

## Important note

Please read this instruction manual carefully before operating the instrument. This user manual is intended to provide essential information about the Leica EM ACE200 coating system. It includes important information regarding operation, servicing and troubleshooting. Following these instructions will help to prevent hazards, reduce repair and downtime costs and prolong the system's life.



Danger!

The Leica EM ACE200 coater system can be handled safely and easily provided it is operated in accordance with the instructions in this manual. **Ignoring safety instructions may endanger people and the system.**

Users must familiarize themselves with the system before operation. Particular attention must be paid to the aspect of safety. The Leica EM ACE200 must not be used beyond the limits specified in the technical data sheet.

When hazardous substances (e.g. radioactive, toxic or explosive substances) are processed, the substance-specific safety precautions must be implemented. It is forbidden to process substances that release corrosive or poisonous gases when they are placed in vacuum and/or are coated.

Any warranty claims can only be considered when the instrument is used according to the guideline given in this manual.

In addition, all generally applicable legal and otherwise binding regulations for preventing accidents and protecting the environment must be observed and communicated.

Leica reserves the right to change technical specifications as well as manufacturing processes without prior notice. Only in this way is it possible to continuously improve the technology and manufacturing techniques used to provide our customers with excellent products.

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## Symbols used in this manual

Danger!



Instructions regarding possible hazards are identified with this symbol. **Ignoring these alerts may result in serious injury!** Users of the instrument must comply with instructions at all times.



Caution!

This symbol alerts the user to important information which may endanger staff or result in damage to the system if it is ignored.



Lifting hazard. Single person lift could cause injury. Use assistance when moving or lifting the coater.

Note!



This symbol indicates further information relating to a previous explanation which does not have a safety-critical function. However, it is important to follow this information to ensure that the system functions optimally.



Wear clean, powder-free gloves.

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# 1.Introduction

The Leica EM ACE200 coating system is used for precise coating of samples for subsequent examination with an electron microscope (EM). The optional glow discharge makes grids hydrophilic. Also optional are quartz crystal measurement to accurately determine the layer thickness and a planetary drive stage for even distribution of coating material.

The samples are metal coated using the sputtering method where an argon plasma erodes a target material. Carbon coating is achieved by carbon thread evaporation. Any material can be processed as long as it is not sensitive to vacuum, argon plasma or the heat generated during carbon coating.

The ACE200 can be configured as a sputter coater, carbon coater or consisting of both where the two processes can be switched over from one to the other (see section 3.12).

The removable internal shielding, shutter, source and door are designed to enable easy and comprehensive cleaning of the system

The vacuum system creates an ultimate vacuum  $\leq 7 \times 10^{-3}$  mbar. Pressure is monitored by a thermal vacuum gauge (Pirani) (see technical data sheet for all specifications, 7.1).

## **Main components:**

The Leica EM ACE200 coating system includes the following main functional units, depending on the configuration:

- Vacuum chamber
- Touch screen control panel (see 3.1)
- Sample stage with 18 positions for 1/2" SEM stubs (exchangeable, see 2.6)
- Removable internal shielding, shutter and door (see 4.2.1)
- Housing
- Carbon source, sputter source with the option of either or both (see 3.8, 3.9 and 3.12)

Optional:

- Vacuum oil rotary vane pump (see 2.4)
- Planetary drive stage (see 2.6.1 and 3.11)
- Sample stage for one 76mm x 26mm (3" x 1") glass slide (exchangeable, see 2.6)
- Glow discharge (see 3.10)
- Quartz (QSG) thickness measurement (see 2.7 and 3.7)

## 1.1 Safety

All electronic components are protected by covers (1-door, 2-source cover, 3-housing). The door and source covers are equipped with sensors which cut off power when they are opened. Additionally there are software interlocks which cut off power when a malfunction is detected (e.g.: vacuum leak, short circuit in the source head, missing contact in the system)



There is an overheat protection for the sources. When reaching 65°C the process is stopped until a temperature of 45°C is reached then the process automatically continues.

When there is a sudden vacuum break down the instrument switches off automatically to protect pump and electric parts.

The housing must not be opened except by an authorized Leica representative.

If the Leica EM ACE200 coating system is damaged or malfunctions, all use of the system should be suspended until the malfunction or damage has been corrected.

All modifications and conversions to the system are prohibited and invalidate the warranty.



Note

For maintenance and servicing the system has to be switched off!

Caution!

There is a danger of electric shock when the housing is removed.



Injuries may be sustained that could lead to death.

The Leica EM ACE200 coating system must not be operated unless all covers are properly in place.

Caution!



Some of the components inside the system may become hot and present a danger of injury.

Burns may be sustained.

## Safety measures during installation

The following measures must be implemented to prevent incorrect use:

- Connecting to electricity and gas must be carried out by a technically qualified person.
- Gas bottles must be secured and stand upright when connected. The system must be connected to the gas supply by a technically qualified person.
- Repairs may only be made by Leica authorized service staff or authorized representatives.
- If the Leica EM ACE200 coating system is installed incorrectly, the system may be damaged and may incur injury to the user.

## Emergency procedure



Caution!

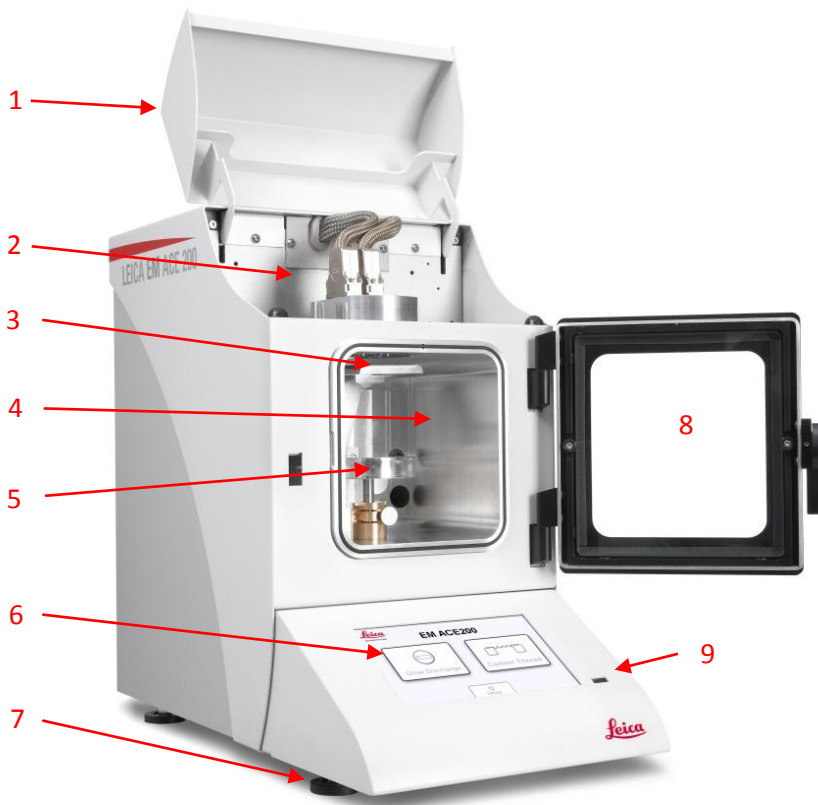
If unusual operating conditions or unaccustomed noises occur, the system must be switched off using the mains switch on the rear of the system.



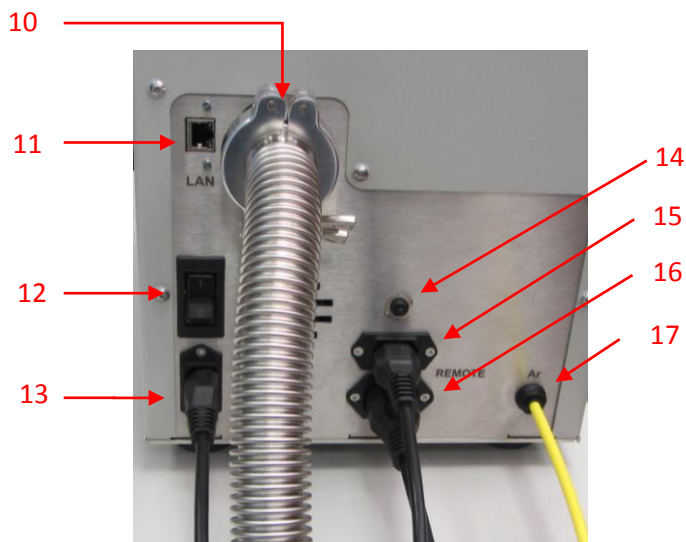
If fire fighting measures are called for, a CO<sub>2</sub> fire extinguisher must be used.

Technical Service must be consulted before resuming work with the system.

## 1.2 Instrument Overview



1. Source cover
2. Source head (sputter or carbon thread)
3. Shutter
4. Chamber
5. Sample stage
6. Touch sensitive control panel
7. Adjustable feet
8. Chamber door
9. USB port



10. Outlet to vacuum pump
11. LAN connection (Inactive, for future connection)
12. Power supply switch
13. Mains power inlet for coater
14. Automatic fuse for the pump
15. Mains power inlet for pump
16. Mains power outlet for pump
17. Argon gas inlet



## 2. Installation and set up

### 2.1 Delivery of the instrument

The Leica EM ACE200 coating system is delivered assembled apart from accessories and the shutter which is packed separately and has to be installed (see 2.4 and 2.6). If the configuration includes a quartz crystal measurement this has to be installed as well (see 2.7).

Please check the condition of the system upon delivery and file a damage report with the shipping company if the equipment is damaged. The customer must immediately inform the Leica representative for any possible damage in transit.

### 2.2 Installation requirements

A working area of about 150mm must be maintained all around the system to allow the supply connections to be made and also to enable access for essential servicing activities by the customer. The Leica ACE200 has to be set up on a stable laboratory workbench with a surface area of at least 660mm depth and 410mm width. The coater is 450 mm high. When selecting a setup location, bear in mind that the system weighs approx. 34 kg. To avoid vibrations do not place the pump or other vibrating instruments on the same bench as the coater.



The instrument must be placed on the bench in such a manner to allow access to the mains switch and mains plug at any time!

External elements (dust, grease, etc.) may prevent the production of the required vacuum.



When working on the vacuum chamber or parts which are in the vacuum chamber of the Leica EM ACE200 coating system it is essential to follow the principles of vacuum hygiene. Gloves must be worn when disassembling and assembling components in the vacuum area, and also for all adjustment work.

All work must be carried out in a clean, oil/grease-free and dust-free environment.

The following connections need to be prepared:

- One electricity supply: 100/115/230VAC, 50/60 Hz
- Pump, maximum power 1000W
- Argon (only if sputter option is available), reduced pressure:  $\sim 500$  (+/- 100) mbar, purity: min. 99.99%

## 2.3 Unpacking and connection

The Leica EM ACE200 coating system should be transported with a forklift truck

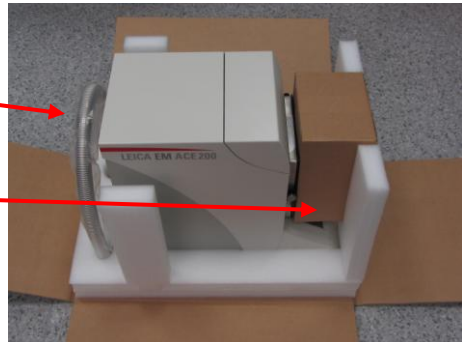


Remove bands and open box



Fold down the sides of the box and remove vacuum hose (if ordered)

Remove accessories box



Lift out the unit by grasping underneath the back and the front as indicated in the picture (packaging recess in for hands) and place it on a table



Help from a second person is advisable to prevent any injury

Place the ACE 200 on a stable bench

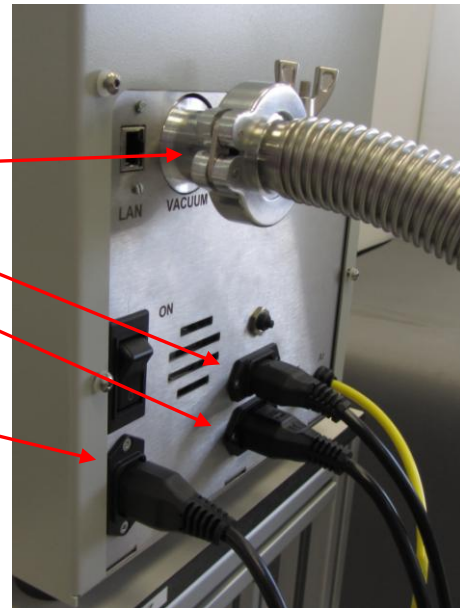
Adjusted all feet and counter lock (flat spanner 13mm) to maintain level and stable positioning.

