APPLICATION NOTE

3D coating of cement and ceramics

LAXOR

At LUXOR SEM Coating, laboratory tests are routinely performed to assess the feasibility of coating a wide range of samples. The aim is to achieve optimal coating results for every client, regardless of how unique or challenging the material may be. This application note describes the development of a gold coating method for cement and ceramic samples.

Cement and ceramics

Cement and ceramics are both widely used in construction and engineering, but they serve different purposes. Cement is a fine powder, mainly from limestone and clay, that acts as a binder. When mixed with water, it hardens and holds materials together, making it essential for concrete and mortar in infrastructure.

Ceramics, on the other hand, are inorganic, non-metallic materials made by shaping and firing substances like clay at high temperatures. They are hard, heat-resistant, and durable but often brittle, and are used in everything from bricks and tiles to advanced applications in electronics, engineering, and medicine.

Why use electron microscopy, and what are the challenges when preparing cement and ceramics samples for SEM analyses?

Electron microscopy enables detailed analysis of the microstructure of cement and ceramics, supporting investigation of mechanical performance and failure mechanisms as well as evaluation of surface topography and chemical composition.

What is sample charging?

SEM images are generated by scanning an electron beam across the sample. This effectively adds electrons to the sample. Sample charging occurs when samples are bad electrical conductors which means there is no conducting path for electrons to flow from the sample surface towards the sample holder. Sample charging causes all kinds of problems such as drift, blur, and low contrast. In other words, blurry and false images.

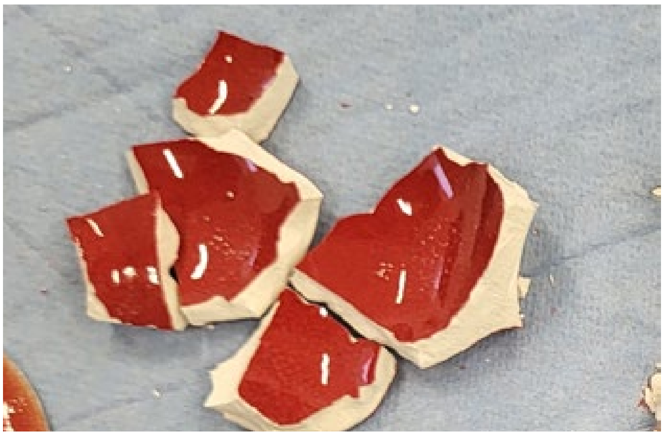
By applying a very thin electrically conducting layer of metal such as gold or platinum (a process known as metal coating or sputter coating) onto the surface topography of the specimen, the electrons can flow from the sample surface towards the sample holder and sample charging is prevented. Other positive effects from sputter coating a sample are an improved secondary electron emission, a reduced beam penetration with improved edge resolution and a better protection of electron beam sensitive samples.

Development of a gold coating method for cement and ceramic samples

The cement and ceramic samples received in the lab presented several interesting challenges.

- Cement and ceramic samples often have irregular shapes, making it difficult to secure them onto SEM stubs with double-sided carbon tape. To address this, the surface is flattened using sandpaper, a file, or a sanding machine to ensure proper mounting.
- Moisture is another common issue. Due to their porous nature and composition, these materials can retain water, so thorough drying is essential before SEM coating in a vacuum. This helps prevent coating defects, ensures reliable imaging, and protects the SEM column from contamination or damage caused by evaporating moisture.
- Additionally, the complex three-dimensional structure of these samples can make uniform coating difficult, requiring careful process adjustments to avoid shadowing effects.

These challenges highlight the value of feasibility testing and parameter selection based on sample porosity, geometry, and moisture content. At LUXOR SEM Coating, we see this as an opportunity to innovate and deliver the best possible results for our clients.



Coating approach with LUXOR SEM coaters for cement and ceramics

Step 1: Pre-treatment: removing liquids and gases

To tackle the challenge of trapped water, solvents, or gases in cement and ceramic samples, LUXOR coaters use specialized pre-treatment methods before coating. High-vacuum evaporation effectively removes volatile substances, thoroughly eliminating gases, moisture, and solvents. In addition, dehydration protocols under low-vacuum conditions gently drive off water without harming sensitive materials. By combining these techniques, LUXOR ensures samples are fully prepared, reducing the risk of vacuum instability and enabling high-quality coating and imaging results.

