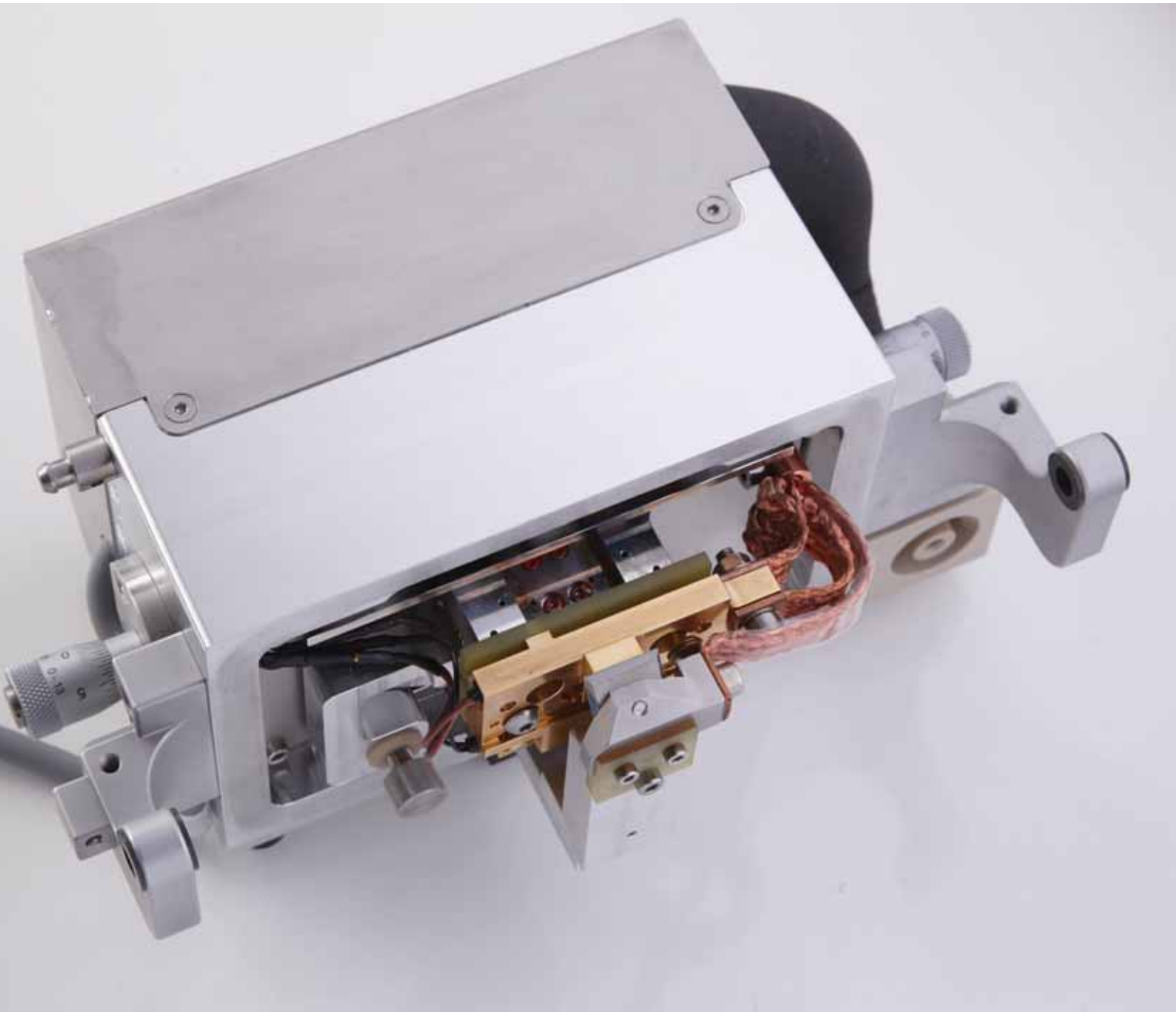
Leica EM TIC 3X Cooling Stage

Operating Manual

Version 10/2016



Important Note

Leica Mikrosysteme GmbH 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.

Issued by:

Leica Mikrosysteme GmbH
Hernalser Hauptstrasse 219
A-1170 Vienna

Leica EM TIC 3X Cooling Stage

Operating Manual

Leica EM TIC 3X Serial Number:

Date of purchase:

For the instrument serial number, please refer to the name type label on the back of the instrument!

Please read this instruction manual carefully before operating the instrument.
For Research use only!

Foreword

This user manual is intended to provide essential information regarding the correct operation and maintenance of the **Leica EM TIC 3X Coolilng Stage**. The appendix helps with specific processing of solid samples for subsequent investigation with an electron microscope (EM) or light microscope (LM).

For correct use of the Leica EM TIC 3X system please refer to the instrument's operating manual.

This user manual describes commissioning of the **Leica EM TIC 3X Cooling Stage**, phased testing and adjustment of all components and movement sequences.

Service and operating staff must familiarize themselves with all components of the system before commissioning. Particular attention must be paid to the aspect of safety.

This user manual must be retained for future reference.

Texts, schedules and tables may not be copied, reproduced, or divulged to third parties without our express consent.

It should be noted that this user manual does not constitute a part of any existing, prior agreement or covenant or legal relationship.

All obligations are derived from the purchase agreement, which is also the sole document of record regarding the terms of the warranty. Contractual provisions are not affected by the user manual.

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

Table of contents

1. Introduction

In order to ensure the safety of service technicians and operators, and to prevent any damage to the Leica EM TIC 3X Cooling Stage, it is essential to read this manual carefully before beginning any work with the system.

This user manual is intended to help the user to understand the system more completely, to use it within the specified limits of its working capabilities, and to maintain and service it in accordance with its physical parameters.

This user manual includes important information regarding the correct installation, operation and maintaining. Following these instructions will help to prevent hazards, reduce repair and downtime costs, and prolong the system's service life.

In certain chapters, a symbol refers to the function or respective section of text and is relevant for the operation or maintenance of the system, or indicates important descriptions or additional remarks:

Symbols in this manual and their meaning:

Danger!
All paragraphs in the Technical Documentation that contain instructions regarding possible hazards are identified with this symbol. Failure to observe 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.

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 work gloves!

Symbols and indications on the instrument and their meaning:

Danger of pinching the fingers when closing the flange (stage).

Hot surface, during and immediately after processing the sample. Allow to cool before servicing the ion source.

LN_2 is used to cool down the sample. To avoid serious burns or frostbite, follow safety information before disconnecting fluid lines.

Lifting hazard. The volume of the LN_2 dewar is 25l. Single person lift could cause injury. Use assistance when moving or lifting the dewar.