Connect vacuum hose to the ACE200

Connect MAINS IN cable for rotary pump

Connect MAINS OUT cable to rotary pump

Connect mains cable



**For sputtering process only:**

Connect argon gas (6mm diameter)

Purge argon line before connecting

Regulate argon pressure to ~0.5 bar over atmosphere



We strongly recommend using a two-stage pressure regulator on the argon bottle



## 2.4 Pump set-up

If a pump is not included, a roughing pump with a capacity of 5m<sup>3</sup>/hour or higher and an ultimate vacuum of at least 5x10<sup>-3</sup> mbar is needed. This has to be attached to the coater (KF 25 ISO flange, see picture below)



If the delivered parts include a rotary vane pump please set up the pump as described below.

Open the box and unpack the rotary pump

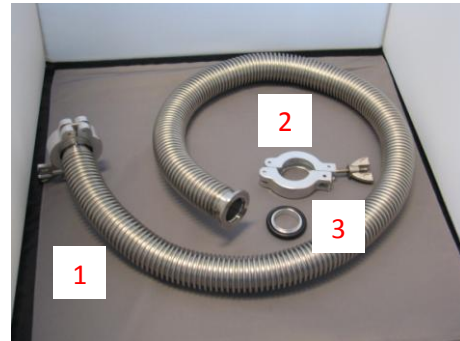


Help from a second person is advisable to prevent any injury

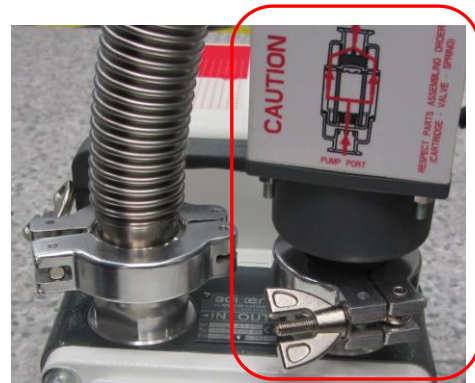


A hose ordered with the instrument includes

1. A metal hose
2. Two clamps
3. Two seals



With the oil pump (If ordered) you will get an oil mist filter, another clamp and a seal (like above). Install oil mist filter to rotary pump exhaust flange using the seal and clamp (see 3 and 2 above)



Fill the pump with oil included in the pump delivery to the level indicated by the white bar

Min. / max. Level indication



Attach the hose to the pump and coater using the delivered seal and clamp



## 2.5 Shutter installation

Remove the shutter from the packaging

- Shutter without the glow discharge electrode

Thumb screw (1)

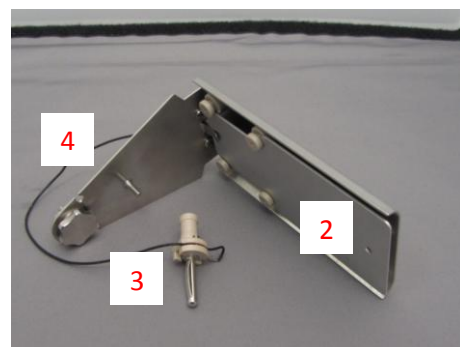


- Shutter with a glow discharge electrode and contact (optional)

Electrode plate (2)

Glow discharge connection (3)

Glow discharge cable (4)

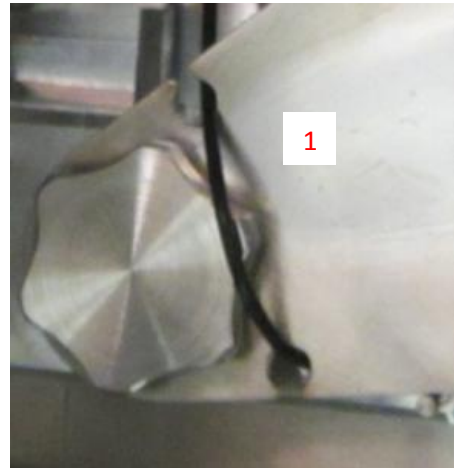


Screw the shutter into the hole in the middle of the rear of the coating chamber by the thumb screw

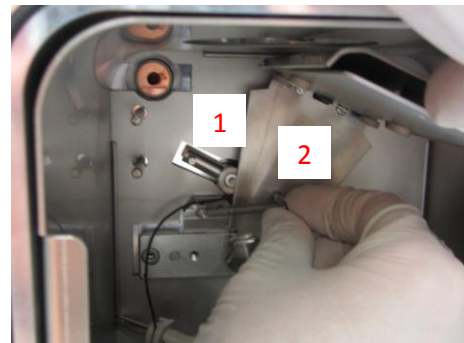
Tighten firmly



If a glow discharge is present guide the cable through the recesses on the shutter (1) and plug in the connection (2)



Slide the shutter spring (1) on to the pin on the shutter (2)



## 2.6 Stage installation

The coater is delivered with a stage to hold up to 18 SEM stubs. Optional stages for a glass slide or a planetary drive stage are available.

This stage is used for loading up to 18 standard 1/2" SEM stubs with a pin diameter of 3.2mm



This optional stage is used for one 76mm x 26mm (3" x 1") standard glass slides



The stage consists of

1. Stage with a fixing screw
2. Rod to adjust the height with grooves every 10 mm

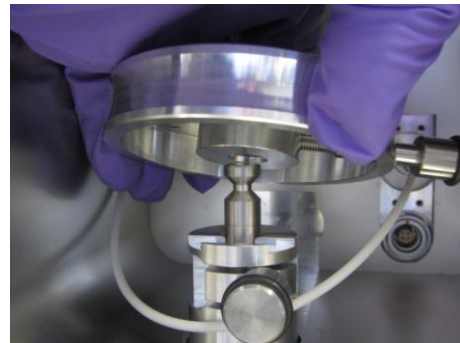




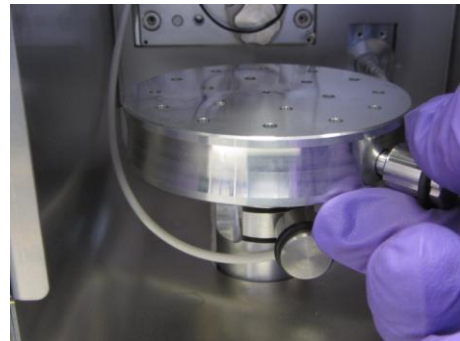
Take the rod and place it as shown in the coating chamber



Place the stage on the rod (stub, glass slide or planetary)

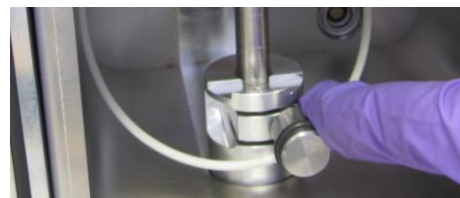


Tighten the thumb screw

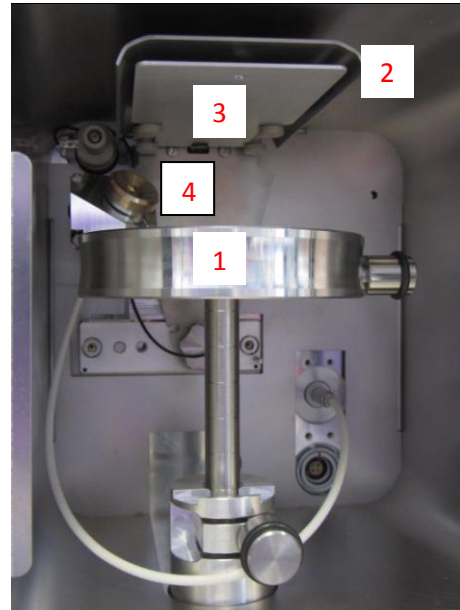


To remove a stage, reverse the above procedure

Adjust the height to the desired level and tighten the thumb screw (see also 2.4.2)



The vacuum chamber equipped with a sample stage (1) a shutter (2) a glow discharge electrode (3) and a thickness measurement quartz (4)



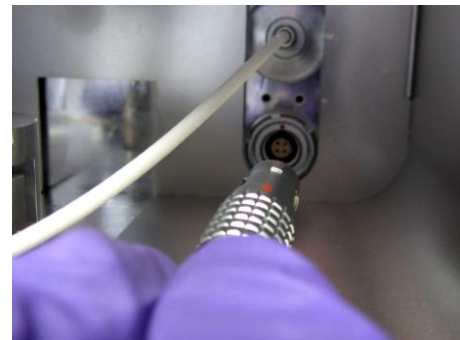
### 2.6.1 Planetary drive stage

The planetary stage is an optional outfit. When ordered, the instrument is outfitted with the necessary feed-through and motor control.

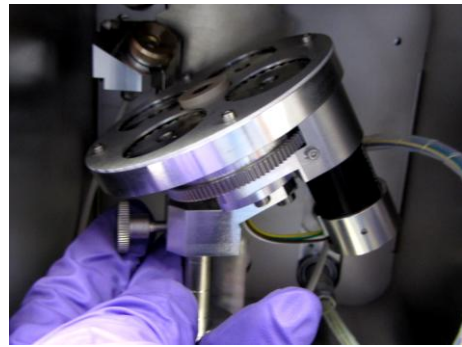
Up to 16 1/2" SEM stubs can be loaded and processed in one session



Face the red dot on the connector upwards and connect to the socket in the rear right side of the chamber



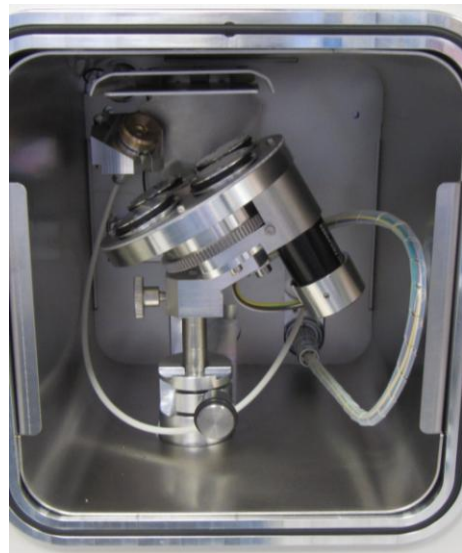
Place the planetary drive stage on the vertical rod and fasten it by tightening the thumb screw



When placing the x4 SEM stub holder take care to align the undelaying pin into the hole in the sprocket. This can be easily done by turning the stub holder till it drops into place



Installed planetary drive stage



## 2.6.2 Setting the stage height

On the defined zero reference mark (lower reference pin) the distance to the target is 30mm. Grooves of 10mm distance on the rod allow an accurate setting of the sample height.

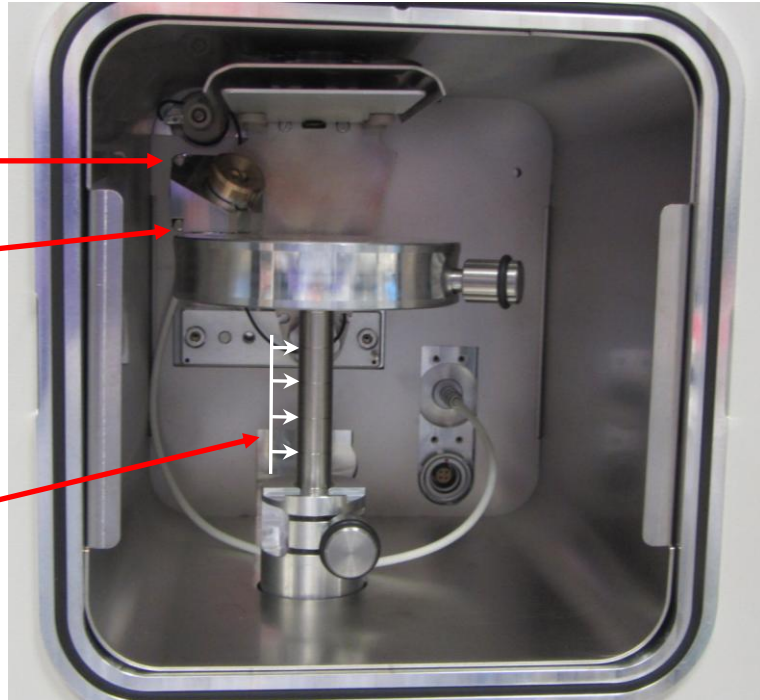
### Upper reference pin:

Pin aligned with table/sample surface is the minimum distance from source (30mm, correction input for quartz 20mm)

### Lower reference pin:

Pin aligned with table/sample surface is the **0 reference mark** (distance from the source 50mm, no correction for quartz required)

**-** **0 mm** **+** every 10mm  
to read out the height correction for quartz measurement



The height adjustment is especially important for the quartz film thickness measurement. For example, if the sample surface is 10mm below the defined 0 mark, then -10mm has to be set (3.7, last image) for the stage height correction. If the surface is 10mm above 0 mark, this means +10mm correction. The instrument will calculate the right tooling factor for the quartz thickness measurement (see 3.7).