Step 2: 3D Coating

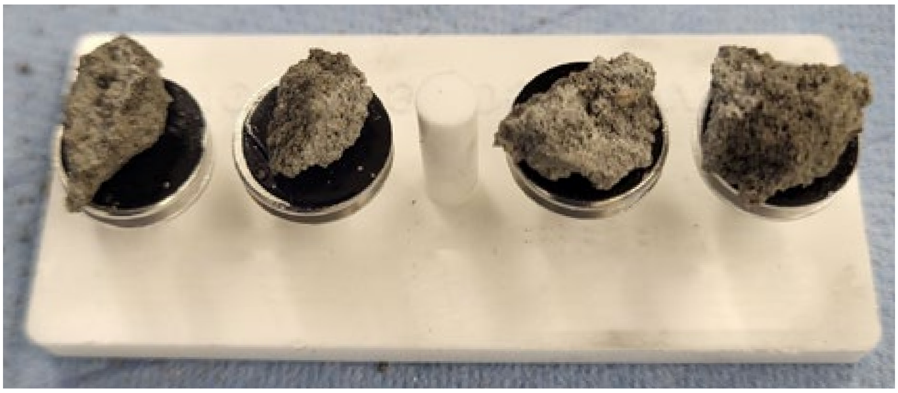
LUXOR coaters feature advanced A² technology, delivering fully automatic and reproducible coating results. This system maintains a stable vacuum with minimal fluctuations, allowing for a consistent, very low coating current.

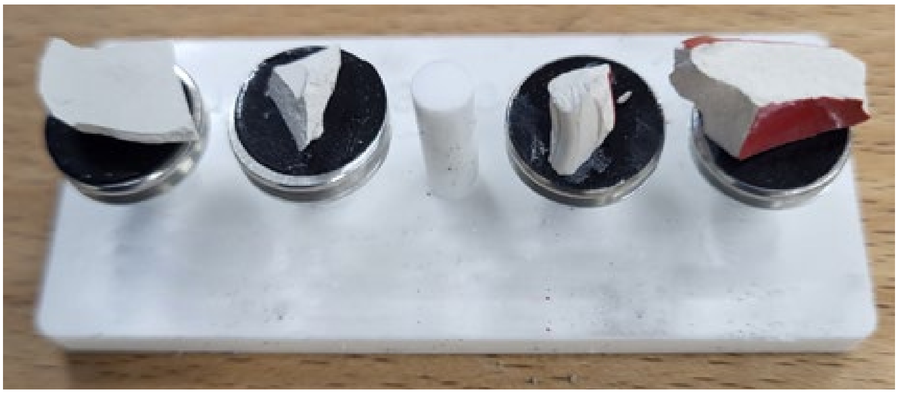
Low coating currents are crucial in SEM coating, as they produce smaller particles that form a uniform, fine coating layer. In LUXOR coaters, the target sits at the bottom of the chamber while samples are positioned at the top; an “upside-down” design. This ensures that only the finest particles reach the sample surface, while larger particles fall away.

This approach, known as **3D coating**, allows the coating material to penetrate porous and complex three-dimensional structures, providing smooth and uniform coverage even on irregular surfaces. As a result, additional equipment such as rotating or planetary stages is not required.

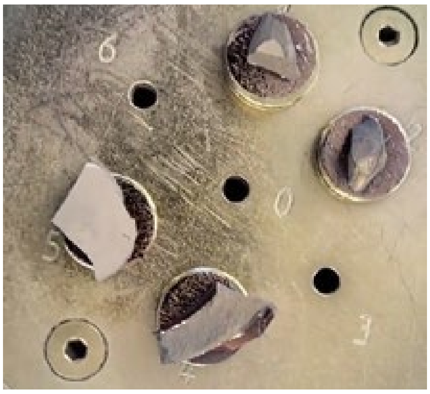
Lab procedure

For both cement and ceramic materials, the same sample preparation procedure was used to maintain consistent analysis conditions.





