Target selection

Article 3/3

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Know how

Target Material selection for Sputter Coating of SEM Samples

This article describes target material options for sputter coaters that deposit a thin metal coating on non-conductive SEM samples. Coating a sample with a conductive metal renders an insulating sample conductive enough to minimize charging effects on the SEM image. In most cases, coating SEM samples with only a few nanometres of a metal results in crisp, clear images. Proper target material selection is dictated by overall imaging requirements, the SEM available, the specimen material being evaluated, and whether X-ray microanalysis will be required.

Introduction

Since its commercial introduction in 1965, the scanning electron microscope (SEM) has evolved to incorporate many improvements in imaging and microanalysis capabilities, yet the problem of charging in non-conductive samples remains. The SEM user is still required to cope with the examination of non-conductive samples on a case-by-case basis. Fortunately, there are a number of strategies to aid in this process.

Charge mitigation

The problem is as follows. Negative charge builds up on a non-conductive specimen at normal electron accelerating voltages (kV), particularly above 10 kV, because more electrons land on the specimen that leave as secondary electrons (SEs) or backscattered electrons (BSEs). This can produce in the SEM image strong bright areas and scan raster shifts. These image artifacts can be so severe that the resulting image has no relationship to the object being scanned. While charging can be minimized by imaging at low beam energies near 1keV, only recent SEM models, particularly those employing field emission electron guns (FE-SEMs), can maintain small electron beam probe sizes on the specimen at such a low accelerating voltage (kV). Alternatively, a variable-pressure SEM, operating in low-vacuum mode (specimen chamber pressure about 1torr=133 Pa), produces positive ions that can neutralize surface charging. A third method of suppressing charge build up is to deposit on the non-conducting specimen surface an extremely thin conductive coating, typically a metal that adds minimal structure to the true specimen surface. The latter method is easy, dependable, and can be used with any SEM. Some coatings exhibit a grain structure that can be observed in modern SEMs, especially those equipped with field-emission (FE) electron guns. There are a range of metals for sputter coating, some for use at low magnifications and others for use at high magnifications in an FE-SEM. An additional benefit of metal coating is that the yield of secondary electrons (SEs) is usually much higher than for the bare non-conducting surface.

Coating selection

The coating metal should be selected to achieve optimum performance based on the type of analysis to be performed: for example, low-magnification, high-magnification imaging, or microanalysis. The safematic CCU-010 / SP-010 sputter coater permit quick target changes, allowing the microscopist to select an appropriate coating metal for the task at hand. The sputtered coating should have a high secondary electron emission yield so that the signal-to-noise ratio will be high. The ideal coating should have no structure (grains or islands) that would interfere with the details of specimen features. Thus, coatings with large grains would be suitable only for low magnifications, where the structure of the coating would be too small to see. Some metals that produce fine-grained coatings suitable for high-magnification imaging, deposit at slower rates; but, this is not a problem because useful coating thicknesses are quite small, typically 1–3 nm. Some coating materials have X-ray lines that may interfere with the detection of elements in the specimen. However, at typical accelerating voltages, this should not be a problem when the coating is only 1–2 nm in thickness. If there is a serious interference, another coating metal could be selected to coat that specimen. Finally, there is a cost factor since the most useful coating materials are precious metals.

Materials and Methods

While not exhaustive, the list of materials below describes the most common metals used to sputter coat samples for the SEM. Keep in mind that this information is only valid when using a modern DC magnetron SEM sputter coater with pure argon as the process gas. Some coatings require “high-resolution” sputter coaters that operate at better vacuum to reduce the possibility of oxidation during processing; in fact, some systems employ a shutter to shield the sample while oxide is sputtered off the target itself in a pre-conditioning step. Carbon is commonly used as a conductive coating for microanalysis samples, but this material should be deposited by vacuum evaporation or ion-beam sputtering.

Instrumentation

The sputtered films for this article were produced using a CCU-010 HV (turbo-pumped) safematic Coating System on glass slides. Pure argon was used as a backfill “process gas.” The system above could be described as a “high resolution” sputter coater because a turbo pump is employed to obtain a higher (and cleaner) vacuum environment, and pure argon gas is backfilled in the chamber to remove air and increase sputter efficiency. Film thickness measurements were obtained using the quartz thickness monitor (operating at 6 MHz) inherent with the system. Coatings in Figure 1 were imaged with a Zeiss Merlin FE-SEM.

