

Target Material selection for Sputter Coating of SEM Samples



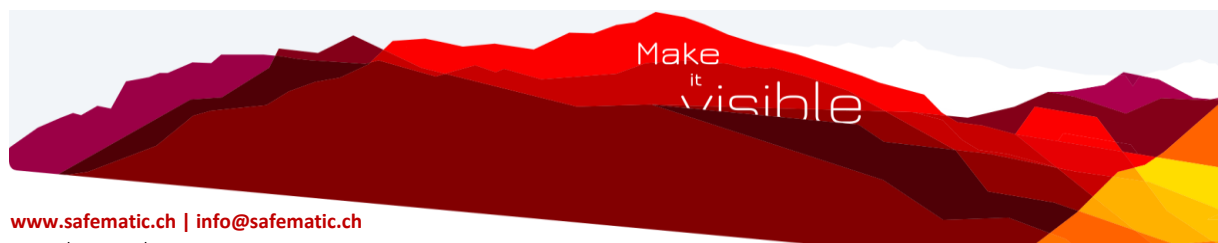
This article describes target material options for sputter coaters that deposit a thin metal coating on non-conductive SEM samples. Coating a sample with a conductive metal renders an insulating sample conductive enough to minimize charging effects on the SEM image. In most cases, coating SEM samples 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

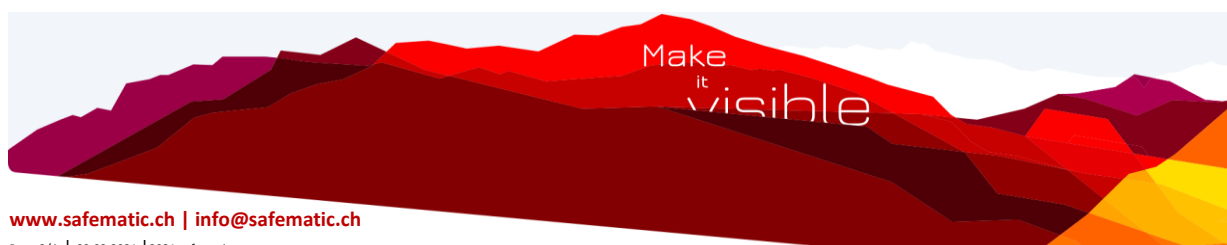
The problem is as follows. Negative charge builds up on a non-conductive specimen at normal electron accelerating voltages (kV), particularly above 10 kV, because more electrons land on the specimen that leave as secondary electrons (SEs) or backscattered electrons (BSEs). This can produce in the SEM 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.