This dewar contains LN_2, follow safety procedures when handling with liquid nitrogen.

Warning! Improper use of the instrument can cause serious harm. Read the manual before operating the system.

1.1 Identification

1.1.1 Product

Leica EM TIC 3X Cooling Stage

1.1.2 Name and address of the manufacturer

Leica Mikrosysteme GmbH
Hernalser Hauptstrasse 219
A-1170 Vienna

Tel.: +43 1 488 99-0
Fax: +43 1 488 99-350

Internet: http:// www.leica-microsystems.com

2. Product description

2.1 Field of application and proper use

The **Leica EM TIC 3X Cooling Stage** is used for precise processing of samples for subsequent examination with an electron microscope (EM) or light microscope (LM). For this purpose, a cross-section of a sample is created with the Leica EM TIC 3X Triple Ion Beam Cutter system.

The samples are processed with ion beams in a vacuum. The system generates ions in the energy range from 1 keV to 10 keV.

Almost any material can be processed, e.g. semiconductors, metals, rocks, ceramics, polymers.

The ion energy causes the temperature of the sample to increase during processing. The operator must ensure that this does not cause the sample's state of aggregation to change. If so, enable cooling in the menu in order to cool down sample holder and mask up to -160 °C.

The **Leica EM TIC 3X** must not be used beyond the limits specified in its technical data sheet.

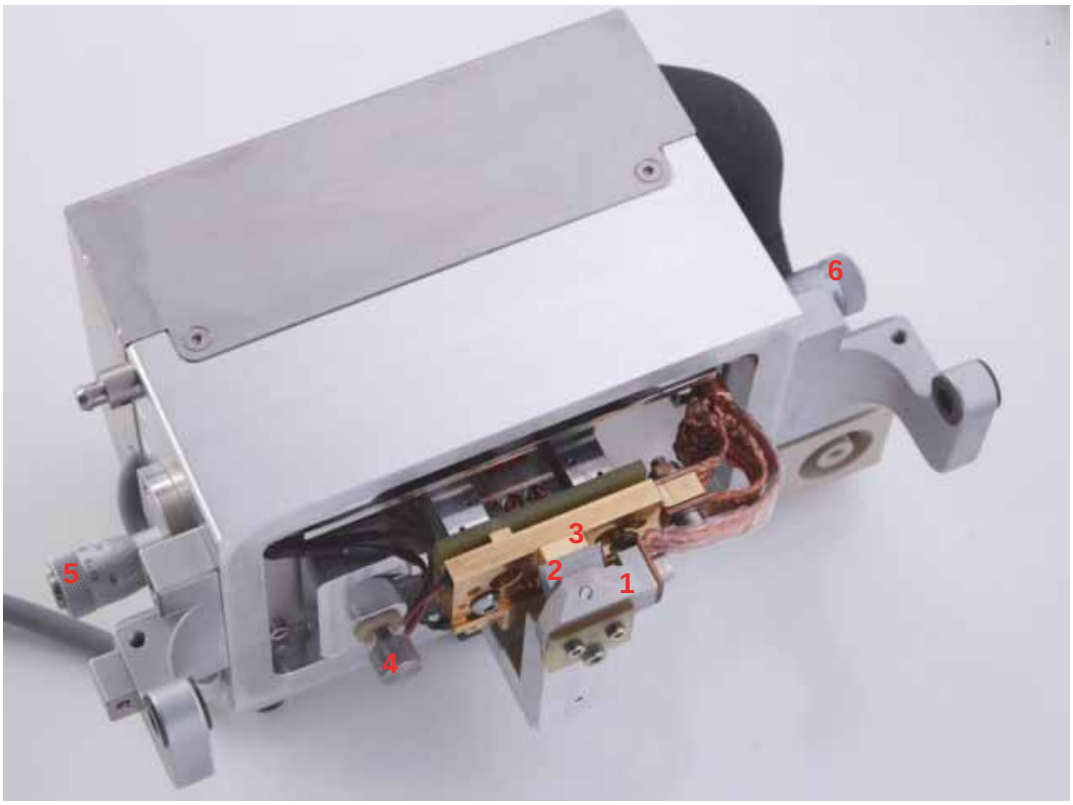
An inert gas, preferably argon, is used as the working gas. If other gases are used for reactive processing of samples, the appropriate safety measures (purging, removal of exhaust air) must be put in place, not only for the working gas, but also for the reaction by products created during processing. To make sure the system will be suitable for other gases than argon please contact the Leica representative prior to use.

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 cut with an ion beam. When hazardous substances have been processed the system (chamber) must be decontaminated before servicing the instrument.

Applications other than those described are inconsistent with proper use and are therefore prohibited. If the system is used incorrectly, all claims under warranty will not be accepted.

In cases of doubt, please consult your local Leica representative.

2.2 Instrument overview



1. Mask holder
2. Mask
3. Sample holder (different types available)
4. Set screw for setting the distance between sample and mask
5. Set screw for adjusting the sample laterally relative to the mask
6. Set screw for adjusting the sample vertically relative to the mask (protruded length above the mask edge
7. LN_2 Dewar
8. LN_2 pump
9. LN_2 hose

2.3 Safety information

2.3.1 General instructions

Danger!
The Leica EM TIC 3X Cooling Stage can be handled safely and easily provided it is operated in accordance with the instructions in this manual.

Non-observance of these safety instructions may endanger people and the system.

2.3.2 Design safety measures

All electronic components are protected by covers (doors, panels, etc.). These covers must not be opened except for servicing by an authorized Leica representative.

Caution!
There is a danger of electric shock when the cover is removed.

The Leica EM TIC 3X Triple Ion Beam Cutter 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.

The Leica EM TIC 3X Cooling stage must not be operated unless all covers are properly in place.

2.3.3 Safety measures at the installation site

The following measures must be implemented to prevent incorrect use at the installation site:

- The system must be operated by trained and authorized personal.
- Repairs may only be made by authorized staff of Leica representatives.
- If the Leica EM TIC 3X Cooling Stage is installed incorrectly, the system may be damaged.

Note!
The system should be switched off for maintenance and servicing only, not at the end of a work session. Please keep the system under vacuum condition even if a sample is not being processed.

2.3.4 Qualification of operating personnel

The operating personnel must be familiar with and follow the recognized provisions for safety at work.

The operating personnel must be trained and familiar with the duties that have been assigned to them and for which they are responsible.

2.3.5 Residual hazards

The Leica EM TIC 3X Triple Ion Beam Cutter system represents the latest technology and conforms to recognized safety regulations: even so, hazards may still exist.