Ceramic samples before coating



Ceramic samples after coating

- The samples were first mechanically reduced in size and flattened on one site to create a smooth surface for mounting on the carbon tape.

- Next, the pieces were attached to 12 mm stubs using double-sided adhesive carbon tape. Four samples were processed simultaneously for both cement and ceramic, increasing the total surface area and making it more challenging to achieve the required vacuum conditions.

- An high vacuum evaporation step was then performed to remove residual moisture and stabilize the vacuum. Cement samples required more than 15 minutes to remove sufficient moisture to achieve a stable vacuum level suitable for sputter coating, while ceramic samples reached this level in about 6 minutes, reflecting differences in porosity and moisture retention.

- Finally, a gold coating of 25 nm was applied. Although 5 nm to 10 nm of gold coating is sufficient for most types of samples, a thicker layer was used to ensure proper conductivity, considering the porosity and three-dimensional surface features of both cement and ceramic samples.

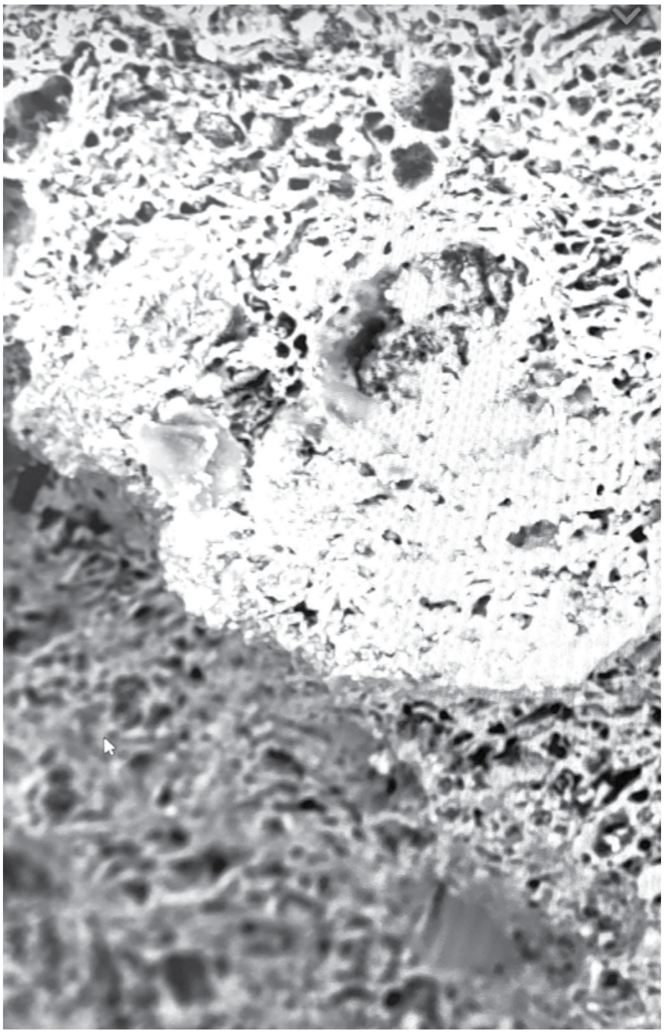
SEM imaging: conditions and results

SEM imaging was carried out on a Thermo Phenom XL G2 electron microscope. Images were acquired using backscattered electron (BSD) detection at 20 keV and a chamber pressure of 0.1 Pa, with a magnification of 1,500x to clearly highlight surface features and structural details.