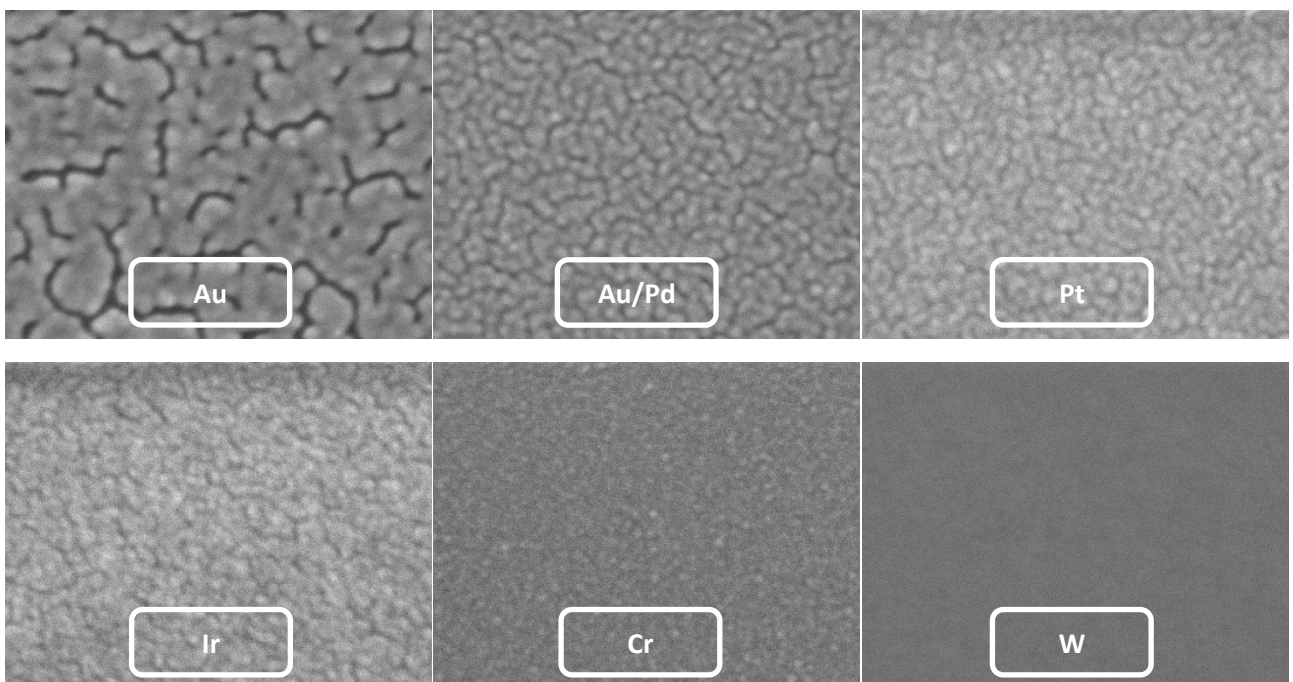
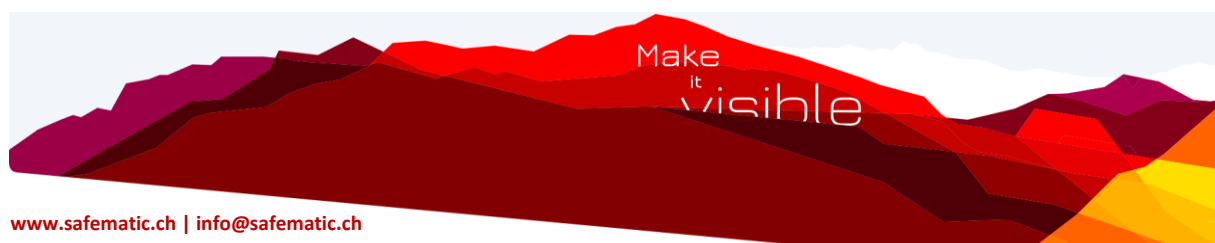


Figure 1: Secondary electron SEM images of various sputter target materials. All coatings were 2 nm thick deposited on glass and imaged at 10 keV.



Target Metal Selection

Gold is perhaps the most widely used coating material for non-conductive SEM samples, but it is not recommended as a sputter coating for research purposes where high-magnification images are required (see Au/Pd below). Gold has a high secondary electron yield and sputters relatively rapidly, but the coating structure is composed of large islands (grains) that can be observed at high magnifications in most modern research-level SEMs (Figure 1). Thus, it should only be used for imaging at low magnifications, say less than 5000×, where the coating structure will not interfere with the structural details of the sample. An advantage shared by most other precious-metal coatings, Au coatings do not oxidize in laboratory air. X-ray emission lines of the Au M-series (2.12 keV) may interfere with X-rays from S and Nb, while the Au L-alpha line (9.71 keV) may interfere with X-rays from Ge. If the Au coating is appropriately thin, however, there should not be significant problems with qualitative X-ray microanalysis.

Gold/palladium sputtered alloys (60/40 and 80/20) have smaller grain size and are the recommended metal coatings for general research purposes. Secondary electron yields are high, and sputter rates for Au/Pd are only slightly lower than for pure Au. The Pd L-series X-ray lines at 2.84 keV do not overlap important lines from other elements; thus, no additional interference with X-ray microanalysis would be expected beyond that mentioned above for Au.

Platinum has a finer grain size than either Au or Au/Pd, which makes it more suitable for higher-magnification applications. A sputtered Pt coating exhibits a high SE yield, but Pt has a lower sputtering rate than Au (Table 1). Pt has been observed to crack. This effect could be “stress cracking” and could be attributed to oxygen deposition in the sputtered coating, indicating the need for a sputter coater with better vacuum. The characteristic X-rays of Pt (particularly the M-series at 2.05 keV) have the potential to overlap with lines from P and Zr, but interference should be minimal for 1–2 nm thick coatings.

Platinum/Palladium alloy (80/20) has a similar small grain size and high SE yield as pure Pt, but it is less sensitive to “stress cracking.” The Pt/Pd alloy is a suitable all-round coating material for high-magnification applications.

Next Article

Palladium, Iridium, Chromium, Tungsten