If the Leica EM TIC 3X Triple Ion Beam Cutter system is damaged or malfunctioning, 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 leads to exclusion of guarantee!

2.3.6 Emergency procedure

If unusual operating conditions or unaccustomed noises occur, the system must be switched off using the main switch on the system if necessary.

If firefighting measures are called for, a CO_2 fire extinguisher must be used.

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

2.3.7 Liquid Nitrogen (LN_2) used for the cooling stage

The volume of the LN_2 Dewar is 25 l. When working with liquid nitrogen (LN_2) please bear in mind that LN_2 is extremely cold. It boils at -196 °C. Nitrogen gas (GN_2) escapes at very low temperature from the boiling LN_2. Both LN_2 and GN_2 as well as cooled elements (e.g. pipes, valves, hoses, containers or stoppers) can cause severe frost bite and burns to the skin and eyes.

When LN_2 evaporates, it expands in a ratio of 1:700. 1 litre LN_2 produces almost 1 m^3 of GN_2. Care should therefore be taken to ensure that when large quantities of nitrogen evaporate (e.g. when transferring LN_2), the room should always be well ventilated.

Removing LN_2 waste: dump LN_2 into an outdoor pit or container filled with gravel, where it will evaporate rapidly and safely.

GN_2 is odourless and tasteless and will be inhaled like air. GN_2 is non-toxic, but a high GN_2 content in the air (> 78 %) reduces the oxygen-content (< 21 %) and produces immediate fainting and deep unconsciousness without any previous symptoms.

When there is doubt about the adequacy of ventilation, use an oxygen analyser (0 to 25 % scale) to check for oxygen. The content of oxygen must not drop below 18 %.
If an unconscious person stays in a low oxygen environment then death may occur.
If breathing stops, apply artificial respiration at once and notify doctor and ambulance immediately!

For the reasons given above, never put LN_2 Dewars in a closed storage room or chamber. The evaporation rate from Dewar vessels can rise to several litres a day if they are defective due to improper handling or to natural wear over many years of use.

Always keep the working area well ventilated.

Bring objects at room temperature carefully into contact with LN_2. Initially an insulating gas layer is formed preventing a large transfer of heat. During this initial period little LN_2 evaporates. However, once the object has cooled down there may occur unexpected strong boiling and spurting of LN_2.

In the case of burns from LN_2 splashes, rinse the affected skin immediately with plenty of water at hand temperature. For serious burns arrange for a skin specialist to see them at once.

In the case of LN_2 affecting the eyes, rinse immediately with water at hand temperature and arrange for an eye specialist to see it at once.

Never use glass Dewar vessels in the lab (especially glass Dewars larger than 2 litres capacity) without complete metal envelope: Glass Dewars often burst for no obvious reason or due to unintentional mishandling (e.g. contact with metal instruments etc.). Never work without open protective glasses when using LN_2 in a glass Dewar.

Estimation of lethal GN_2 – concentration in closed rooms.
Full-load values (10 kV, 3.5 mA, -150 °C), room temperature ~25 °C

Size of the room [m^3]	***10***	***21***	***31***	***42***	***52***	***62***	***73***	***83***	***94***	***104***
Time to achieve critical concentration [h]	***1***	***2***	***3***	***4***	***5***	***6***	***7***	***8***	***9***	***10***

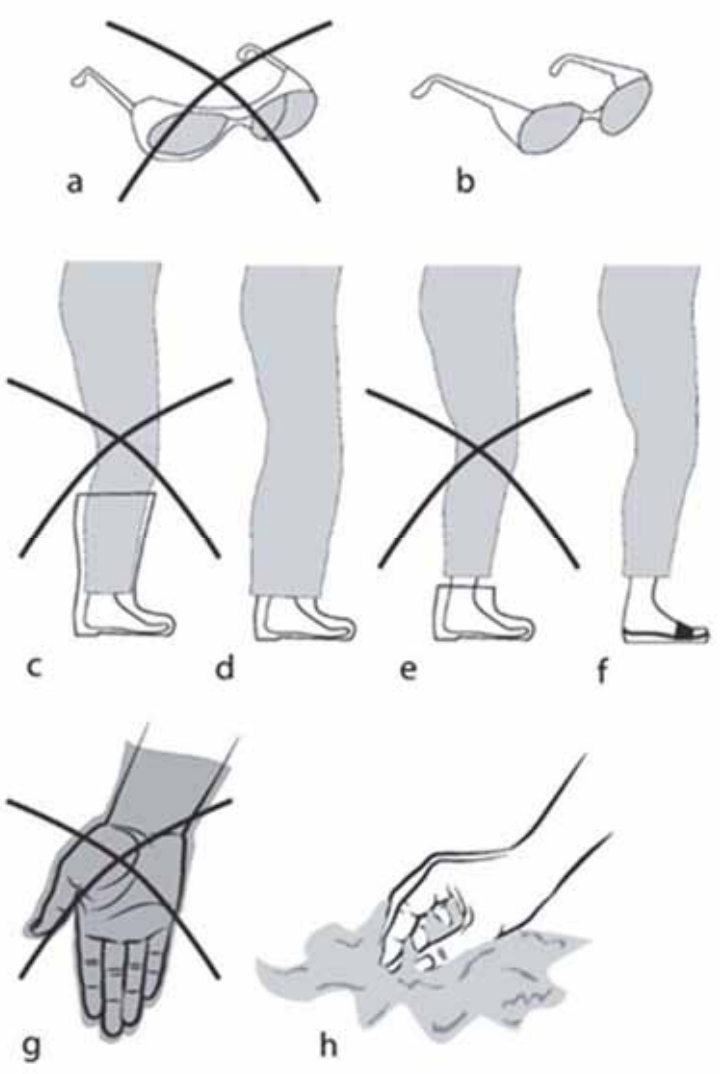


Fig.1.1: When working with LN_2 for refilling the Dewar avoid protective glasses (a), boots (c), walking shoes (e) and protective gloves (g) out of which the LN_2 cannot easily escape if entered. LN_2 splashing into the closed protective glasses (a), open boots (c), shoes (e) or protective gloves (g) evaporates suddenly and can cause serious burns.

Always use protective glasses (b) with side protection which are open at the top and at the bottom. Only use boots if you have loose (not narrow) trousers coming outside the boots (d) and completely covering the gap. Wear only open slip-on sandals (f) in the lab, no walking shoes or court shoes. Always wear cuffless trousers if you wear slip-on sandals. Never wear protective gloves when pouring LN_2 or when putting the Dewar head on the Dewar vessel. Just use an open flannel cloth (h) to protect your hands from the cold. Gloves should be used only to grasp dry cold parts. They are unsuitable for LN_2 work.

