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Ion Beam Sputtering: Practical Applications to Electron Microscopy

Introduction

Electron microscope specimens, both scanning (SEM) and transmission (TEM), often require a conductive coating applied to the sample prior to analysis. Conductive coatings serve several purposes in electron microscopy applications and are generally designed to minimize charging of the sample, conduct away heat from the sample during imaging, and to increase the secondary electron yield of the sample (1). There are a wide range of instruments capable of producing these types of coatings and the selection of the instrument depends on the resolution of the microscope, the sample material, and the application for which the microscope is being utilized. Some typical examples of instruments designed for producing conductive coatings are DC magnetron sputter coaters, vacuum evaporators, electron beam evaporators, and ion beam sputtering systems.

To deposit conductive coatings, sputtering instruments are often used. Sputtering is a deposition process which physically removes a target material and deposits a firmly bonded coating onto the sample. The sputtering process occurs by bombarding the surface of the target material with gaseous ions (normally Ar^+) under high voltage acceleration. As these ions collide with the target, atoms or occasionally entire molecules of the target material are ejected and propelled against the sample, where they form a very tight bond. The resulting coating is held firmly to the surface by mechanical forces, and in some cases an alloy or chemical bond may result (2). Although both DC magnetron and ion beam systems utilize the sputtering process to produce conductive coatings, the coating quality, sample quality, and resulting image quality are dramatically different. These differences in properties make the selection of the equipment critical in electron microscopy (EM) applications.

Ion Beam Sputtering: Basics

Ion beam sputtering utilizes an ion source to generate a relatively focused ion beam directed at the target to be sputtered. The ion source consists of a cathode and anode with a common central axis. Applying a high voltage field of 2-10 kV to the anode creates an electrostatic field inside the ion source, confining electrons around a saddle point in the center of the source. When argon gas is injected into the ion source, the high electric field causes the gas to ionize, creating a plasma inside the source region. The ions are then accelerated from the anode region to the exit aperture (cathode) creating a "collimated" ion beam. The resulting ion beam impinges upon a target material and, via momentum transfer between the ion and the target, sputters this material onto the sample.

Ion beam sputtered films have many advantages over other sputtering methods used in EM. Since the coating material is passed into the vapor phase by a mechanical rather than a chemical or thermal process, virtually any material can be deposited on the sample. This flexibility makes ion beam sputtered films ideal for a variety of analytical techniques used in EM. Another by product of the physical sputtering process is the lack of thermal radiation presented to the sample, allowing heat sensitive samples to be processed. This is in direct contrast to magnetron methods where samples are exposed to high energy ion bombardment, electron flux, and high temperatures. Ion beam sputtering is also a highly controllable process due to the lower deposition rates when compared with magnetron methods. This reduction in deposition rate allows for ultra-thin films to be deposited uniformly onto the sample, an advantage for high resolution SEM and various TEM applications.

For these reasons ion beam sputtering is the preferred method for depositing thin film coatings onto samples for EM, especially where resolution and reduced artifacts are of primary concern.

Model IBS/e

The Model IBS/e is a tabletop ion beam sputter deposition system designed to utilize the advantages of ion beam sputtering and produces ultra-fine grain films of any material. The IBS/e implements a large vacuum chamber pumped by a high vacuum turbo molecular pump, producing a clean and hydrocarbon free vacuum environment. A unique stage design allows a wide variety of movements during deposition or etching modes. Independent control of tilt angle, tilting speed, and sample rotation speed can all be controlled at any time during the deposition process. Sample sizes 4” (100mm) or less can be mounted into the stage and allows a wide range of applications to be explored. Figure 1 shows an example of the Model IBS/e and the chamber configuration.