## 2.7 Quartz crystal measurement installation

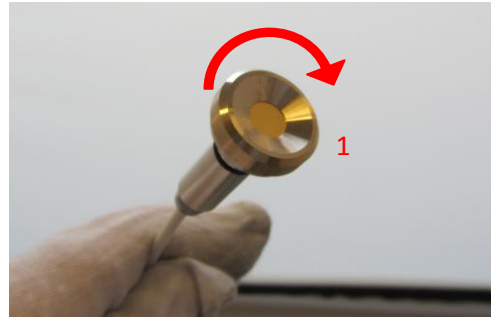
The quartz crystal measurement (QSG) is an optional feature for accurately determining the thickness of the applied coating layer (see further 3.7).

The QSG equipment for installation includes

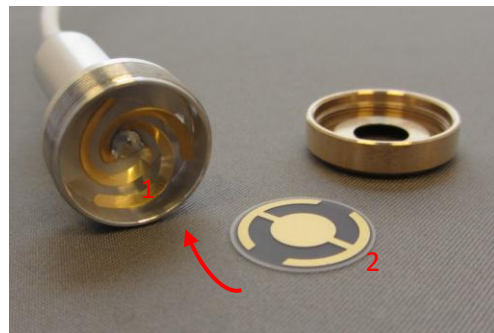
1. Quartz head with cable
2. Holder
3. Quartz crystals



Unscrew the cover of the quartz holder



Place a quartz crystal on the copper springs (1), the grey part facing downwards (2)



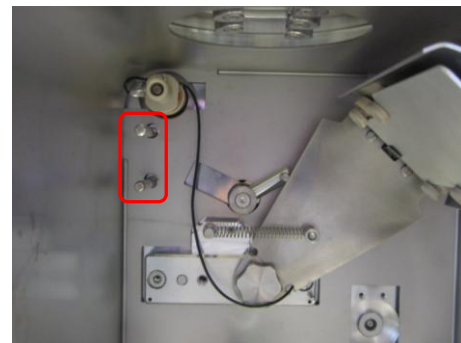
Replace the cover and tighten gently



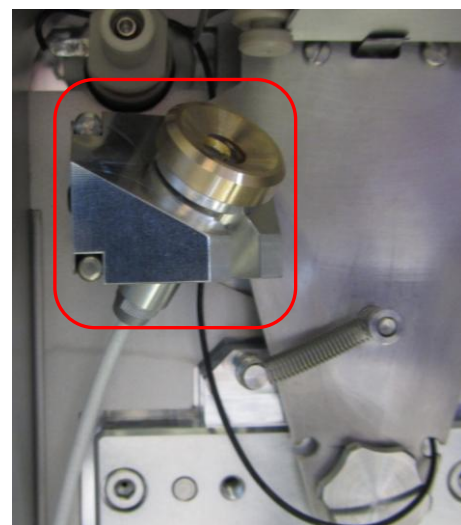
Place the quartz holder into the adapter block by sliding it in, the quartz head on top and the cable on the bottom



In the rear left of the coating chamber are two pins to receive the quartz holder



Place the quartz holder block into coating chamber by push sliding it onto the two pins



Connect the plug of quartz measuring cable to the feed through in the rear right of the coating chamber



For better access the sample stage can be removed

Replace the sample stage (see 2.6)



# 3.Operation

## 3.1 Touch screen control panel

The LCD control panel is used for communicating with the Leica EM ACE200 coating system as well as for data input and output. The parameters for the coating process are edited via the touch screen.

For operating the screen a touch screen pen can be useful. For calibrating the screen a pointy but round device has to be used (like a touch screen pen)



Be careful with too pointy devices, the touch screen panel may be scratched.

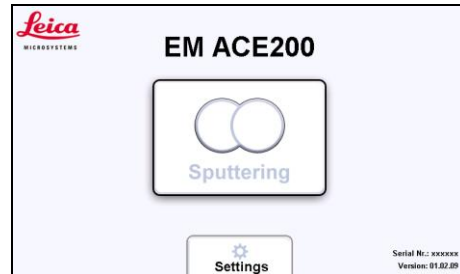


The coater is delivered calibrated. If the touch seems to loose it's accuracy calibrate the screen (see 3.1.1)

After switching on, the first screen will show the options according to the configuration you ordered:

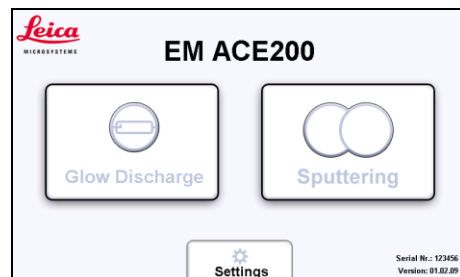
An instrument equipped with:

- Sputtering



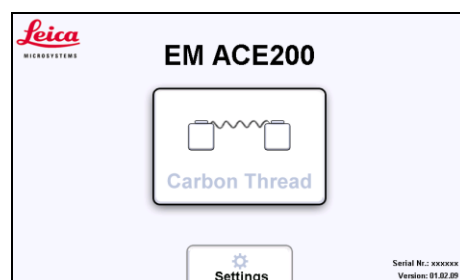
An instrument equipped with:

- Sputtering
- Glow discharge



An instrument equipped with:

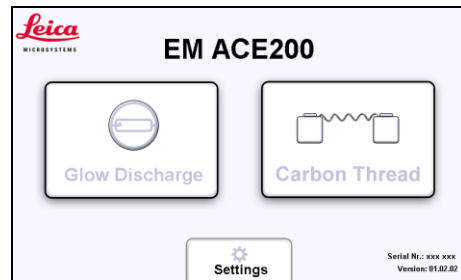
- Carbon thread





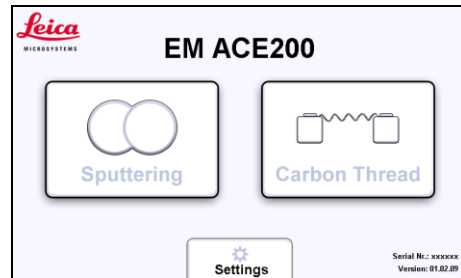
An instrument equipped with:

- Carbon thread
- Glow discharge



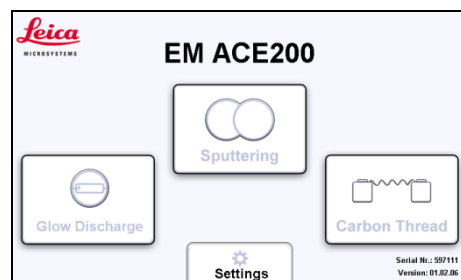
An instrument equipped with:

- Sputtering
- Carbon thread




An instrument equipped with:

- Sputtering
- Carbon thread
- Glow discharge



The software can be updated by the customer using a USB stick. During the update the instrument must not be switched off otherwise the system may not function.

### 3.2 Settings

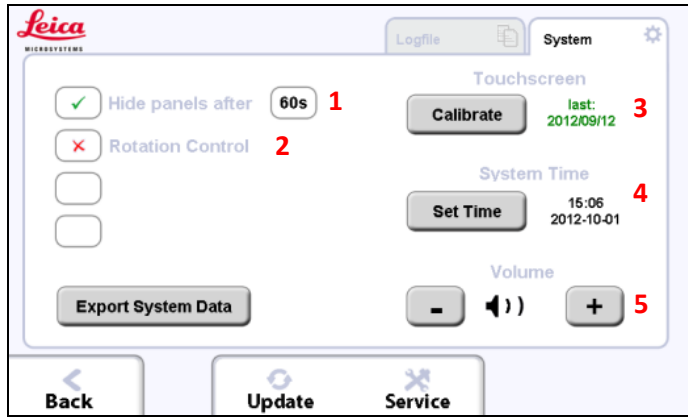
Push  on the first screen after switching on the instrument to set time and date, touch screen calibration, volume level and to download log files. Software updates can be performed (by the user) and the service interface accessed (by Leica service).

1. Time until a panel will close automatically
2. Rotation control activated (when planetary drive stage is present)
3. Calibrating the touch

screen (only if needed)

4. Set system time
5. Set volume

To confirm press 

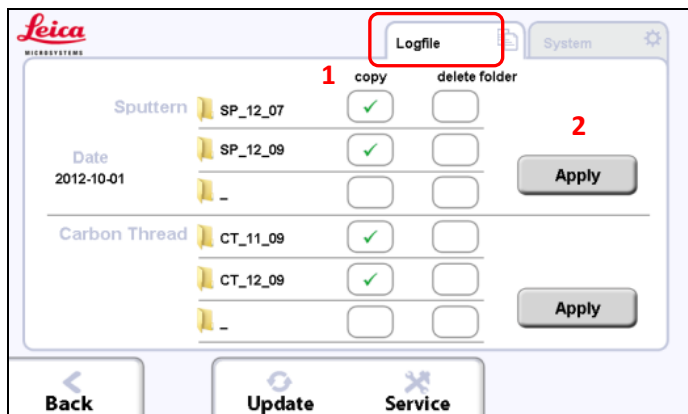


The coater is delivered calibrated. If the touch seems to loose it's accuracy calibrate the screen (see 3.1.1)

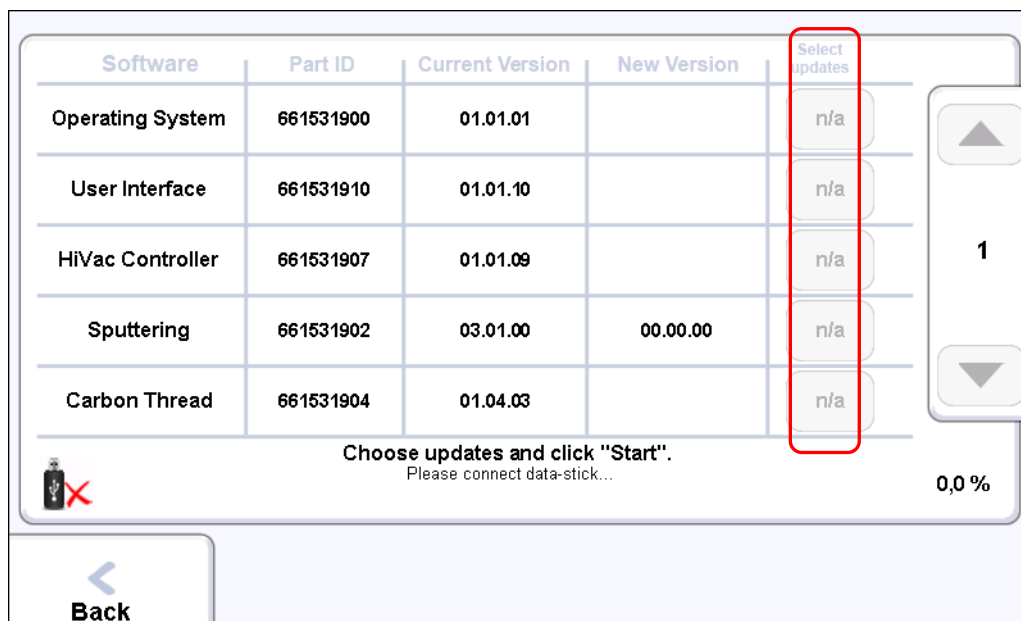
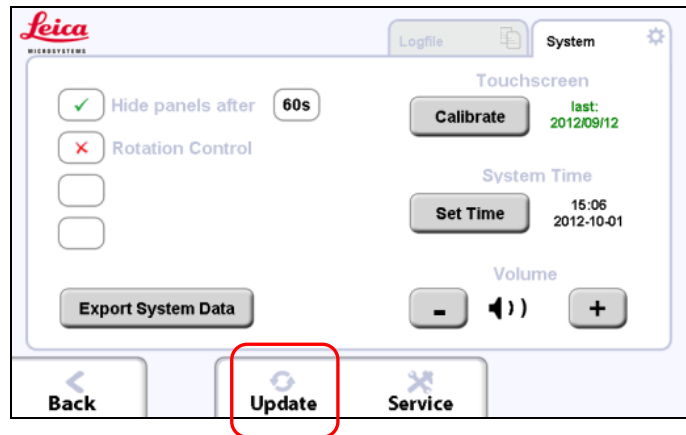
Connect a USB stick to the port on the touch screen panel and select 'Log file' tab on the settings screen to download log files



1. Select the files which shall be exported
2. Press "Apply" to copy them on the USB stick



Enter the menu, connect a USB stick to the port on the touch screen panel (see 3.1.3,) and press update





Choose from the list which updates shall be performed by pushing the respective button on the select update column



It is recommended to first update the user interface and then the controller update

### 3.3 Pump and Vent

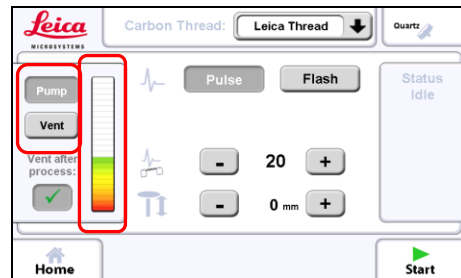
The system can be pumped down at any time. It can be vented at any time when a process is not running. The vacuum bar indicates the pumping and venting progress.