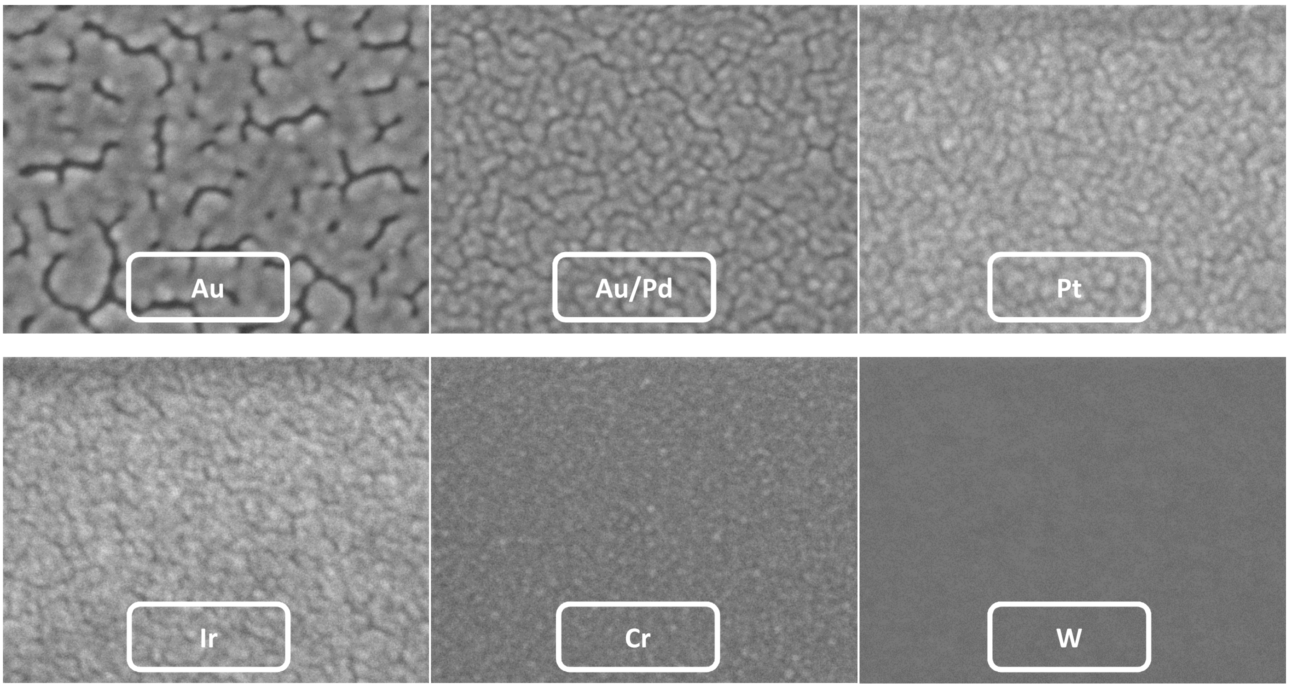

Figure 1: Secondary electron SEM images of various sputter target materials. All coatings were 2 nm thick deposited on glass and imaged at 10 keV.

Target Metal Selection

Tantalum
Ta is also a candidate for high resolution coating (most refractory and high melting materials exhibit fine grain size). It oxidises quite rapidly, similar to Cr. Low sputtering rates, but due to high atomic number the SE yield tends to be higher. Samples must be imaged immediately after coating or stored under high vacuum.

Nickel
Ni is an alternative coating material for EDX applications and BSE imaging. Not ideal for SE imaging, the coating oxidises slowly. It has a (very) low sputtering rate due to the low work functions and the fact that as a magnetic materials it "short circuits" the magnet in the DC magnetron sputter head with a less dense plasma as a result. In a standard SEM coater, the coating contains a mixture of Ni and Ni-oxide. The Ni coating layer can enhance elements through X-ray fluorescence. If needed, the Ni coating can be removed if needed with a Hydrochloric acid or Nitric acid.

Copper
Cu is an alternative low cost material for EDX applications and BSE imaging. Suitable for low and medium magnification ranges. Lower SE yield coatings will slowly oxidize. In a standard SEM coater the coating consists of a mixture of Cu and Cu-oxide. However, it is a low cost alternative for educational applications to demonstrate and to investigate the influence of coating parameters. The Cu coating layer can be used to enhance the analysis of transition materials through X-ray fluorescence. If needed, the copper coating can be removed with Ferric chloride or Nitric Acid.

Titanium
Ti is seldom used as coating material, but has applications where it is chosen to avoid any interference with EDX analysis. Low atomic number gives less interference with BSE imaging. Ti oxidises quite rapidly and samples need to be imaged directly after coating.

Carbon
Carbon is the material of choice for coating non-conductive samples to allow for EDX analysis and BE imaging. It is has a low atomic number, is conductive and is inert at room temperature. It can be sputtered with the CCU-010 / SP-011 instrument but it tends to deposit as DLC material which is non-conductive. Carbon can be used in carbon evaporators (CCU-010 / CT-010) to coat SEM samples or to produce carbon support films for TEM.

Next Article

Summary Target selection

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