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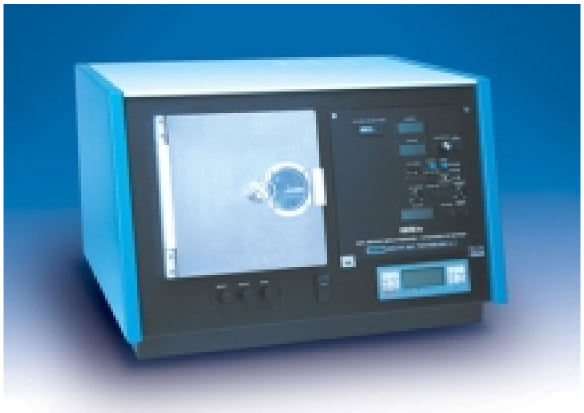

Figure 1: The Model IBS/e Ion Beam Sputter Deposition and Etching System. The inside of the vacuum chamber is shown at right with the Large Area Stage (LAS) installed. Targets are mounted to the chamber lid and can hold up to 4 different target materials.

Many applications are affected by the conductive coating that is applied to the sample, the selection of which should not be taken arbitrarily. Some basic guidelines are used when selecting a target such as the desired magnification and resolution, analytical techniques used (if applicable), and sample compatibility. A basic guideline to some common targets is given in Table 1.

TARGET	MICROSCOPE TYPE	MAGNIFICATION	DESCRIPTION
Au, Au/Pd	STANDARD SEM	< 2,000 X	USED FOR LOW MAGNIFICATION WORK
Ir, Pt	FESEM, FETEM	< 200,000 X	BOTH SIMILAR IN GRAIN STRUCTURE; IRIDIUM PROTECTS SAMPLES FROM BEAM DAMAGE BETTER
Ta, Ti, W, Cr	FESEM, FETEM	> 200,000 X	FILMS SHOW NO STRUCTURE UP TO 500,000 X; SHOW UNIFORM COVERAGE
C	FESEM, FETEM	< 500,000 X	AMORPHOUS AND INDISTINGUISHABLE FROM STANDARD C SUPPORT GRIDS; LOW BACKGROUND FOR EDX

Table 1: Description of some common target materials and their applications to electron microscopy.

Biological Applications

As discussed, ion beam sputtering offers many unique features for an array of EM applications. For biological materials, the use of the Model IBS/e is particularly useful due to the nature of the ion beam sputtering process. Many biological materials can be very sensitive to heat and thermal effects which makes the deposition of a conductive film without introducing artifacts of particular importance. An example of this can be seen in Figure 2. This image shows an example of the ability of the IBS/e to produce excellent conductive coatings to heat sensitive samples. A conductive coating of iridium was applied to the sample using 8 kV and 3 mA of ion gun current. Deposition time was approximately 5 minutes.

Figure 2: SEM image of cheese product as coated using the Model IBS/e. The image shows no damage from the deposition process and shows excellent image quality. (Image courtesy of William McManus, Dept. of Nutrition and Food Science, Utah State University)

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Another example of the usefulness of the IBS/e for deposition of thin films is shown in Figure 3. These images show immunogold staining of samples coated with chromium and imaged with a backscattered electron detector at 10 kV. Chromium provides sufficient backscatter to enable contrast between the coating and colloidal gold particles without masking the small 10-30 nm gold particles. The ability of the IBS/e to deposit uniform films over the entire surface area of the samples and to remain featureless in the microscope make it ideal for samples that are irregular in shape and geometry.