Only use metal Dewars specifically designated for storage of LN_2, since only containers of this kind exclude risks during storage. For routine cryopreparation metal troughs (1 cm Styrofoam insulation), Styrofoam containers or plastic troughs are eminently suitable and ensure low risk cryopreparation.

Check the evaporation rate of your metal Dewar regularly every three months and compare these rates with the rate given by the manufacturer. The evaporation rate of an undamaged metal Dewar should be well below 1 litre of LN_2 per day. Defective Dewar vessels with higher evaporation rates are a safety risk, and should be taken out of work or repaired.

Do not leave LN_2 standing in open vessels where it can exchange with the room atmosphere. The boiling point of LN_2 (-196 °C) is lower than liquid oxygen's boiling point (-183 °C). When the exchange surfaces are extensive enough, oxygen from the air will be taken up in exchange for nitrogen. LN_2 with high liquid oxygen content has a faintly bluish colour. Concentrated liquid oxygen promotes vigorous burning!

Make sure that your Dewar vessel is filled only with LN_2. Apply a note in the central distribution place stating clearly

ONLY LIQUID NITROGEN

or similar if different liquefied gases are delivered from there. Check the colour of cryogen: Bluish colour indicates the presence of a high percentage of liquid oxygen.
The concentration of liquid oxygen increases during long periods of storage as its boiling point (-183 °C) is higher than the boiling point (-196 °C) of LN_2.

Main supply must be assured: 100 – 240 VAC, 50 / 60 Hz.
The instruments are equipped with protected ground. Before connecting it to the local electrical supply make sure that the mains has the required ground and that the instrument is connected to it according to the local regulations.
Unplug the instrument before installing or changing fuses.

HAZARD WARNING

LIQUID NITROGEN, LN_2

Suffocation

- Any vessel containing LN_2 is a potential hazard
- One litre LN_2 produces 700 litres N_2 gas
- N_2 gas is odourless and tasteless
- Oxygen levels can quickly drop in confined spaces due to displacement of oxygen
- by N_2 when using or dispensing large volumes of LN_2
- This can cause immediate fainting and unconsciousness
- Always use LN_2 in well-ventilated areas
- Treat it with respect!

Storage

- For reasons mentioned above do not store full LN_2 Dewars in confined spaces

Burns

- LN_2 boils at -196 °C. It is extremely cold and can cause serious burns. Please read the safety instructions provided with all Leica products for the correct handling of liquid nitrogen!

3. Installation and setup

3.1 Transportation and storage conditions

The Leica EM TIC 3X Cooling Stage is delivered properly packed and in the assembled state or in case of delivered with the Leica EM TIC 3X already installed in the instrument. The customer must 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 of any possible damage in transit.

The packed equipment must be stored in a clean, dry area. It must not be exposed to aggressive or corrosive substances.

3.2 Safe storage of the packing material

The packing material for the Leica EM TIC 3X Cooling Stage should be retained for future use in case the instrument needs to be transported. Damage to the system may occur if it is not transported in the original packaging. The packaging material is designed for transportation and storage of the instrument. Storage of the stage should be in the black foam part of the package, please do not dispose of this part.

3.3 Storage location for the instructions

The user manual and associated supplementary documentation (e.g. documentation for suppliers' components) must be kept close to the Leica EM TIC 3X Triple Ion Beam Cutter system for fast access.

3.4 Preparing the LN_2 pumping system

Connect the LN_2 hose with its threaded connection to the instrument.

Remove the yellow protective cup from the lower end of the pump. Slowly lower the pump into the Dewar filled with LN_2. Hold the pump for a while until the strong boiling decreases and carefully lower the pump until it is completely immersed.

The clean Dewar vessel has to be filled with LN_2 according to the safety precautions.

Connect the LN_2 hose with its threaded connection to the LN_2 pump. Open the shutter by pushing the lever before connecting the hose.

Connect the LN_2 pump on the rear side of the instrument.

Remove the nozzle protection, close to the vacuum chamber.

4. Operating instructions

4.1 Introduction

The ion beam cutting process is performed using argon. Argon ions that have been ionized and accelerated by high voltage collide with the sample and displace surface atoms. The sample is shielded by the mask, so that a 90° cut (cross-section) is created into the sample which protrudes at a certain distance above the mask edge. Hence, the roughness of the pre-prepared sample surface can be left at a lower degree. As a rule of thumb mechanical artefacts of a distance about 3x the grain size of the abrasive (used for pre-preparation) can be introduced in the sample. For example, the protruded distance above the mask edge should be set > 30 µm if a 9 µm lapping foil was used for pre-preparing the sample.

The stage is used for loading the system with the sample and must be opened in order to change samples. The Cooling Stage supports both the mask holder with the mask and the sample stage, which can be moved in three axes using micrometer set screws. The following displacements can be carried out for the individual axes:

- lateral: 10 mm
- vertical: 2 mm
- travel range between mask and sample: 6 mm

The stage can be tilted through 90° to adjust the sample.
The sample is secured on a removable sample tray.

The Cooling Stage is mainly used to prepare heat sensitive samples. Hence, the sample holder and the mask can be cooled down to -160 °C using LN_2 supplied from an external 25 l Dewar. Heating up of the sample can only be done under vacuum conditions, thus water contamination on the sample is avoided. The Cooling Stage can also be used without LN_2 cooling.