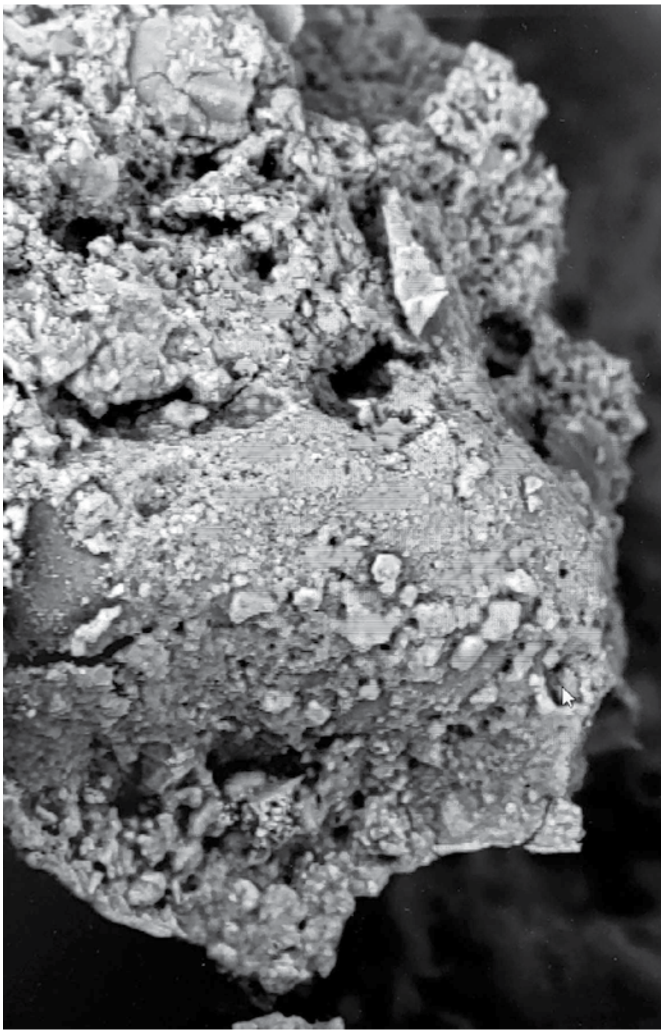
Uncoated ceramic and cement samples in SEM holder

Coated cement samples in SEM holder

Uncoated cement sample charging in the SEM



Coated cement sample not charging in the SEM



Why choose LUXOR?

A² technology: precision coating for high-resolution imaging

LUXOR's unique and innovative A² technology generates a plasma with exceptional precision and control, resulting in a uniformly thin and homogeneous coating. This process begins by creating a vacuum in the chamber, followed by the application of a high voltage. To ensure stability, the resulting coating current is automatically regulated by introducing small amounts of process gas into the reactor, allowing the target current to be reached and maintained.

What distinguishes LUXOR metal coaters from other commercially available instruments is the unique method used to control and adjust this process. For scanning electron microscope (SEM) operators, this means obtaining more consistent and uniform metal coatings, which significantly enhance image quality with higher resolution and contrast. Additionally, the coating process is fully automated, removing the need for manual intervention and allowing for a seamless and worry-free experience.

Innovative upside-down design for greater functionality

At LUXOR, we adhere to a 'form follows function' philosophy, which is why our metal coaters feature a unique design where the samples and target are mounted upside down. While this may seem unconventional at first, it offers several significant advantages.

- **Safety first**
  All voltage and current wires are securely housed within the sputter device, significantly reducing the risk of electric shocks. This safety feature allows you to operate the machine with complete peace of mind.

- **Effortless sample handling**
  The upside-down design makes the lid, which doubles as a sample loading station, easily accessible. This allows you to apply or remove samples quickly, without the need for special tongs or tweezers. This simplicity not only enhances ease of use but also boosts productivity by streamlining the process.

- **Clean coating process**
  This design ensures that loose particles are removed during the coating process, helping to keep your Scanning Electron Microscope (SEM) optimally protected from debris. Consequently, this contributes to more consistent and reliable results.

- **3D coating**
  Additionally, larger particles in the plasma are drawn towards the pump rather than reaching the samples due to their weight, resulting in improved coating quality. This effect, combined with the algorithms of our A² technology, eliminates the need for a rotary or planetary table, greatly simplifying the setup for coating three-dimensional and porous samples.

Fully automated

The coating process is completely automated. Once the samples are loaded, simply select the desired coating thickness and press the start button. This user-friendly system greatly reduces the likelihood of human errors. New operators and lab personnel can learn to operate the device within just a few minutes of basic training.

In our E-BOOK "INTRODUCTION TO METAL COATING TECHNOLOGY FOR ELECTRON MICROSCOPY" you can learn more about sample charging, ways to avoid it, and all there is to know about sputter coating.

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