In one case, Boston Scientific approached McCrone Associates for a pre-clinical analysis of a novel metal alloy, a modified 316L stainless steel for a vascular stent. It had been modified by addition of platinum to increase radiographic density. Two TEM
analyses evaluated the potential impact of the modification on the elemental and structural properties of the alloy. Key to both studies was the preparation of suitably thin sections from the samples provided. Since the TEM utilizes a beam of high-energy electrons passing through the material, samples must be flat sections 100 nanometers or less in thickness.

A variety of techniques can produce thin sections for TEM analysis, and the method must be determined on a case-by-case basis. Bulk samples of self-supporting materials such as metals are reduced in size to produce a disk three millimeters in diameter, and the center of the disk is thinned until it perforates; the edges of the hole thus formed will be thin enough for TEM analysis. Milling with an argon ion beam or electropolishing with a strong acid solution are two typical methods of preparing thin metal specimens. An optical photomicrograph of a three-millimeter diameter metal thin section prepared by ion milling is shown in Fig. 2.

Samples included a section of the prototype metal embedded in epoxy and polished for metallographic analysis, and sections of 316L and prototype tubing with diameters smaller than one tenth of an inch. Thin section preparation was done by the Electron Microscopy Service of the Research Resources Center at the University of Illinois at Chicago. In the case of the embedded section, small pieces of stainless steel were removed with a diamond saw and thinned by argon ion milling. The small-diameter tubes were cut open lengthwise and flattened; disks were then cut from the flattened sections. Tubing made of standard 316L stainless steel was thinned by electropolishing, but when this technique was tried on the prototype stainless steel modified with platinum, the metal discolored, and ion milling was used instead. This illustrates the care that must be taken with preparation of TEM samples, as any step in the process may introduce artifacts into the material. Understanding of the material, the properties to be analyzed, and the possible effects of various preparation methods is critical to producing a good specimen and representative results.

**Analysis of Specimens**

In the study done on the polished section, the purpose was to identify the crystalline phase and elemental composition of precipitates observed in an optical microscopic examination of the section. Presence of such precipitates is not uncommon in austenitic stainless steel alloys, where a chromium oxide coating forms on the metal and inhibits corrosion. Chromium in solution in the steel may migrate to the grain boundaries, which are interface regions between grains comprising a polycrystalline material such as a metal. The chromium can form precipitates of chromium carbide in the case of a high carbon steel, or chromium oxide in a low carbon steel such as 316L. Because this material had been modified by addition of platinum, it was necessary to determine the composition of the observed precipitates.

TEM imaging provided information about precipitate size, and EDS analysis showed them to contain only chromium and oxygen. Figure 3 shows an image of a typical precipitate about two micrometers in size. Measurements of atomic plane spacings from the accompanying diffraction pattern were compared to spacings for known materials. These spacings were from the crystallographic database maintained by the International Center for Diffraction Data; the precipitates were identified as probable $\text{Cr}_2\text{O}_3$.

In a second study, samples prepared from 316L tubing and platinum-enhanced 316L were compared to determine whether the platinum was uniformly distributed, or whether its addition led to formation of secondary phases that might compromise the physical properties of the metal alloy.

TEM imaging showed no evidence of formation of inclusions or precipitates in the metal, and also indicated that the crystalline grains comprising the platinum-enhanced...
The alloy were larger than those in the standard 316L stainless steel. Extensive EDS analysis was done, both by mapping of large areas and by spot analysis of several individual grains and grain boundaries. Segregation of elements into these thin boundary regions between grains can greatly impact the strength properties of an alloy, and it was critical to determine whether such segregation of the platinum had occurred. Figure 4 is a TEM image of a triple grain boundary in the platinum-modified alloy, and Figure 5 is an EDS spectrum from the grain boundary region. TEM EDS analysis showed the 316L and the platinum-modified alloy to have very uniform elemental distributions. There was no indication that secondary phases had formed, or that platinum had segregated into the grain boundaries of the modified alloy.

Using transmission electron microscopy, information about feature size and morphology, crystallinity, and elemental distribution can be gained on scales ranging from submicrometer to near-atomic. Sample preparation may be challenging, but few other techniques can provide similar insights into materials properties on the near-atomic level. The result is increased understanding of structural properties and performance, and enhanced ability to develop new, innovative materials and devices.

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