For pump or vent press the respective button  and  on the left side of the main screen on the control panel (see screen below). If the door is not closed the

pumping cycle will not start and an error message will appear. A time out message shows if there is a leak when pumping down (e.g. the source is not placed correctly).

Pump and vent buttons


The vacuum level is visualised using a vacuum bar

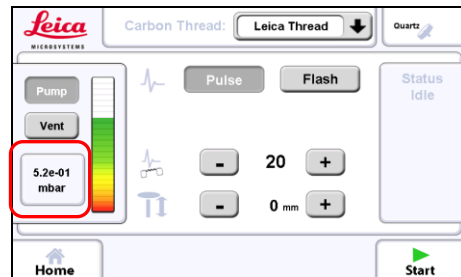


Tapping on the vacuum bar will show the vacuum read out

Tapping on it again, the panel disappears.



In  (Main screen) it is defined for how long panels are shown before they close again



The instrument should not be left under vacuum it could happen that small amounts of oil can be sucked from the pump into the coating chamber. Do not switch of the pump separately always operate it via the coater interface.

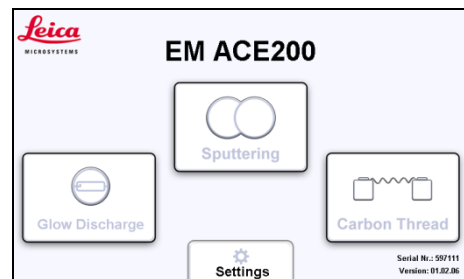
### 3.4 Vacuum test

After connecting the coater as described in 2.3, the coater can be tested for vacuum.

Switch on the instrument on the rear by pushing the main switch up



Select any of the processes



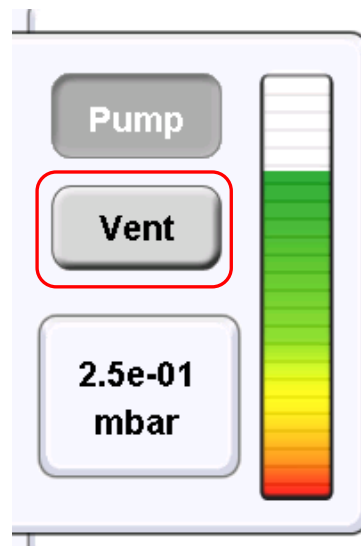
\*Screen shows instrument equipped with three processes

Push the pump button to start pumping down.

- When tapping on the pressure bar the current vacuum will be displayed



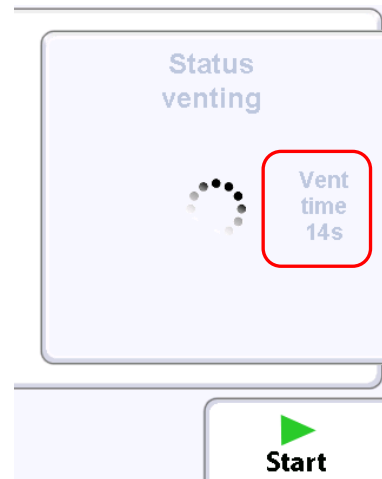
If the vacuum does not reach 0.5 mbar after 30s, the pump has to be checked or a vacuum leak needs to be found.





To vent the chamber push the



button  
Venting in progress, vent time counting



### 3.5 Process start and stop

The coating process can be started at any time (system evacuated or not) as long as the door and source cover are closed (electronic safety switches prohibit starting with open door or source cover) and the source is connected. The  button will turn into a  button when the system starts the coating cycle.

Once the “Start” button is activated the coater will automatically run the complete coating cycle. The system either stays under vacuum or vents automatically (vent after process is activated, see section 3.6)

Pressing the “Stop” button will terminate the process after confirmation, regardless of the step of the process. The system either stays under vacuum or vents automatically (vent after process is activated, see section 3.6)

### 3.6 Vent after process

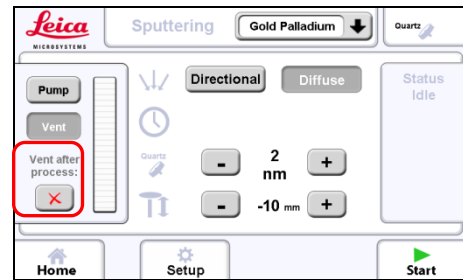
There are two options for the state of the system after the coating cycle is finished. Either the instrument stays under vacuum or it is vented to take out the sample.

Vent after process is activated, the system automatically vents when coating process is finished



Vent after process is deactivated, the pump keeps running after coating process

Venting is done manually by pushing the “Vent” button





### 3.7 Thickness monitoring and geometrical correction (QSG)

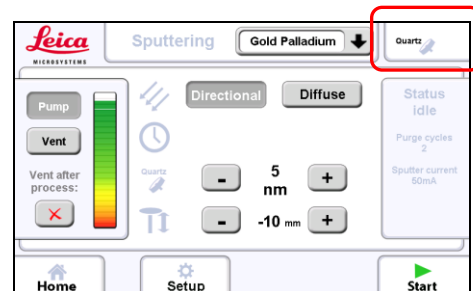
A quartz crystal oscillates at a certain frequency. Coated with a material, this frequency reduces according to the material and the applied thickness. With this information the accurate film thickness coated during a process run can be determined.

When the system is equipped with the optional QSG film thickness measurement, it is possible to terminate the process at the desired thickness. When terminating by time the resulting thickness can be shown.

Important to note is that the tooling factor to correct a height different to the zero position is set automatically by keying in the stage height. According to the selected metal (sputtering) or carbon thread (single or double) the parameters are automatically placed into the formula to calculate thickness.

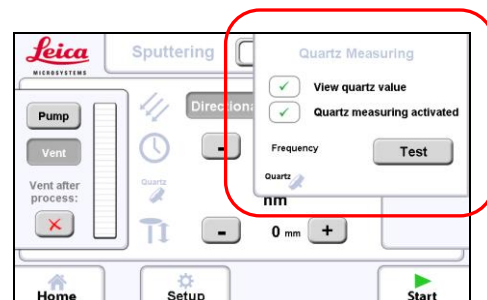
The QSG button is found on the top right of the main operating screen

(choose  or  on the main screen)



Tap on the  button to open the panel

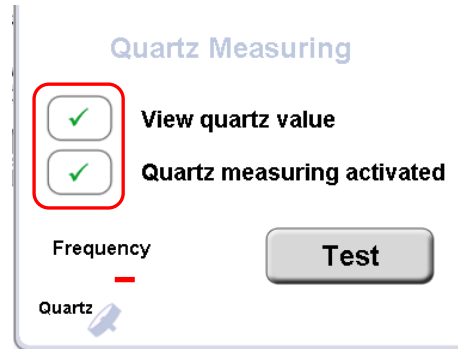
Tap on  to check the quartz function



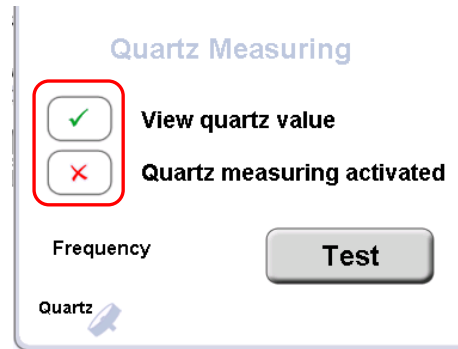
The frequency of a new crystal is  
~6 MHz and should be stable



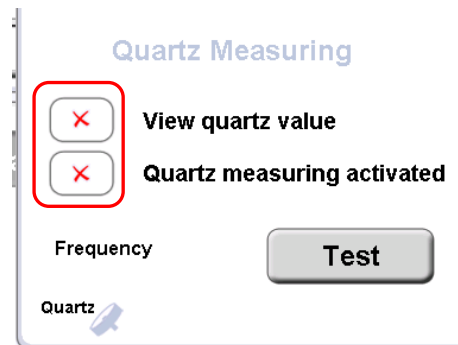
In this case the system will terminate after a set thickness.



In this case the system will terminate after a set number of pulses or flashes for carbon coating or time for metal sputtering and the achieved thickness will be measured and shown.

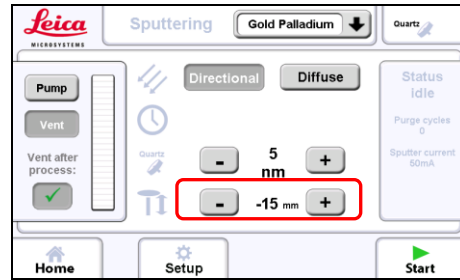


In this case the system will terminate after a set number of pulses or flashes for carbon coating or time for metal sputtering and the achieved thickness WILL NOT be measured.



**Automated tooling factor:** When the sample surface is not in line with the “0” position, this has to be defined to enable a correct thickness measurement ( 2.4.2)





When the sample surface is below the 0 reference mark the height correction number is set to the appropriate **negative** value.

### 3.8 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. If the optional quartz thickness measurement (QSG) is on during the process, the layer thickness can be calculated as a result of the change in quartz crystal resonance frequency (see 3.7).

After setting the coating parameters and pressing START the system will perform the following steps automatically.

- Pumping until base vacuum is reached
- Checking the availability of at least one carbon thread section, otherwise venting



Checking threads after reaching vacuum is necessary because the threads are heated slightly for measuring. When the vacuum is too low, there is a risk of oxidation.

- When reaching base vacuum ( $3 \times 10^{-2}$  mbar), outgassing (pre-heating), the first available thread for 45 seconds
- Opening shutter and starting rotation (if present and activated, see 3.11)
- Pulsing to the desired thickness or pulsing/flushing the requested number of pulses/sections. Each section required to fulfil the set protocol is outgassed separately before use.
- Closing the shutter
- Displaying the results of the process (thickness, number of pulses / flashes, availability of further threads)
- Venting or staying in vacuum

### 3.8.1 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

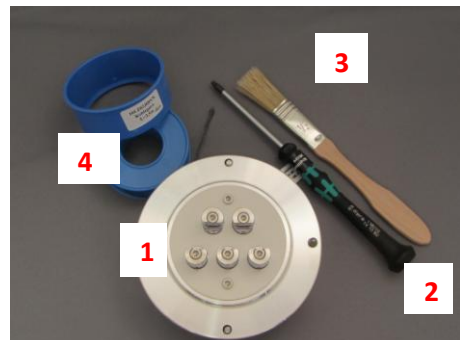
Make sure the instrument is vented (see 3.3)

1. Open the source cover
2. Unplug the connectors
3. Unscrew the 2 evaporation head screws to remove the flange

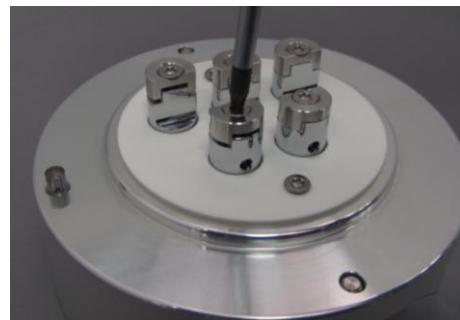


Prepare the following parts on a clean desk

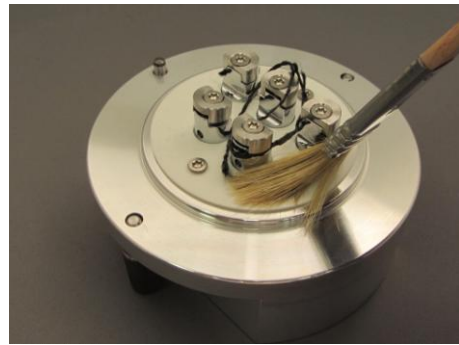
1. Carbon head
2. Torx TX 10 key
3. Brush
4. Carbon thread



Loosen all 5 clamp screws with the Torx key

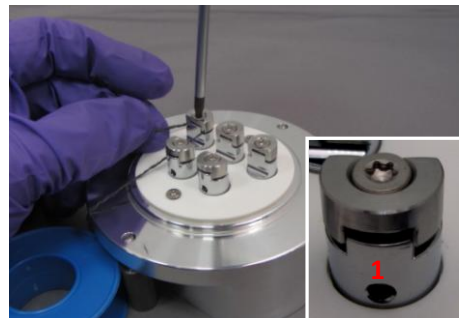


Remove any carbon thread residue using the brush supplied



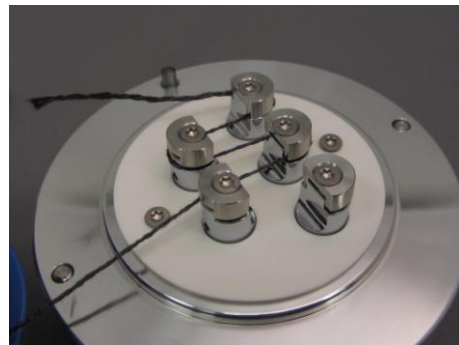
Loop the carbon thread around the first clamp and pull both ends gently to the left

