
Leica EM ACE200 Sputter Coating: Quick Start Guide for Science Users

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Introduction

With the Hitachi TM3000 SEM, sputter coating is not required for all samples. Images up to a magnification of $\times 4000$ are of high quality without any sputter coating once the **Charge-up Reduction Mode (Settings > Observation Mode)** is selected.

For detailed observations in magnifications greater than $\times 4000$ and very delicate fossils, sputter coating before using the SEM can be used to potentially receive higher quality images.

Apparatus and Materials

The Leica EM ACE200 coating system includes the following main functional units (*Figure 1*):

- Vacuum chamber
- Touch screen control panel
- Sample stage with 18 positions for 1/2" SEM stubs
- Carbon source and sputter targets
- Vacuum pump



Figure 1.

Targets

The following targets are used for sputter coating:

- Gold
- Gold-Palladium

Argon gas supply

The working gas (argon) must be supplied under a pressure of ~ 500 mbar (± 100 mbar). The gas should be at least 99.99% pure.

Sputter coating

The coating process can be set to directional or diffuse (which provides more even coating on a bigger surface and is better for fissured samples). This also influences the coating rate (diffuse means slower rate) and the grain size (directional means finer grains).

Setting up a Sputtering Process

Sputter Parameters

Argon: switch on the argon.

Target material: select from a drop-down list; the instrument automatically chooses parameter settings.

Directional mode: intended for relatively flat samples; base vacuum is 4×10^{-2} mbar for this mode.

Diffuse mode: intended for more topographic samples; base vacuum is 8×10^{-2} mbar for this mode.

Sputter current/purge cycles: automatically set when target is selected. Change under the **Setup** button.

Process termination time: select the **Clock** icon then use the +/- buttons to select a time in seconds.

Rotation speed: press the **Rotation** button, check **Rotation On**, and use the +/- buttons to set the speed.

1. Turn on the main power supply to the instrument, make sure the chamber door is closed, the cover is closed, and the sputter head is connected, and switch on the argon.
2. Select the **Sputtering** button.
3. Press the **Pump** button to start pump-down.
4. During pump-down, set the process parameters:
 - Target material (last material used is preselected)
 - Mode (**Directional** or **Diffuse** button)
 - Sputter current/purge cycles (preset when target was selected)
 - Process termination time (e.g., 12 sec)
 - Rotation speed (e.g., 1)
5. Press the **Start** button (which then becomes a **Stop** button).

Note: if the vacuum cannot stabilize, make sure the argon supply is open.

Sputter Process Start

The coating process can be started at any time (system evacuated or not) as long as the door and source cover are closed and the source is connected. The **Start** button will turn into a **Stop** button when the system starts the coating cycle.

Once the **Start** button is activated the coater automatically runs the complete coating cycle. The system either stays under vacuum or vents automatically (if **Vent after process** is activated).

Pressing the **Stop** button terminates the process after confirmation, regardless of the step of the process. The system either stays under vacuum or vents automatically.

After setting coating parameters and pressing **Start** the system performs the following steps automatically.

1. Pump until base vacuum is reached.
2. Stabilize the plasma.
3. Pre-sputter, if target requires (to clean the target from oxidation and enable a stable sputter rate).
4. Start the sputtering process by opening the shutter and starting rotation (if activated).
5. Terminate sputtering by time.

6. Close the shutter.
7. Display results of the process.
8. Vent or stay under vacuum, per settings.

Carbon thread coating

The carbon coating process is carried out by evaporating a carbon thread. It is possible to coat using short pulses of 150 milliseconds or evaporating the thread completely with maximum power, a so-called flash.

Loading a carbon thread

The carbon thread can be loaded as a single thread or as a double thread. Thin layers from 1 to at least 20 nm can be achieved (there is variance in threads).

To minimize carbon thread waste, when loading a double thread, cut a piece of thread twice as long as the width of the black door frames of the coater. Fold the thread into half and load it as follows (there is an ACE200 youtube video where the loading of the thread can be seen at <http://www.youtube.com/watch?v=yKd1lyKIDT8>).

Before beginning, prepare the following on a clean surface: gloves, carbon head, Torx TX 10 key, brush, carbon thread.

1. Make sure the instrument is vented.
Wear gloves.
2. Open the source cover and unplug connectors.
3. Unscrew the 2 evaporation head screws to remove the flange.
4. Loosen all 5 clamp screws with the Torx key.
5. Remove any carbon residue using the brush.
6. Loop the carbon thread around the first clamp, then pull both ends gently to the left while tightening the screw.
7. Wind the thread around the other clamps, taking care that the thread slides into the clamping groove.
8. Pull the thread gently and tighten the last clamp.
9. Tighten the 3 remaining screws and trim thread on both ends (*Figure 2*).
10. Replace the head, gently tighten the fastening screws, connect the cables, and close the cover.



Figure 2.

Processing Carbon Thread Coating

After setting coating parameters and pressing **Start** the system performs the following steps automatically.

1. Pump until base vacuum is reached.
2. During pump-down, set the following parameters:
 - Target thread:** select **2** if double thread (the instrument automatically sets parameters)
 - Process pulse/flash:** use the +/- buttons to set number of pulses or flashes
3. Check availability of at least one carbon thread section.
4. Outgas (preheat) the first carbon thread for 45 sec.
5. Open shutter and start rotation.
6. Pulse/flash according to the settings.
7. Close the shutter.
8. Display results of the process (number of pulses/flushes, availability of further threads).
9. Vent or stay under vacuum.

Switching between Processes

To switch between carbon thread and sputter coating, exchange the source (sputter or carbon thread) and choose the process on the main screen of the instrument. The instrument automatically detects if the correct source is installed and gives an error message if it does not match the selected process.

Safety

All electronic components are protected by covers (door, source cover, housing). The door and source covers are equipped with sensors that cut off power when they are opened. Software interlocks cut off power when a malfunction is detected (e.g., vacuum leak, short circuit in the source head, missing contact in the system).

Overheat protection: at 65°C the process is discontinued until the temperature drops to 45°C, then the process continues.

In case of a sudden vacuum breakdown, the instrument switches off automatically to protect pump and electric parts.

If the coating system is damaged or malfunctions, all use of the system should be suspended until the malfunction or damage has been corrected.

Do not operate the coating system unless all covers are properly in place – there is danger of electric shock and burns from high-temperature components.