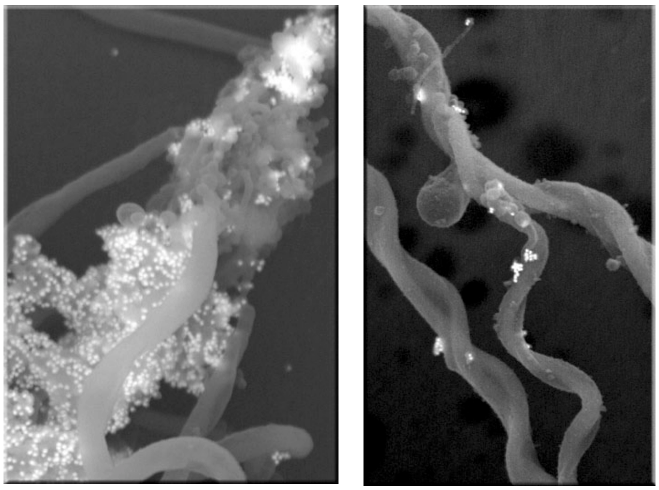

Figure 3: Backscatter SEM images of immunogold stained samples with a conductive layer of Cr deposited onto the samples using the Model IBS/e. The ability of the IBS/e to deposit thin, featureless layers to the sample without masking the gold particles is an important feature. (Images courtesy of D. Dorward, National Institute of Health)

The quality of the image is also an important factor when considering a coating process. Thin, continuous, and uniform deposition of the coating material is critical for samples that are prone to charging. Figure 4 shows an image of a softwood fiber material that is a chemical bleached pulp product. The sample was freeze dried and then iridium coated for 5 minutes using the Model IBS/e. Individual fibrils can be seen in the image that would otherwise be masked using other coating methods. The image was taken at 1 kV at 10,000x magnification.

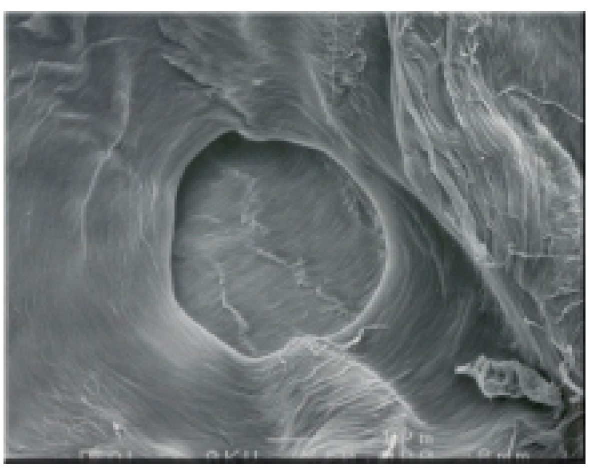

Figure 4: SEM image of a softwood fiber material as coated with Ir using the Model IBS/e. The image shows good resolution of the fibrils which are not masked by the coating material. 1 kV, 10,000x magnification. (Image courtesy of G. de Silveira)

Materials Applications

Materials samples also require conductive coatings for examination in many EM applications. Both SEM and TEM require the samples to have a uniformly deposited layer of material over the entire area of the sample. The deposited film can be affected by the rotation and tilting capability of the IBS/e stage, and the ability of the stage to create ideal deposition conditions to enhance the uniformity is another added feature of the system.

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Pd/SiO_2 particles can be used as model catalysts when deposited with a thin continuous metal film. Figure 5 shows a TEM image of Pd/SiO_2 particles deposited with 8Å of tungsten metal using the Model IBS/e system. The sample was rotated continuously and tilted at 30° during the deposition process to ensure a uniformly deposited layer. As can be seen in the image the tungsten film is amorphous and very continuous over the entire surface of the particle.