At the same time tighten the screw as shown in the picture

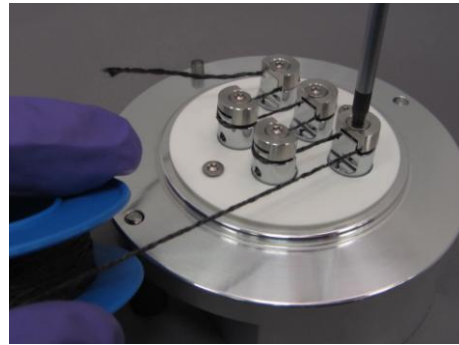


Wind the thread around the other clamps

Take care that the thread slides into the clamping groove (1) follow the path:



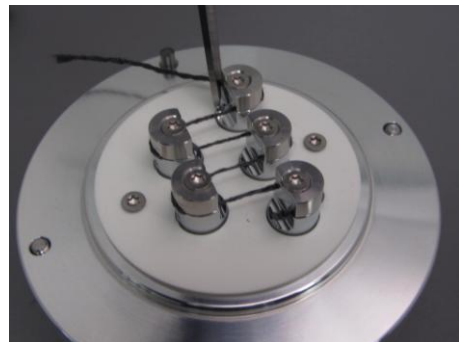
Pull the thread gently and tighten the last clamp



Tighten the remaining 3 screws and trim thread on both ends



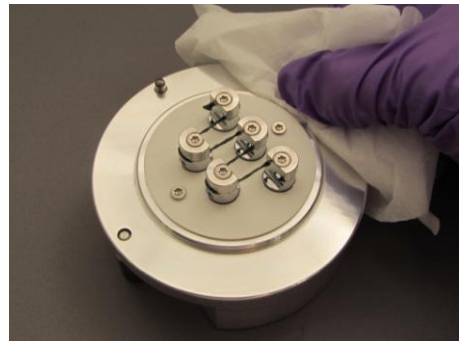
If all screws are not tightened the instrument may not recognize the thread section



Clean the sealing surface with a lint-free tissue



There is an ACE200 youtube video where the loading of the thread can be seen  
<http://www.youtube.com/watch?v=yKd1lyKIDT8>



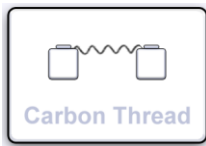
After the carbon thread is loaded, replace the head, gently tighten the fastening screws and connect the cables and close the cover



### 3.8.2 Setting up a carbon thread process

When the head is inserted after completing the preparation in 3.8.1, an evaporation process can be run.

Turn on the mains switch on the rear of the unit. Start screen will appear



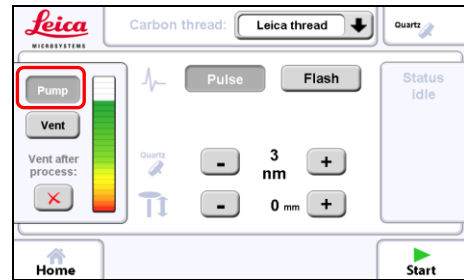
Press on



Press pump button to start pumping

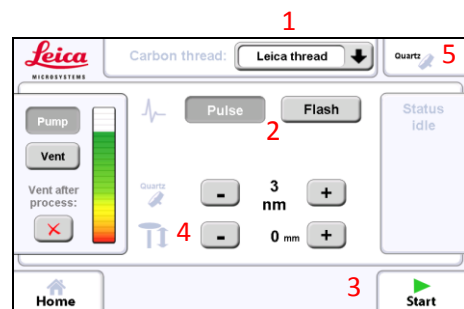


If the chamber door is not closed, an error message will indicate it (see 3.8.6)



While the system pumps down to  $3 \times 10^{-2}$  mbar all process parameters can be set:

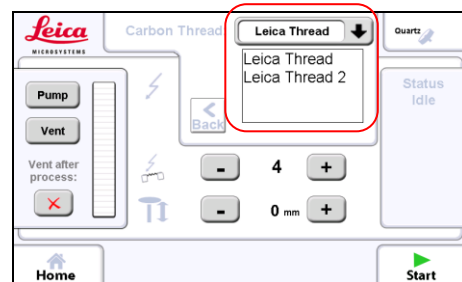
1. Target thread
2. Process pulse or flash
3. Process start or termination
4. Stage height from defined "0" position (only for QSG, see 2.5.2)
5. QSG if available (see 3.7)



### 3.8.3 Selecting the thread type

Push **Leica Thread** to open the drop down menu and select thread (thread 2 is a double thread)

The instrument automatically chooses the correct parameter settings



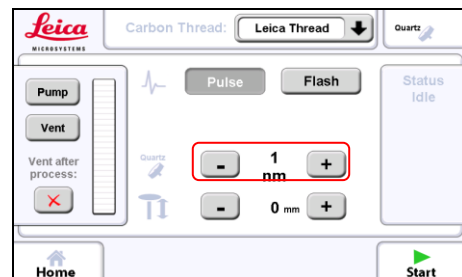
### 3.8.4 Pulse mode

When using the pulse mode the process can be terminated according to the desired coating thickness (quartz needs to be available) or a set number of pulses. For QSG setting please refer to 3.7.

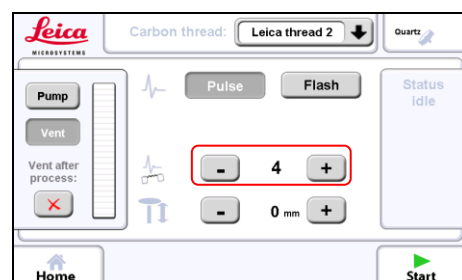
Select **Pulse**

When terminating the process using the quartz thickness monitor, the desired thickness is set with the +/- buttons

For details of the quartz setting please refer to 3.7



When terminating the process using the number of pulses, the desired number is set with the +/- buttons



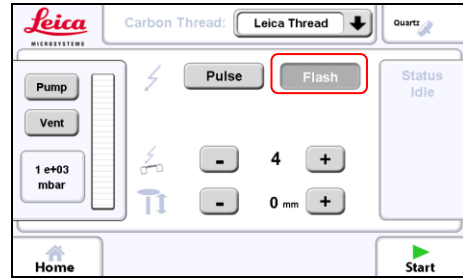
### 3.8.5 Flash mode

When using the flash mode the process is terminated after the selected number of carbon thread sections has been evaporated. If a QSG is available the resulting thickness can be shown. For QSG setting please refer to 3.7.

Select 


Using the flash mode, the process is defined by the number of flashes (1-4)

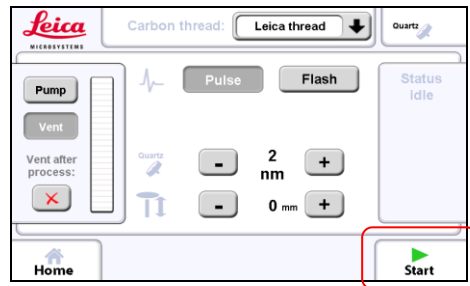
Power is run through the section of thread to burn it completely.




To minimize carbon fibre residues dropping onto the sample we recommend using the “Pulse” mode rather than the “Flash” mode.

### 3.8.6 Running a carbon thread process

After setting the parameters as described in 3.8.2 - 3.8.5 , the process can be run by pressing the  ...

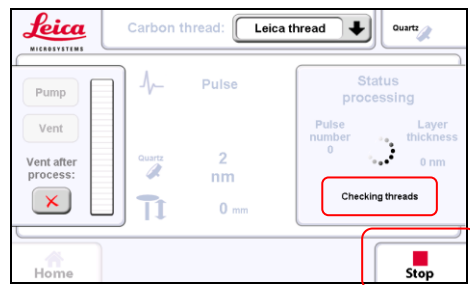


... which will turn into a  button



Detecting “Checking” the threads:

The system will first check if sufficient threads are available.

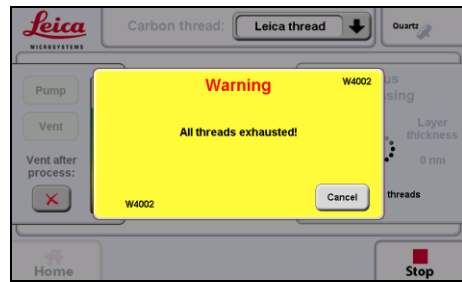




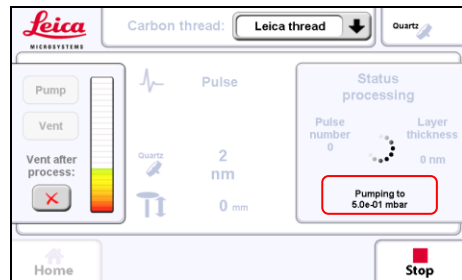
If not sufficient threads are available a warning will be shown.



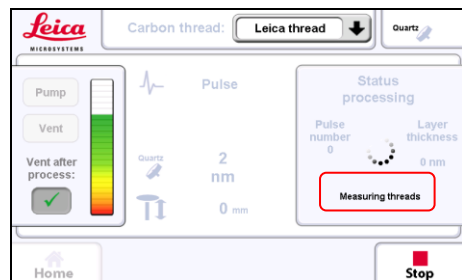
If no threads are available a warning is shown.



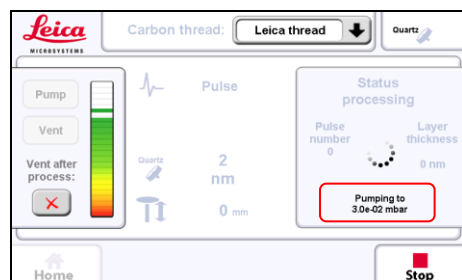
Pumping to  $5 \times 10^{-1}$  mbar



Measuring the threads



Pumping to base vacuum  $3 \times 10^{-2}$  mbar

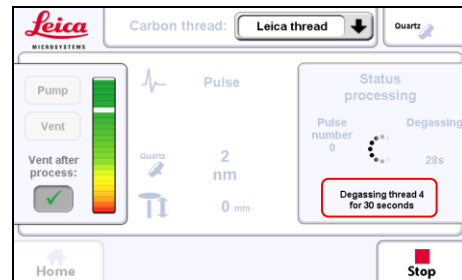




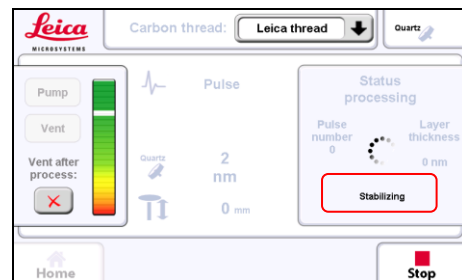
## Degassing the threads



Each thread is degassed separately before it is used.

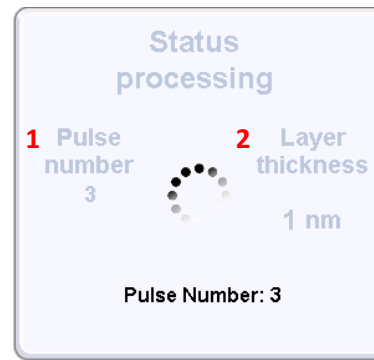


The system waits after outgassing to stabilize the vacuum



## Coating in progress

Number of pulses (1) performed and present thickness are displayed (2)



Coating is finished, the report summary indicates the number of pulses/flushes and (if quartz crystal measurement is available) the deposited thickness in nm



If starting a new coating without venting the total layer thickness accumulates, therefore the display shows the total thickness.



It is indicated how many threads are still available

### 3.9 Sputter coating

Sputter coating is performed using ionized argon to create a plasma. The argon ions are accelerated by high voltage and directed towards the source via a magnet where they

collide with the target and displace surface atoms. Due to this collision the surface atoms are directed towards the area below the target and coat the sample. This coating process can be set to directional or diffuse (more even coating on a bigger surface and better for fissured samples), depending on the process pressure. This also influences the coating rate (diffuse means slower rate) and the grain size (directional means finer grains). If the optional quartz thickness measurement (QSG) is present during the process the layer thickness can be calculated (3.7).

#### Targets

The following targets can be used for the ACE200

- Gold
- Gold-Palladium
- Silver
- Platinum
- Copper
- Nickel

#### Argon gas supply

The working gas (argon) must be supplied under a pressure of ~500 mbar (+/- 100mbar). The gas may be supplied via a fixed line or from a gas bottle. The gas should be at least 99.99 % pure (see also 2.2).