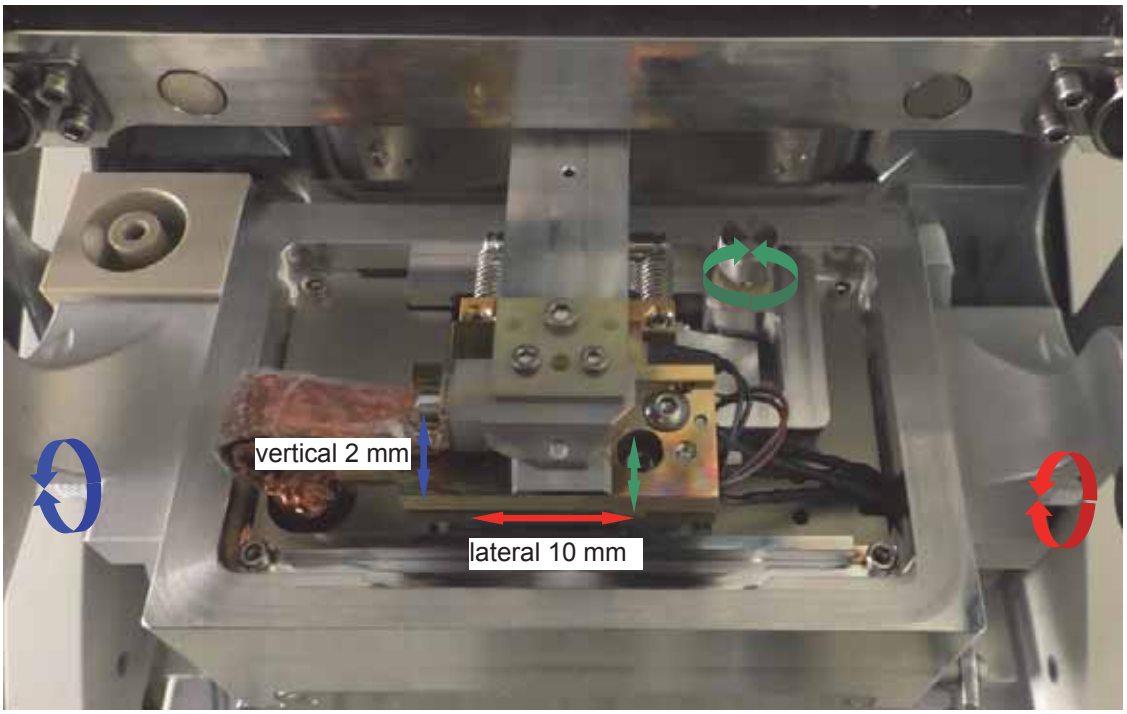
Three types of gold plated copper sample holders are available:

for sample size: 10 x 10 mm and 0 to 4 mm thickness

for sample size: 10 x 10 mm and 2 to 7 mm thickness

for sample size: 25 x 25 mm and 0 to 5 mm thickness

Table flange with Cooling Stage and displacement paths:



vertical position is set using the left hand set screw

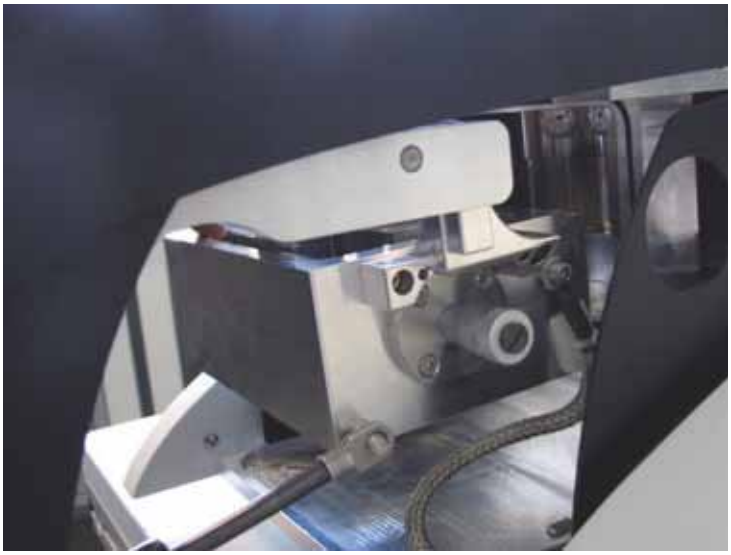
lateral position is set using the right hand set screw

distance between sample and mask is set by using the centre set screw (travel range: 6 mm)

4.2 Exchanging the stages (when different stages are ordered)

The instrument is delivered with one stage built into the instrument. To exchange the stage please proceed as follows.

1. Open the flange and swivel the stage to its horizontal position.



2. Retract and unhinge the flap damper.

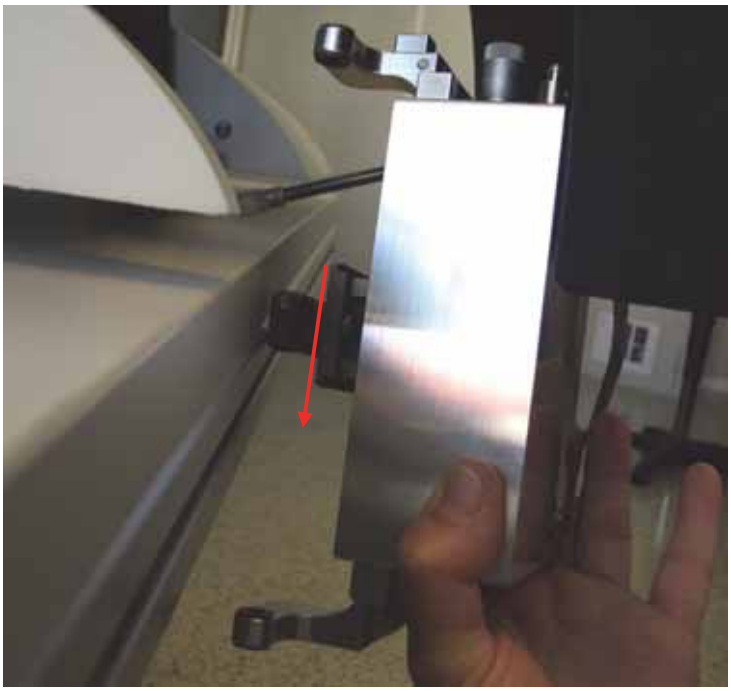
Disconnect the plug.

The plug is equipped with a locking mechanism. Please do not pull on the cable! Grasp the knurled part of the plug and retract for disconnecting the cable!

3. Slide the preventer until the bearing is completely visible.

4. Move the complete stage to the left.

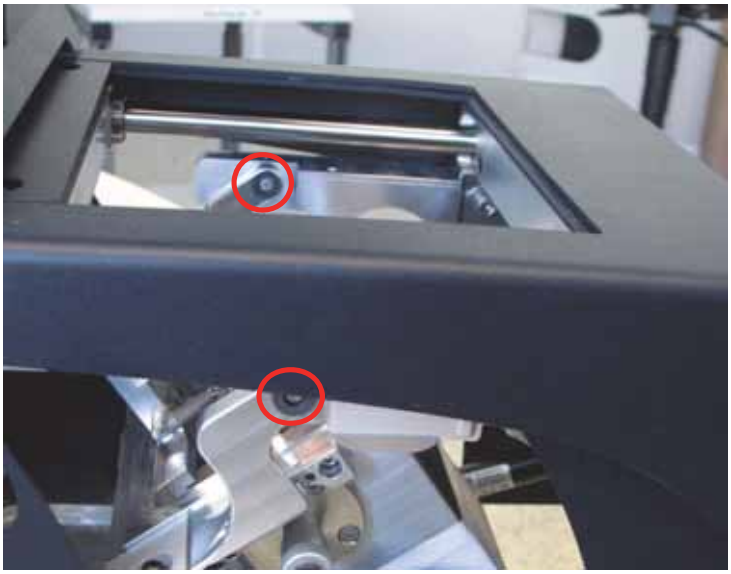
5. Turn the stage 90° and withdraw it in the vertical direction.