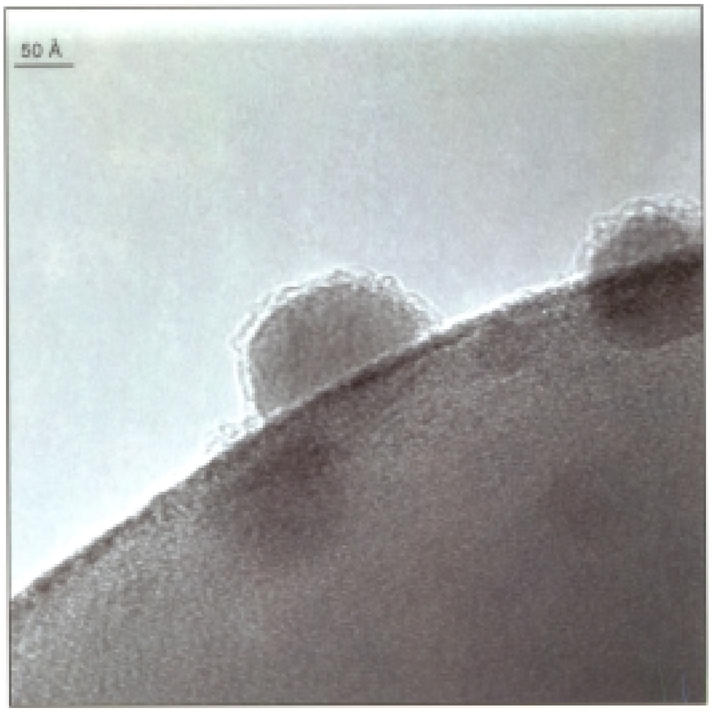

Figure 5: TEM image of Pd/SiO_2 particles deposited with 8Å of tungsten film using the Model IBS/e. From the image it is clear that the film is thin, continuous, amorphous, and uniform over the entire sample area. (Image courtesy of L. Allard, Oak Ridge National Lab and D. Joy, University of Tennessee)

Another example of the amorphous nature of IBS/e deposited films is shown in Figure 6. This image shows both an SEM image (at left) and TEM image (at right) of a carbon black powder material. The sample was deposited with 40Å of chromium for both the SEM and TEM, and the sample was imaged at 250,000x magnification. The lack of grain structure on the surface of the carbon powder particle clearly shows the amorphous nature of the deposition process along with the ability to image the sample without an artifacts present from the coating.

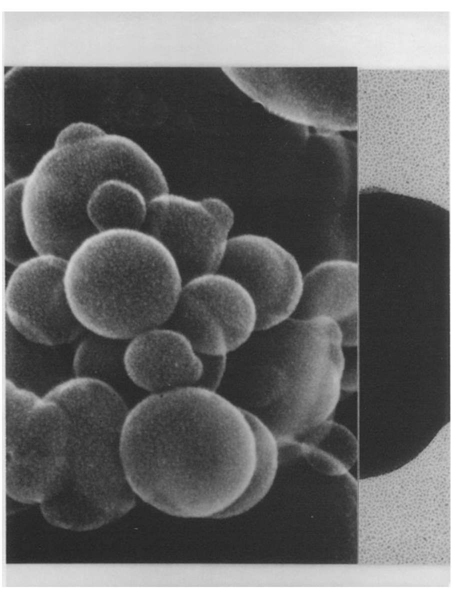

Figure 6: SEM and TEM image showing the carbon black powder as deposited with 40Å of chromium using the Model IBS/e. The SEM image on the left shows no visible grain structure at 250,000x magnification and allows for accurate imaging of the sample without artifacts present. The small TEM image shown on the right has the carbon black particle suspended on a Carbon support film. No grain structure is visible in this image as well. (Image courtesy K. Ogura, JEOL LTD.)

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Conclusion

It is clear that the Model IBS/e can be used to deposit thin, continuous conductive films for various EM applications and is a valuable tool in any microscopy laboratory. The ability to exploit the advantages that ion beam sputtering has over other tools, coupled with the unique stage design, make the IBS/e system uniquely equipped to handle virtually any application that may arise. Applications to thin film research, surface cleaning, ion beam etching of materials, and general microscopy can all be explored and improved using the ion beam deposition system.

References

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3. Image courtesy of William McManus, Dept. of Nutrition and Food Science, Utah State University.
4. Images courtesy of D. Dorward, National Institute of Health.
5. Image courtesy of G. de Silveira.
6. Image courtesy of L. Allard, Oak Ridge National Lab and D. Joy, University of Tennessee.
7. Image courtesy K. Ogura, JEOL LTD.