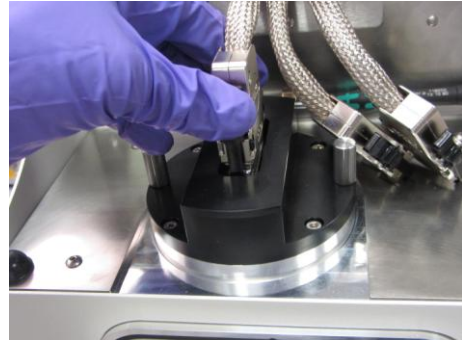
After setting the coating parameters and pressing START the system will perform the following steps automatically.

- Pumping until purge vacuum ( $5 \times 10^{-2}$  mbar) is reached
- When reaching the purge vacuum a number of purge cycles is executed
- Pumping until base vacuum is reached ( $5 \times 10^{-2}$  /  $3 \times 10^{-2}$  for diffuse/directional)
- Stabilizing the plasma
- Pre-sputtering, if target requires, (to clean the target from oxidation and enable a stable sputter rate)
- Starting the sputtering process by opening the shutter and starting rotation if present and activated
- Termination of sputtering by either time or thickness
- Closing the shutter
- Displaying the results of the process (thickness and/or time)
- Venting or staying under vacuum

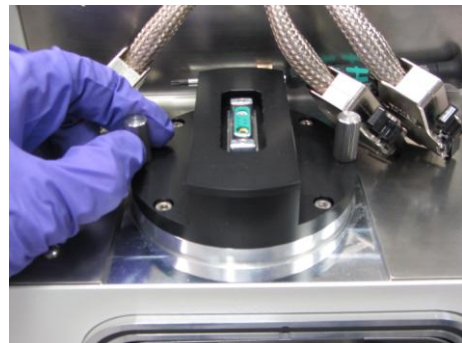
### 3.9.1 Loading the sputter target

Make sure the instrument is vented (see 3.3)  
and open the source cover

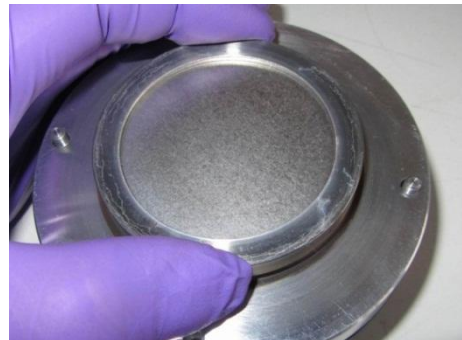
Unplug the connectors



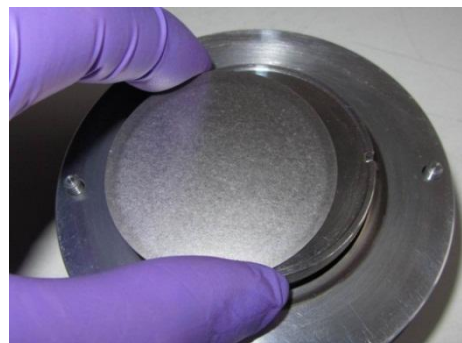
Unscrew the 2 sputter head screws to  
remove the flange



Remove the bayonet ring by turning and  
insert the sputter target



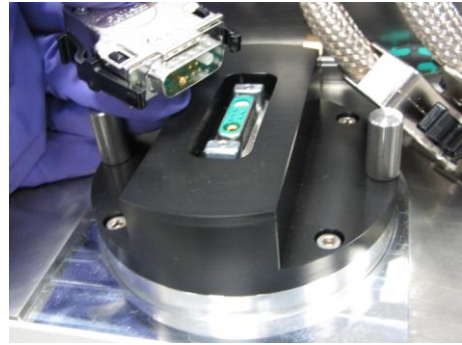
Fix target by gently tightening the bayonet  
ring.





Tighten the ring by hand only.

When the target is secured replace the sputter head, gently tighten the fastening screws and connect the cable and close the cover



There is an ACE200 youtube video which also shows the loading of the target

<http://www.youtube.com/watch?v=Kd1IyKIDT8>

### 3.9.2 Setting up a sputtering process

When the head is inserted after completing the preparation in 3.9.1 a sputtering process can be run.

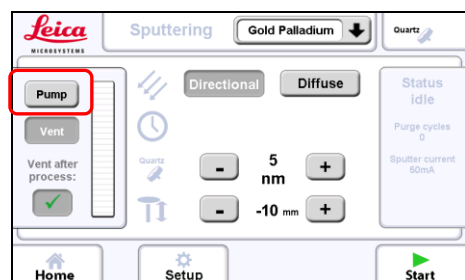
Turn on the mains switch at the back of the unit Start screen will appear



Press on



Press pump button to start pumping



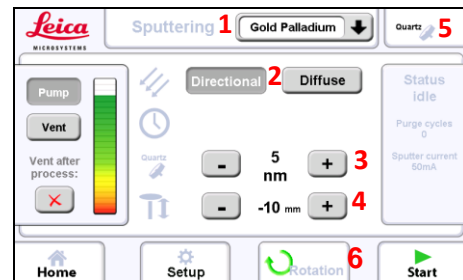


If the chamber door is not closed, an error message will be shown.




While the system pumps down to  $3 \times 10^{-2}$  mbar all process parameters can be set:

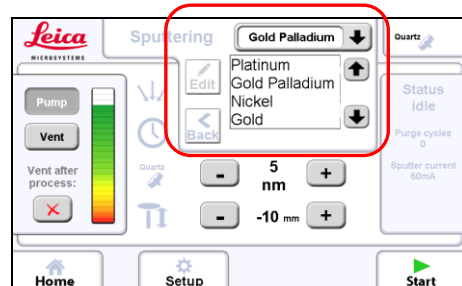
1. Target material
2. Process: directional or diffuse
3. Process termination
4. Stage height from defined "0" position (only for QSG, see 2.6.2)
5. QSG if available (see 3.7)
6. Rotation if available (see 3.11)



### 3.9.3 Selecting the material

Push  (last chosen material is pre-selected) to open the drop down menu and select the target material

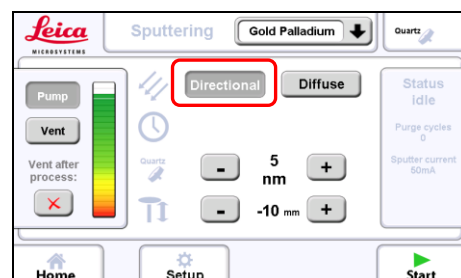
The instrument automatically chooses the right parameter settings



### 3.9.4 Directional mode

The directional mode is intended for relatively flat samples. The vacuum for directional sputtering is  $4 \times 10^{-2}$  mbar.

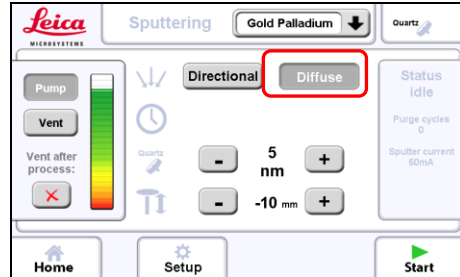
Select directional



### 3.9.5 Diffuse mode


The diffuse mode is intended for more topographic samples. The vacuum for diffuse sputtering is  $8 \times 10^{-2}$  mbar.

Select diffuse



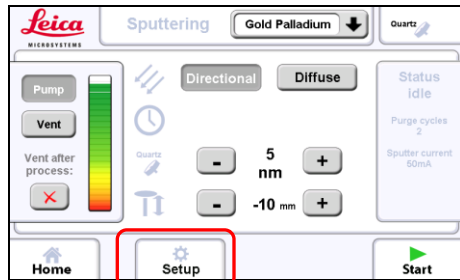
### 3.9.6 Sputter current and purge cycles

Different targets require different sputter currents. A recommended value is pre-set when the target is chosen (see 3.9.3). The vacuum chamber can be purged with argon.

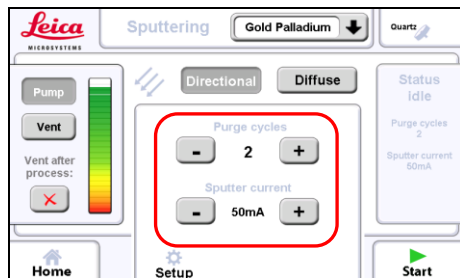
Press  to open the panel to adjust the number of purge cycles with argon and to set the sputter current



The last settings are stored.



Adjust the parameters using the +/- buttons



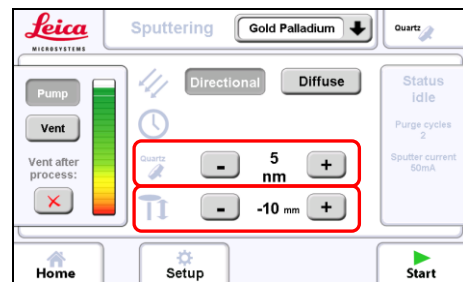
### 3.9.7 Sputtering by time or thickness measurement

The sputtering process is terminated either by the desired thickness or time.

Quartz measurement is activated

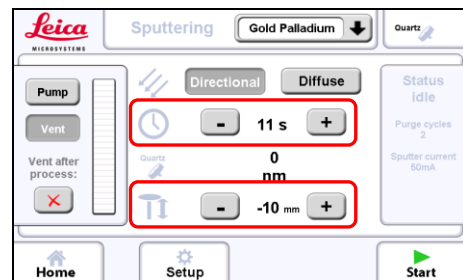
Height adjustment for the correct quartz tooling factor

For quartz details refer to 3.7

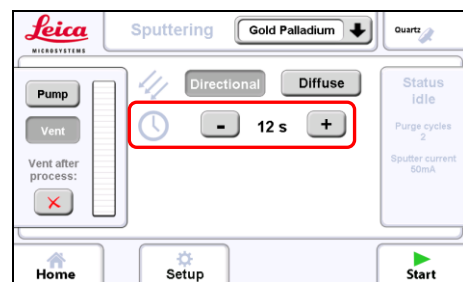


Termination by time

Quartz measures the achieved thickness



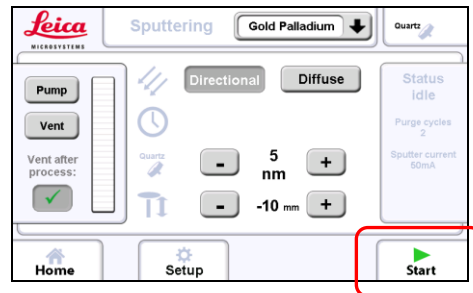
No quartz available, termination by time



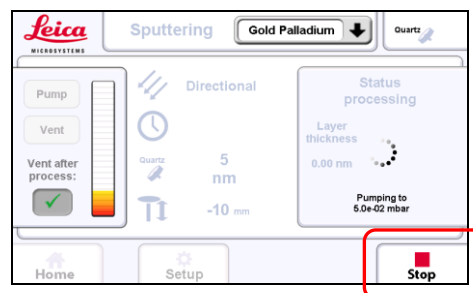
### 3.9.8 Running the sputtering process

After setting the parameters as described in 3.9.2 - 3.9.5 , the process can be run by

pressing the  ...



... which will turn into a  button



If the door is not closed an error message will be shown.



If the sputter head is not connected or the carbon thread head is connected an error message will be shown.



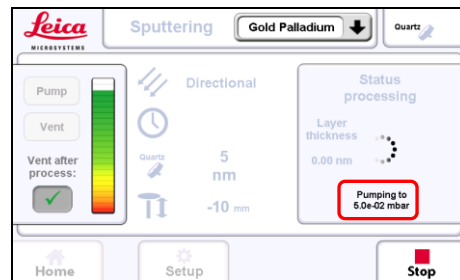




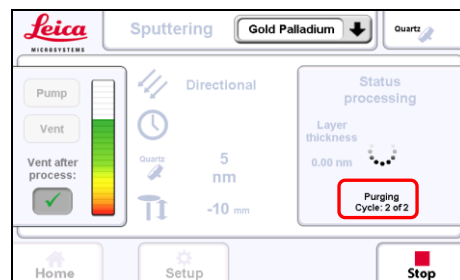
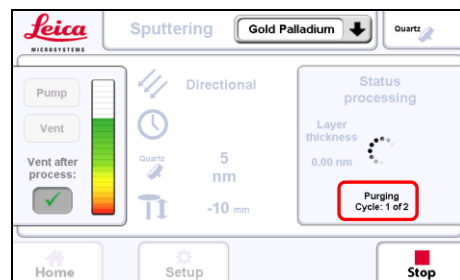
If the sputter head cover is not closed the an error message will be shown.



