

Practical Information on Selecting a Target Material to Coat SEM Samples

Selection of the optimum coating material depends on the (non-conductive) sample, the application and cost. Most SEM sputter coaters allow for a relatively easy target change. When using different coating (target) materials than Gold, the use of Argon as process gas is paramount. One should be aware that each sample material behaves differently when coated. *The coating quality is obviously affected by the target material* and by the interaction of the sample material and metal used for the coating. Coating the SEM sample with a (highly conductive) metal makes non-conductive SEM samples conductive. An additional benefit is that most coating materials have a higher secondary electron (SE) yield than the non-conductive sample material. Coating material with lower atomic numbers is more suitable for backscattered electron (imaging) whereas coating with higher atomic numbers is more suitable for SE imaging. Below you will find some practical guidance information for selecting the target material. The information is only valid when using modern DC magnetron SEM sputter coaters and Argon as process gas.

If EDX analysis of the sample is needed, then always choose a coating (target) material which is not present in the sample, creates overlapping peaks in the EDX spectrum or otherwise interferes with the EDX analysis.

Gold

Gold is an excellent and most widely used coating material to coat non-conductive samples for standard SEM applications. Due to the low work function it is a very efficient to coat and when using cool sputter coaters and thin layers, there is hardly any heating of the sample surface. The grain size is visible when using high magnifications on modern SEMs. Gold can be used for table top SEMs with air as process gas.

Gold/Palladium

The Au/Pd alloy (60/40) is less efficient to coat than with pure gold, which results in lower sputter rates. Au/Pd is often recommended to achieve a smaller grain size. Au/Pd gives smaller grain sizes when evaporated in high vacuum, but when used in SEM sputter coaters the difference between Au and Au/Pd is hardly visible. Less suitable for heat sensitive specimens and less suitable for EDX analysis due to the extra set of peaks for Pd.

Silver

Ag is a most suitable and low cost alternative for Au in many imaging applications for low and medium magnifications ranges. It is a widely underestimated coating material. Ag has the highest conductivity of all metals. When using EDS, Ag is an alternative for Au on many biological samples if P, Cl and S need to be analyzed. Sputtering rate is similar to Au; the SE yield is somewhat lower than for Pt, Au or Ir. The grain sizes are similar or slightly larger than Au, except for samples containing

halogens which can cause coarser grains. The Ag coating can tarnish (in the presence of halogens) and are less suitable for long term storage. Excellent low cost coating material for many less demanding imaging applications and table top SEMs.

An additional advantage is that the Ag coating can be dissolved and the sample surface can be studied in the original conditions. Removal of Au, Au/Pd, Pt and Ir coatings is more difficult and involves very harsh chemicals. The Ag coating can be removed with Farmer's reducer (Mixture of Potassium Ferricyanide and Sodium Thiosulfate)

Platinum

Pt has a finer grain size than Au or Au/Pd and is therefore more suitable for higher magnifications. Excellent SE yield. The higher work function results in slower sputtering than Au. Pt tends to be sensitive for "stress cracking" when oxygen is present (oxygen can come from porous samples). More expensive than Au due to higher fabrication costs.

Platinum/Palladium

Pt/Pd alloy (80/20) has a similar grain size and SE yield as pure Pt, but is less sensitive to "stress cracking". Suitable all-round coating material for FESEM applications when thin coatings are used. Best results are achieved when using high resolution sputter coaters

Iridium

Ir exhibits a very fine grain size on virtually all materials and is an excellent all-round fine grain coating material for FESEM applications. It is the material of choice for high and ultra-high resolution FESEM imaging. With the added benefit of being a non-oxidizing material and a high SE yield, it is replacing Chromium for high resolution sample coating. It requires the use of a high resolution sputter coater and has lower sputtering rates. The targets are thicker due to fabricated constraints, but overall costs are lower than for Pt or Pt/Pd. Ir is also an excellent alternative material for coating samples which have to be analyzed for carbon by EDX or WDX. A thin layer is enough to create excellent conductivity and since the material is very rare it hardly interferes with EDX or WDX analysis.

Chromium

Cr has a very fine grain size, especially on semiconductor type materials and has proven to be a useful coating material for FESEM applications. Cr requires the use of a turbo pumped, high vacuum, high resolution sputter coater with a target shutter since the presence of oxygen (as in standard SEM coaters) causes oxidization during coating. The Cr on the sample surface will oxidize in air and samples must be viewed immediately after coating. Samples can be stored in high vacuum or in inert

gas. Cr has lower sputtering rates and the target tends to heat up. Lower SE yield than Pt, Pt/Pd or Ir. Excellent coating material for high resolution BE imaging of low Z and biological samples.

Tungsten

W is an excellent alternative for ultra-high resolution coating. W has a very fine grain size and tends to be less visible than Cr. W oxidizes rapidly, similar to Cr. Low sputtering rates, but due to the high atomic number the SE yield tends to be higher. Samples must be imaged immediately after coating or stored under high vacuum.

Tantalum

Ta is also a candidate for high resolution coating (most refractory and high melting materials exhibit fine grain size). It oxidizes 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.

Palladium

Pd can be used as a lower cost alternative for low to medium magnification ranges. Gives a lower SE signal than Au. When using EDX analysis, Pd can be an alternative.

Nickel

Ni is an alternative coating material for EDX applications and BE imaging. Not ideal for SE imaging, the coating oxidizes slowly. It has (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 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. 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 BE imaging. Suitable for low and medium magnification ranges. Lower SE yield. Coatings will slowly oxidize. In a standard SEM coaters 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. 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 BE imaging. Ti oxidizes 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 has a low atomic number, is conductive and is inert at room temperature. It can't be sputtered in DC magnetron sputter coaters; high energy is needed and if sputtered it tends to deposit as DLC material which is non-conductive. Carbon can be used as a target in ion-beam coaters such as the Gatan models 681, 682 and 685. Otherwise it is used in carbon evaporators to coat SEM samples or to produce carbon support films for TEM.