6. Place the stage into the designated storage box. This is used to protect the flange surface from scratches which might influence the sealing and evacuating the chamber.

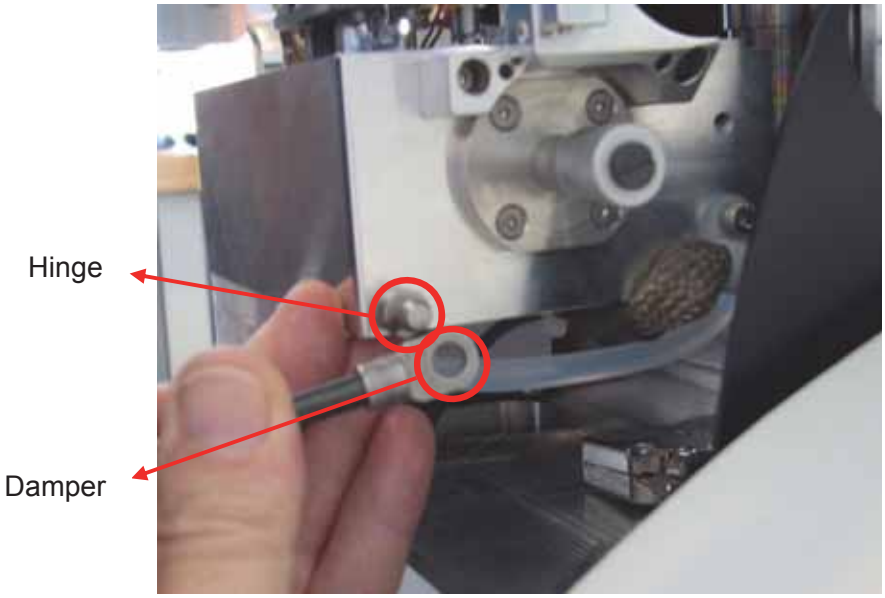
7. Insert the other stage in the vertical position.

8. Hinge the stage on its two bearings.



9. Slide the preventer to cover the bearing completely before you make any other connections.

10. Swivel the stage into its horizontal position, slightly retract the flap damper and connect to hinge point.



11. Connect the plug of the stage.

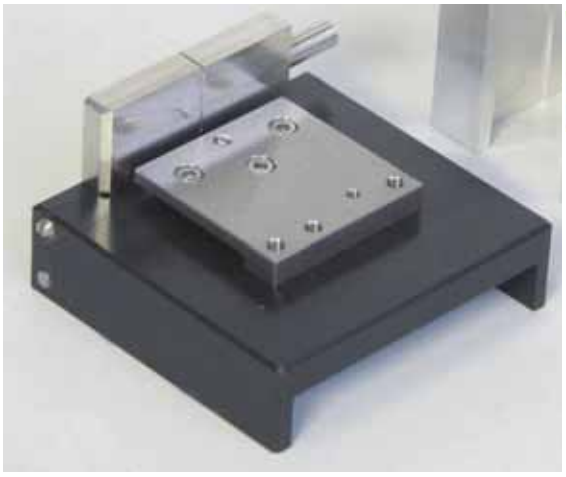
12. In case the instrument is switched on during the stage exchange, select Menu followed by Setup and initialize the stage.

Initialize the cooling stage by pushing the Initialize button in the Setup menu. It takes about 1 minute to detect Cooling stage and Dewar level. The completed initialization is performed as soon the snowflake symbol changes from grey to blue.

Initialization must be performed every time the stage or the light has been exchanged if the instrument is switched on. When the instrument is switched off, the stage (or light) will be initialized when switching on the instrument.

4.3 Mounting the sample

Adjustment jig and sample holders (gold plated copper):

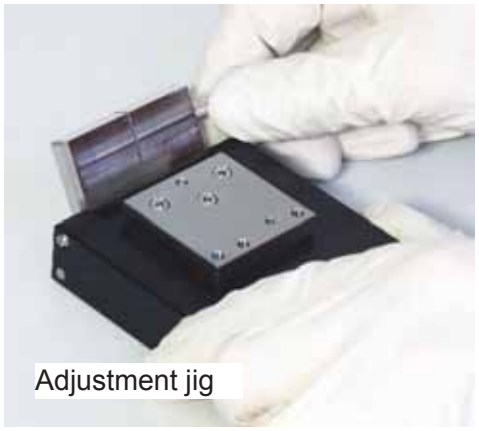


For sample size:
25 x 25 mm
0 to 5 mm thick

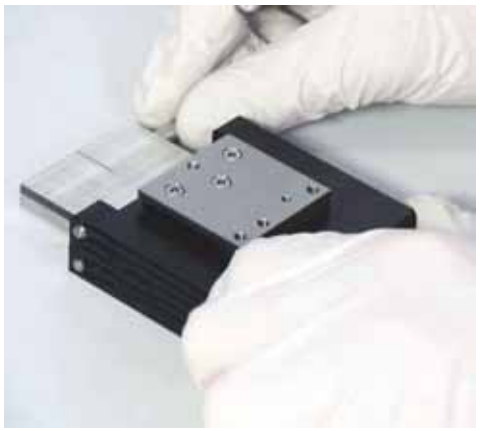
For sample size:
10 x 10 mm
2 to 7 mm thick

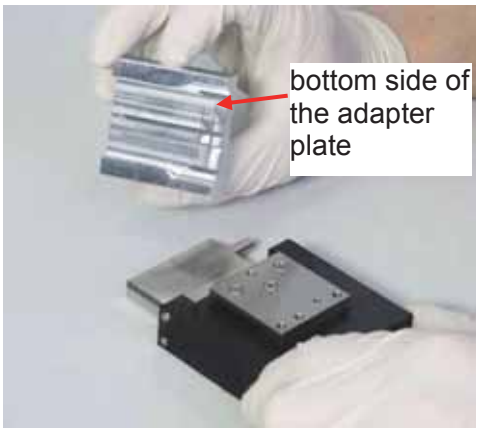
For sample size:
10 x 10 mm
0 to 4 mm thick

Preparing the adjustment jig for attaching the adapter plate:

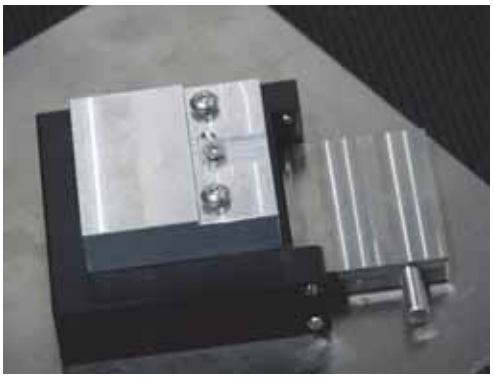


Adjustment jig open and adapter plate ready:





Adapter plate is pushed to the click stop position onto the adjustment jig.