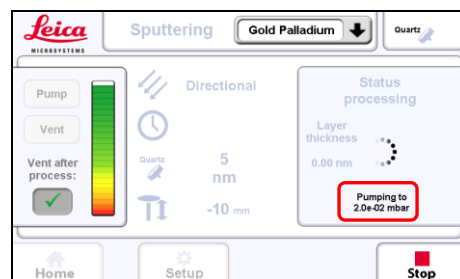
Preparing vacuum level to purge with argon ( $5 \times 10^{-2}$  mbar)



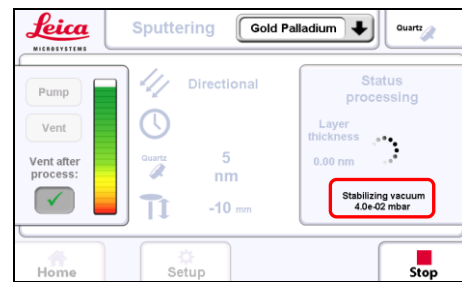
Purging the chamber with argon (number of cycle is displayed)



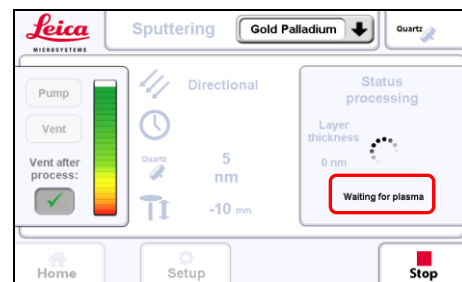
Pumping down to base vacuum ( $2 \times 10^{-2}$  mbar)



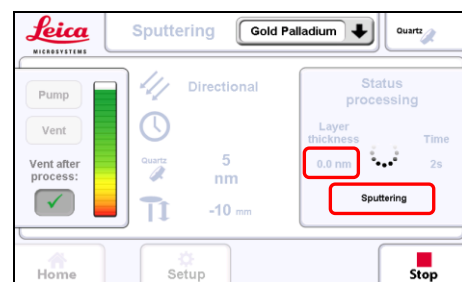
Stabilizing argon flow to reach the process vacuum (in this example  $4 \times 10^{-2}$  mbar for directional mode)



Starting pre-sputtering to clean the target and stabilize parameters



Opens the shutter and start sputter coating  
Current coated thickness



Hint, in case process vacuum cannot stabilize check if the argon valve on the bottle is open



Coating is finished, the report summary shows the coating time and (if quartz crystal measurement is available) the achieved layer thickness

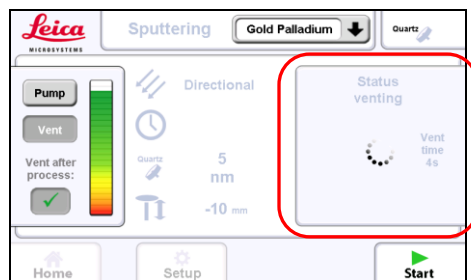


If starting a new coating without venting the total layer thickness accumulates, therefore the display shows the total thickness.

Replace image - this is for carbon



Venting in progress



### 3.10 Glow discharge

The glow discharge is an optional process which is used mainly to make TEM grids hydrophilic. Air is used to create plasma. This process can also be used to clean the sample surface before coating.

After setting the glow discharge parameters the system will perform the following steps automatically.

- Pumping until base vacuum (just under  $3 \times 10^{-1}$  mbar) is reached
- Bleeding in air to adjust vacuum to  $3 \times 10^{-1}$  mbar
- Stabilizing vacuum and starting glow discharge with closed shutter (purple plasma is visible)
- Termination of glow discharge process by the set time
- Displaying the results of the process (current and time)
- Venting or staying in vacuum

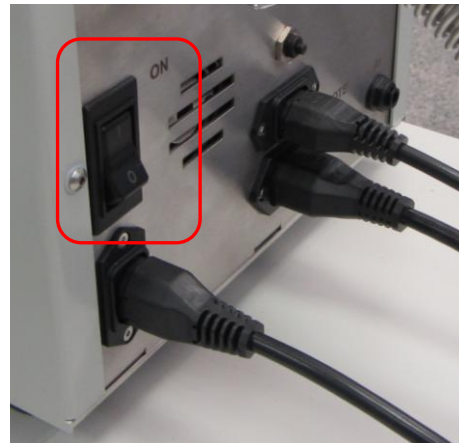
Turn on the mains switch at the back of the unit  
Start screen will appear



Press on



To use the glow discharge option, ensure that the glow discharge electrode is mounted.

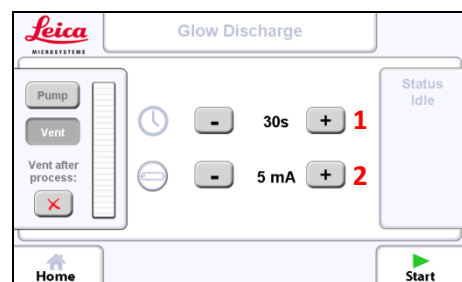


Set desired

1. time and
2. current

by pushing the +/- buttons



The pre-set values are shown

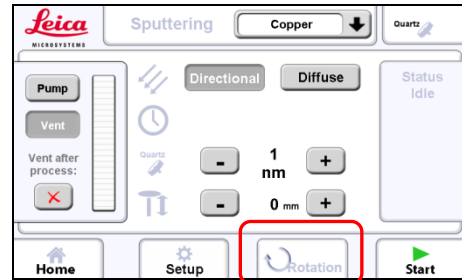


### 3.11 Planetary drive stage

The optional planetary drive stage is useful for coating highly topographic or fissured samples. After installing the planetary drive stage as described in section 2.5.1 the rotation function can be used.

The Rotation button is found on the bottom row of the main operating screen

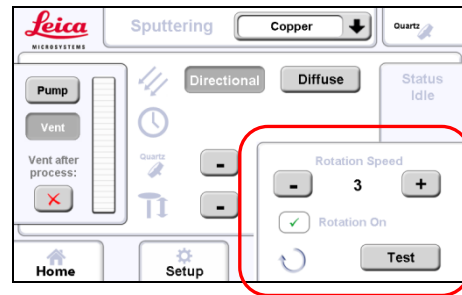
(choose  or  on the main screen first)




Tap on the  button to open the panel

Activate  Rotation On by pressing

Press "Test" to check rotation, press it again to stop



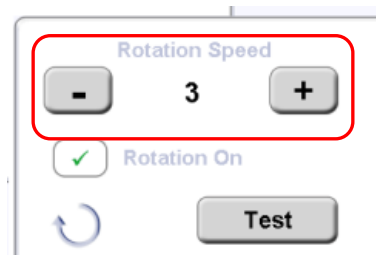
Set speed by pressing +/-

Press the  symbol to close the window

Stage will rotate automatically when the coating process runs



The last rotation speed is stored even if the system is switched off.



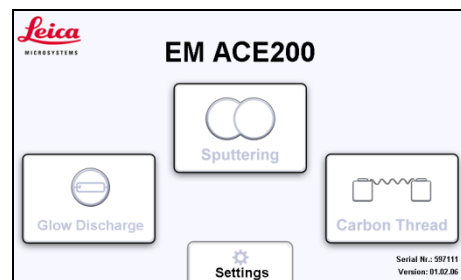
### 3.12 Switching between processes

A coater equipped with several options such as sputter coating, carbon thread evaporation or glow discharge can be easily switched from one process to the other. This is done by exchanging the source and choosing the desired process from the main screen.

The system detects automatically which options are available. When choosing a process the instrument detects if the correct head is connected. The screen below indicates a system equipped with carbon thread, sputtering and glow discharge.

To switch from sputtering to carbon thread (or vice versa) insert and connect the appropriate head and select the respective process

For glow discharge it does not matter which head is connected simply choose the glow discharge process



Error message after pressing the "Start" button if a carbon thread head is installed instead of the sputter head



Error message after pressing the "Start" button if a sputter head is installed instead of the carbon thread head



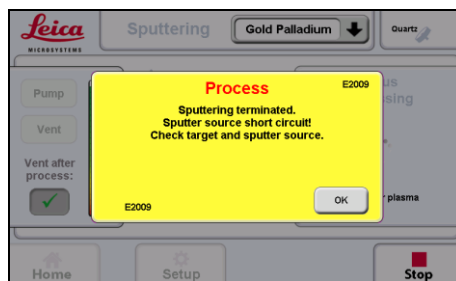
## 4. Maintenance and service

The purpose of these activities is to

- Maintain the optimal operating conditions of the Leica EM ACE200
- Minimize downtime
- Provide a standard maintenance schedule
- Deal with malfunctions

Malfunctions during operation of the Leica EM ACE200 coating system are reported in an information window. A clear text error message provides information about the cause of the malfunction and the action required.

Example:



For further questions regarding malfunctions refer to the error list under chapter 5.



Caution!

Injuries or damage to the system may be caused by servicing and cleaning the system incorrectly.

### 4.1 General instructions for maintenance and cleaning

External elements (dust, grease, etc.) may prevent the production of the required vacuum.



When working on the vacuum chamber or parts which are in the vacuum chamber of the Leica EM ACE200 coating system it is essential to follow the principles of vacuum hygiene. Gloves must be worn when disassembling and assembling components in the vacuum area, and also for all adjustment work.

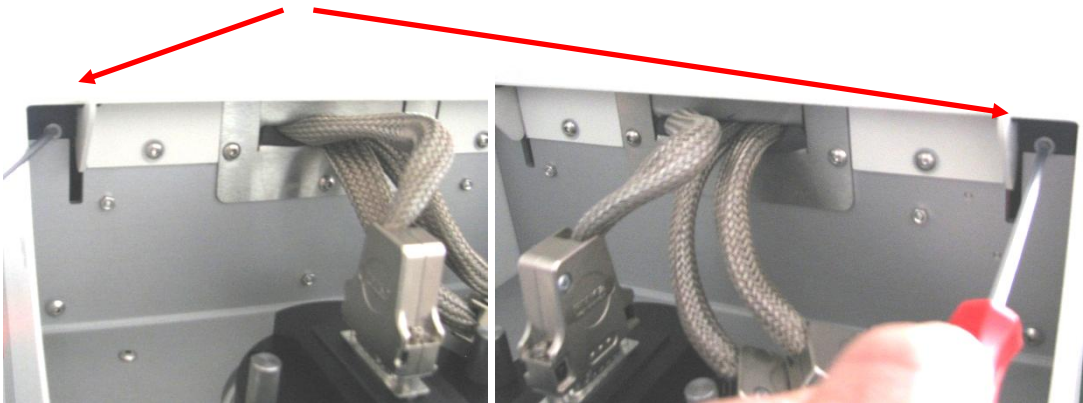
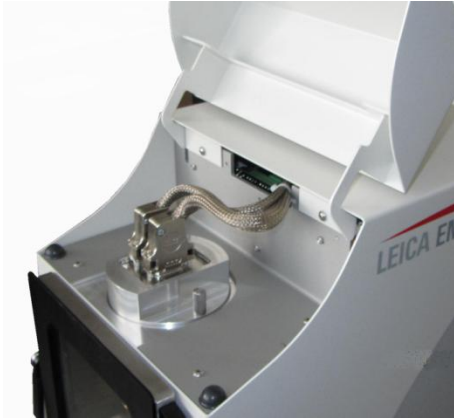
All work must be carried out in a clean, oil/grease-free and dust-free environment.

#### 4.1.1 Vacuum pump

The oil of the vacuum pump normally requires replacement once a year. Please refer to the operation manual of the pump manufacturer.

#### 4.1.2 Source cover

If the source cover closes too quickly, it is possible to tighten the hinges using a 3mm allen key. Be careful not to over tighten.



#### 4.2 Cleaning of the ACE200

The internal shielding and shutter including the glow discharge electrode should be cleaned or replaced when:

- Peeling or thick coating is visible
- If contamination is observed during EM imaging
- Pumping time to reach base vacuum increases significantly

Extensive cleaning and parameter check is recommended once a year by a trained Leica service engineer to ensure flawless operation.



## Recommendation

Usually, only the glass door needs to be cleaned, depending on the layer thickness and your visibility needs. The chamber shielding, especially when always using the same material, does not need to be cleaned very often.

## Cleaning material

Any abrasive works well such as scotch-brite or metal cleaning paste. Washing the parts afterwards with soapy water and letting them dry off is an easy way to finish the cleaning. Also, Isopropanol or Ethanol can be used for the finish.



Caution!

Do not use thin tissues and ethanol for removing platinum – there is a risk of the tissue catching fire.

## Door

- Remove the door by lifting it from its hinges (see 4.2.1)
- Place the door on a flat soft surface (e.g. paper towel) with the inner surface up
- To remove carbon coatings use a 70% isopropanol solution and lint-free tissue for the rough cleaning. Then use fine metal polish paste. Finish with soap-water or isopropanol
- To remove sputter coatings start with the fine polish paste and finish with soap-water or isopropanol

## Internal shielding, shutter and glow discharge electrode

How to remove the chamber parts see 4.2.1

- The internal shielding can be cleaned using Scotch-Brite or metal cleaning paste. Finish with soap water and then clear water or isopropanol
- When cleaning the shutter it is recommended to lubricate the fastening screw hinge with a drop of high vacuum Fomblin (high vacuum lubricant) before assembly.



- The glow discharge electrode is available as a spare part. One extra spare is supplied with the system

### Housing

- All surfaces can be cleaned with a damp, lint-free cloth moistened with either aqueous cleaning agents or 50% ethanol (ethanol - water 1:1)



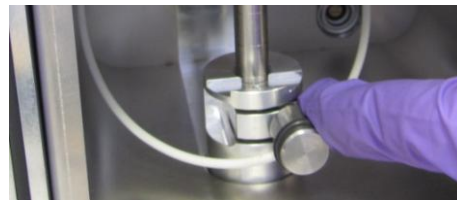
**Do NOT use ACETONE!**

- The LCD control panel should be cleaned with standard commercial screen cleaner when the system is switched off
- Accidental spillage of solvents (e.g. acetone, isopropanol or ethanol) must be removed immediately with a damp, lint-free cloth moistened with aqueous cleaning agents

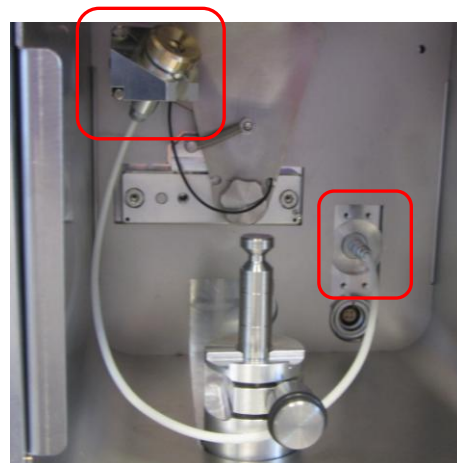
#### 4.2.1 Removing shutter and internal shielding

To remove the shutter and shielding the stage has to be taken out first (see 2.5.).

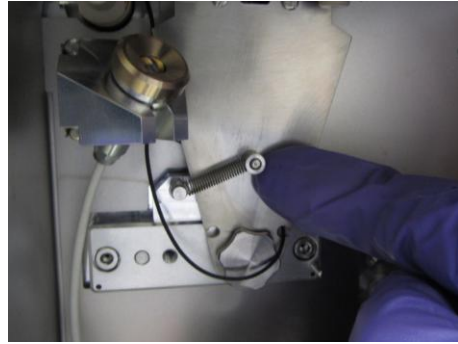
Remove the stage rod by releasing the thumb screw



If present, disconnect the QSG plug and remove the QSG (see 2.5)



Remove the shutter retaining spring



If the instrument is configured with glow discharge, disconnect the push-in connector in the upper left side of the chamber before removing the shutter



Unscrew the shutter thumb screw and take out the shutter assembly

Caution!



There is danger from the edges of the metal sheet of the internal shielding and shutter.

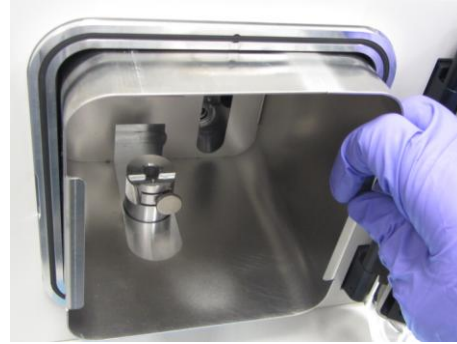
Personal injury (e.g. cuts) may occur.



Make sure that the arm to move the shutter is aligned with the opening of the chamber shielding



Remove the internal shielding by holding it on the side with the small handles and pulling it out



To install shielding and shutter, reverse the above procedure.

#### 4.2.2 Removing the door

To remove the door lift up the door and take it out



Place it flat on a soft surface to clean



The complete glass can be exchanged by unscrewing both screws on the side to hold the glass

Replace the glass and tighten the screws again



# 5. Troubleshooting

## 5.1 Error messages – User self-assistance

Error text	SOURCE x SHORT-CIRCUIT
Possible cause	Short circuit in head caused by extensive coating
Remedy	<p>For the sputter head:</p> <p>Take off the head from the system and remove the target</p> <p>Clean the ring that holds the target in place</p> <p>Clean the head surface from deposition layer</p> <p>Clean the area in the chamber that is around the sputter head</p> <p>For the carbon thread head:</p> <p>Take off the head from the system and remove the carbon thread</p> <p>Remove the ceramic plate which has 5 holes for the carbon fasteners by unscrewing the 2 holder screws</p> <p>Clean the head from all depositions</p> <p>Please take care to clean the groves around the 5 high current pins (caution – O-rings below)</p> <p>Put back the ceramic plate and secure it by the 2 holder screws (do not tighten them, leave some clearance)</p> <p>Clean the area in the chamber around the carbon thread head</p>
Error	NO ARGON PRE-PRESSURE.
Possible cause	There is no argon pre-pressure
Remedy	Check the argon connection and the “OUT” argon pressure to the coater

## 5.2 Malfunction

Malfunction	System does not start when switched on
Cause	The cable is not connected to the mains power supply
Remedy	Connect to the mains power supply
Malfunction	Pump does not start when pressing “Pump” or “Start”
Cause 1	Fuse on the rear of the coater popped out
Remedy	Press “Stop” process and then push in the fuse
Cause 2	No power to the pump
Remedy	Check connections
Cause 3	The switch on the pump isn’t in the “On” position
Remedy	Switch on the pump
Malfunction	Touchscreen does not appear
Cause	No power supply
Remedy	Check connections
Malfunction	Sputter plasma does not start
Cause	The ring holding the target dropped and caused a short circuit
Remedy	Remove the head and replace the target and fastener ring
Cause	Deposited material is peeling off the shutter causing a short circuit
Remedy	Remove the shutter and clean it
Malfunction	Shutter doesn’t move
Cause	The shutter was installed with the actuator arm clamped behind
Remedy	Switch off the system. Loosen the shutter thumb screw and move the shutter fully to the right. Move the shutter actuator arm to the left ~11 O’clock. Re-tighten the shutter thumb screw

## 5.3 Error messages complete list – description for Leica service

### 5.3.1 Carbon thread

Text on the screen	Warning or Error number with description
<ul style="list-style-type: none"> <li>- W4000 Warning Threads available CANCEL</li> </ul>	<p>"W4001"</p> <p>No threads available, cancel only after measurement of resistor.</p>
<ul style="list-style-type: none"> <li>- W4002 Warning Threads exhausted CANCEL</li> </ul>	<p>"W4002" ...no threads left for the process even though needed, only quitting process possible.</p>
<ul style="list-style-type: none"> <li>- W4003 Warning x Threads available flashes not possible CANCEL</li> </ul>	<p>"W4003" ... Number of threads to complete the flashing process is too low.</p>
<ul style="list-style-type: none"> <li>- E4009 Process Process terminated Head over temperature OK</li> </ul>	<p>"E4009" ... Process abort, head has over temperature (above 65°C).</p>
<ul style="list-style-type: none"> <li>- E4011 Process Process terminated. Mains supply voltage fault Check door and cover OK</li> </ul>	<p>"E4011" ... CT- Carbon thread power supply is not switched on, possible door or cover are open.</p>
<ul style="list-style-type: none"> <li>- E4012 Process Process terminated Mains supply voltage fault Check door and cover OK</li> </ul>	<p>"E4012" ... CT- Main Power relay is not switched on possible door or cover are open.</p>
<ul style="list-style-type: none"> <li>- E4015 Process Carbon thread head is not connected Connect cable OK</li> </ul>	<p>"E4015" ... CT- head is not connected, system recognises via the temperature sensor.</p>

### 5.3.2 Sputtering

Text on the screen	Warning or Error number with description
<ul style="list-style-type: none"> <li>- E2001 Process Sputtering terminated Mains supply voltage fault. Check door and cover OK</li> </ul>	<p>"E2001" ....HV-Board is not supplied with voltage door or cover sensor could be the reason.</p>
<ul style="list-style-type: none"> <li>- E2002 Process Sputtering terminated Mains supply voltage out of range! &lt; 85V OK</li> </ul>	<p>"E2002" .... Process terminated. Mains supply voltage out of range.  Supply voltage out of range: &lt; 85 V.</p>
<ul style="list-style-type: none"> <li>-</li> </ul>	
<ul style="list-style-type: none"> <li>- E2003 Process Sputtering breakup Mains supply voltage out of range 150&lt;&gt;210V OK</li> </ul>	<ul style="list-style-type: none"> <li>- "E2003" .... Process terminated. Mains supply voltage out of range</li> <li>- Supply voltage out of range: 150..210V.</li> </ul>
<ul style="list-style-type: none"> <li>- E2004 Process Sputtering terminated Mains supply voltage out of range! 250V OK</li> </ul>	<ul style="list-style-type: none"> <li>- "E2004" .... Process terminated. Mains supply voltage out of range.</li> <li>- Supply voltage out of range: &gt; 250 V.</li> </ul>
<ul style="list-style-type: none"> <li>- E2005 Process Sputtering terminated Current not reached Check target and sputter source OK</li> </ul>	<ul style="list-style-type: none"> <li>- "E2005" .... Process terminated.</li> <li>- Sputtering terminated. Head Problem, sputter current could not be reached, reason could be wrong pressure or wrong or no present target.</li> </ul>
<ul style="list-style-type: none"> <li>- E2007 Process Sputtering terminated Line voltage fluctuations OK</li> </ul>	<ul style="list-style-type: none"> <li>- "E2007" .... Sputtering terminated. Fluctuation of main voltage is too big.</li> </ul>



- E2009  
Process  
Sputtering terminated  
Sputter source short circuit  
Check target and sputter source  
OK
- E2010  
Process  
Ignition fault  
Check argon flow  
Check target and sputter source  
OK
- E2011  
Process  
Sputtering terminated  
Sputter current could not be stabilised OK
- E2012  
Process  
High voltage fault  
High voltage board defect  
Please contact service  
OK
- E2015  
Process  
Sputter head is not connected  
Connect cable  
OK
- E2016  
Process  
Sputtering interrupted - head over temperature! Layer thickness: xxnm  
OK
- E2017  
Process  
Sputtering terminated. – Timeout  
No thickness increase  
Check quartz  
Check shutter mechanics  
OK
- "E2009" .... Sputtering terminated. Short circuit in sputter head, ring or target is not placed correctly or material deposition on the shutter.
- "E2010" ....Sputtering terminated plasma was not ignited, possible reason is argon flow or no target present.
- "E2011" ....Sputtering terminated. Current cannot be controlled to a stable value, plasma is not stable. Check argon flow and clean the target
- "E2012" ....Process terminated. High voltage board is defect. Output cannot be controlled, board may needs to be exchanged.
- "E2015" ....Sputter head is not connected. Detection via the temperature sensor in head.
- "E2016" .... Sputtering terminated. Head overtemperature temperature is over 65°C reason is long sputtering with high current, head could be damaged if temperature gets too high.
- "E2017" .... Sputtering terminated. Timeout - QSG measures no layer deposition. Possible reason, shutter didn't open

### 5.3.3 Low Vac Mainboard Controller

- Text on the screen	- Warning or Error number with description
<ul style="list-style-type: none"> <li>- E1001 Shutter motor Motor is not connected. Please contact service OK</li> <li>- on the Service Screen</li> </ul>	<ul style="list-style-type: none"> <li>- "E1001".... Shutter motor is not connected to board.</li> <li>-</li> </ul>
<ul style="list-style-type: none"> <li>- E1001 Shutter Motor Motor is not connected Please check cable X20 at board 661531106 OK</li> </ul>	
<ul style="list-style-type: none"> <li>- E1003 Shutter motor Endswitch not found Check for obstacles check shutter mechanics OK</li> <li>- on the Service Screen</li> </ul>	<ul style="list-style-type: none"> <li>- "E1003".... When initializing shutter motor end switch was not found. Possible reason is stuck motor movement or sensor is defect. Check on service screen.</li> </ul>
<ul style="list-style-type: none"> <li>- E1003 Shutter motor Endswitch not found Please check cable X9 at board 661531106 OK</li> </ul>	
<ul style="list-style-type: none"> <li>- E1004 Shutter motor Motion not possible Check for obstacles check shutter mechanics OK</li> </ul>	<ul style="list-style-type: none"> <li>- "E1004".... Shutter motor movement is not possible, maybe motor is stuck.</li> <li>-</li> </ul>

## 6. Servicing and repair

The Leica EM ACE200 is covered by a WARRANTY according to the conditions of sale. If functional errors should occur or if the components of the system sustain damage that is subject to warranty coverage during the warranty period, the manufacturer will repair or replace the faulty components following examination thereof.

The manufacturer's warranty covers the system in its original configuration.

Only original replacement parts may be used. The manufacturer accepts no liability for damage caused by use of other replacement parts.

The manufacturer will not accept liability for damage caused by misuse of the system or its use for purposes other than the intended use, nor for damage caused by work on the system that is not described in this manual.

# **7. Attachments**

**7.1 Technical data sheet**

**7.2 EC declaration**