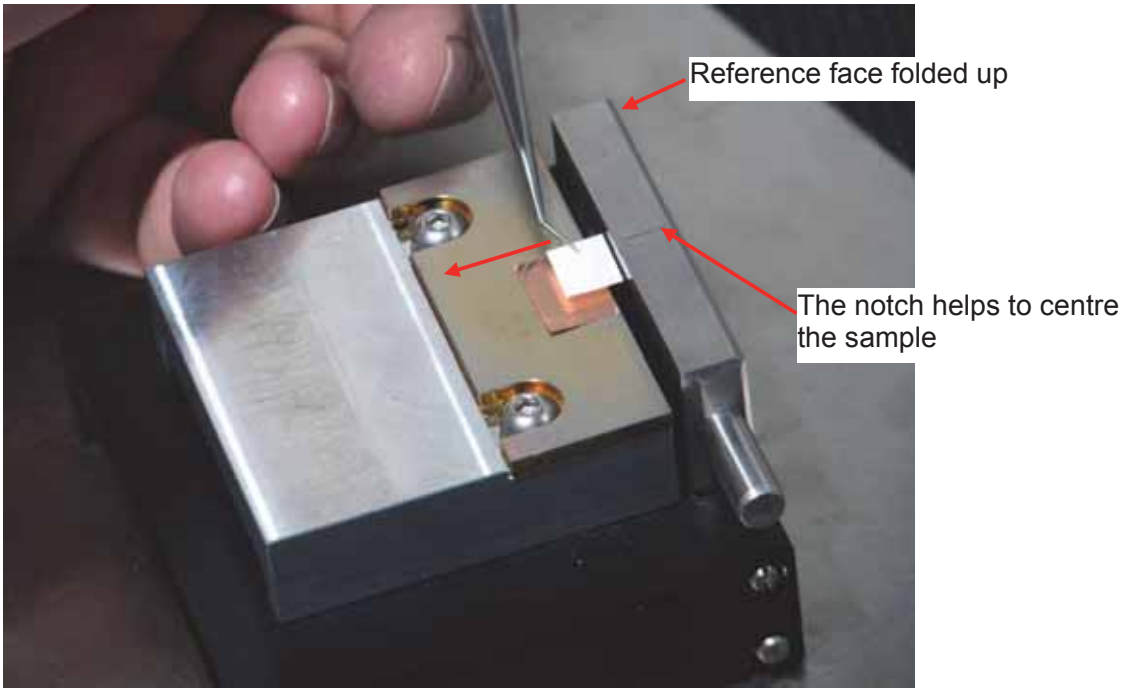


For the best heat transfer it is recommended to secure the sample with double-sided adhesive tape (e.g. C or Cu). However if not possible due to the shape of the sample, the sample can be secured on the holder with a high melting point thermoplastic wax or super glue.

Insert the sample holder and slide it towards the hit stop.

Clamp the sample holder with the hex screws.

Align the sample with the tweezers at the reference face and stick it onto the prepared sample holder.



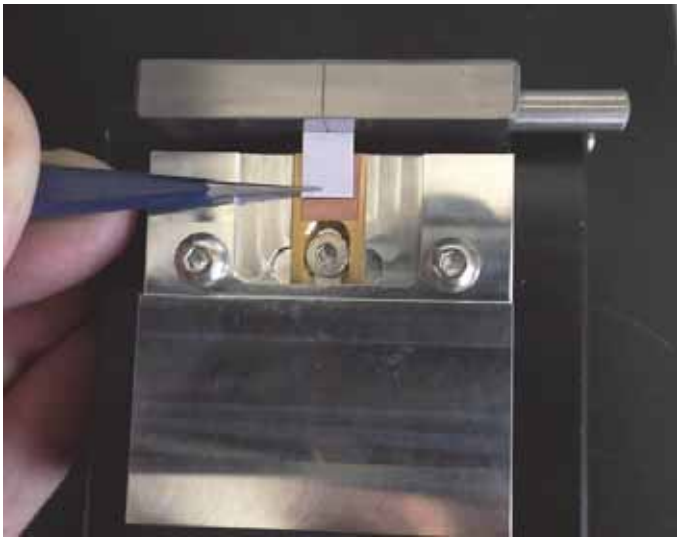
.

The small sample holders are inserted in the centre notch.

Insert the sample holder and slide it towards the hit stop.

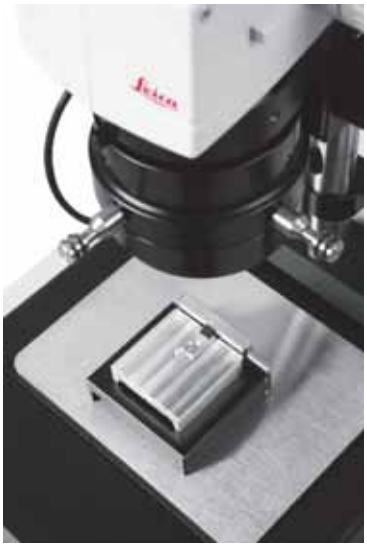
Clamp the sample holder with the hex screw.

Align the sample with the tweezers at the reference face and stick it onto the prepared sample holder.



Once the sample is positioned on the holder, flap the reference and remove the sample holder by opening the centre screw.

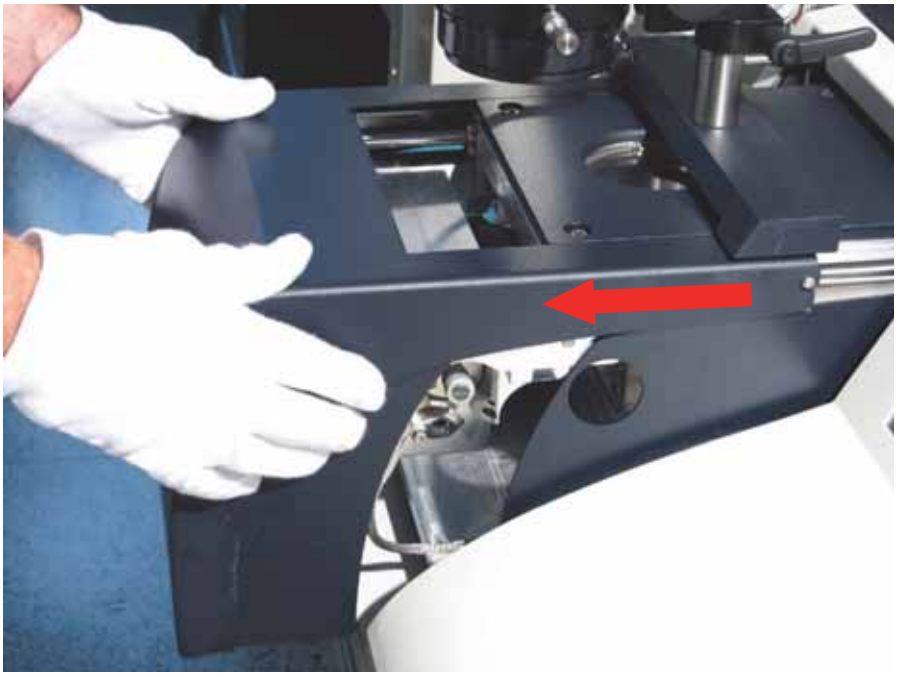
For accurate alignment, it is recommended to use the stereo microscope of the instrument. For doing so place the workstation plate (option) directly onto the viewing port of the chamber and set the stereo microscope proper position (red ring on the column for M80 and S6, black ring M80 equipped with the 1.6x objective).



4.4 Inserting the sample

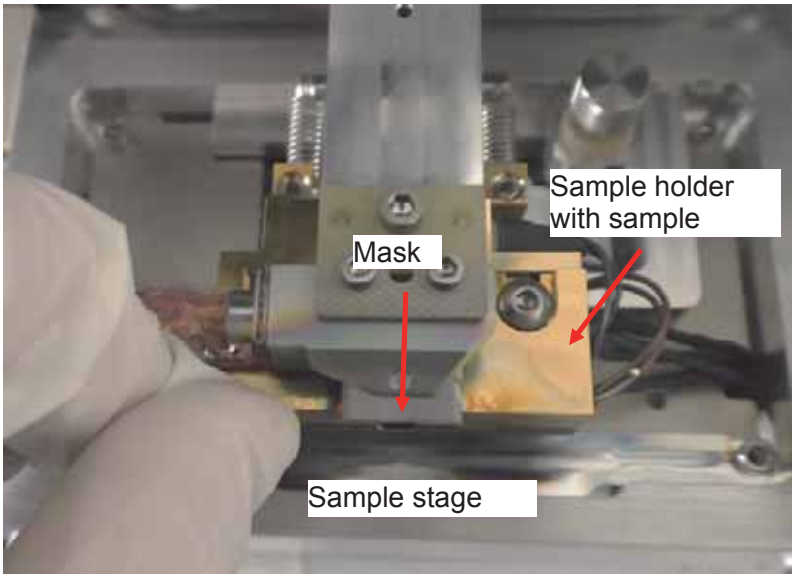
Due to the different coefficient of expansion, the set values of the sample might change within µm when cooling down the sample. However, the sample can be re-adjusted in vertical and lateral directions using the setscrews due to the openings in the housing on the left- and right hand side.

Vent system and open the table flange of the Leica EM TIC 3X.

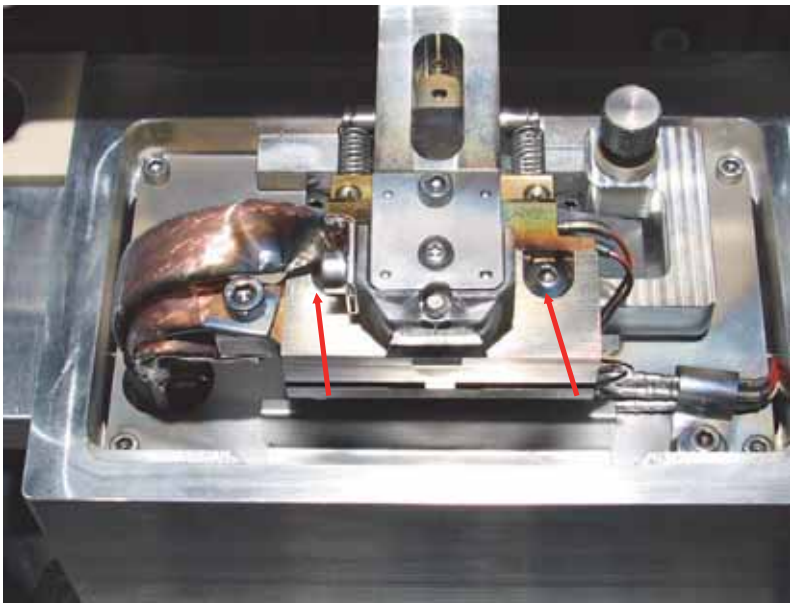


Before inserting the sample holder make sure that the centre screw is screwed in. If not use hex key inserted through the hole of the mask holder.

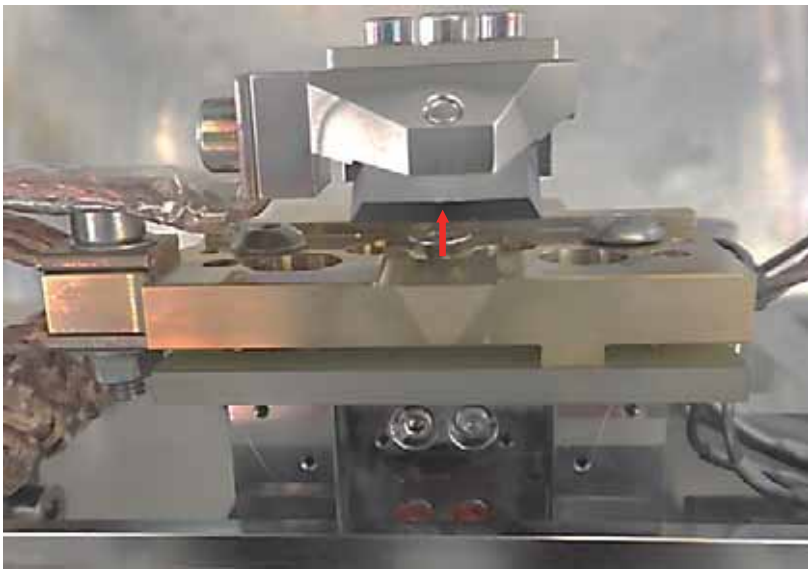
Make sure the sample will not interfere with the mask edge, use the set screw to retract. Insert the sample holder with the sample in the stage in horizontal position until the sample holder rests at the limit stop.



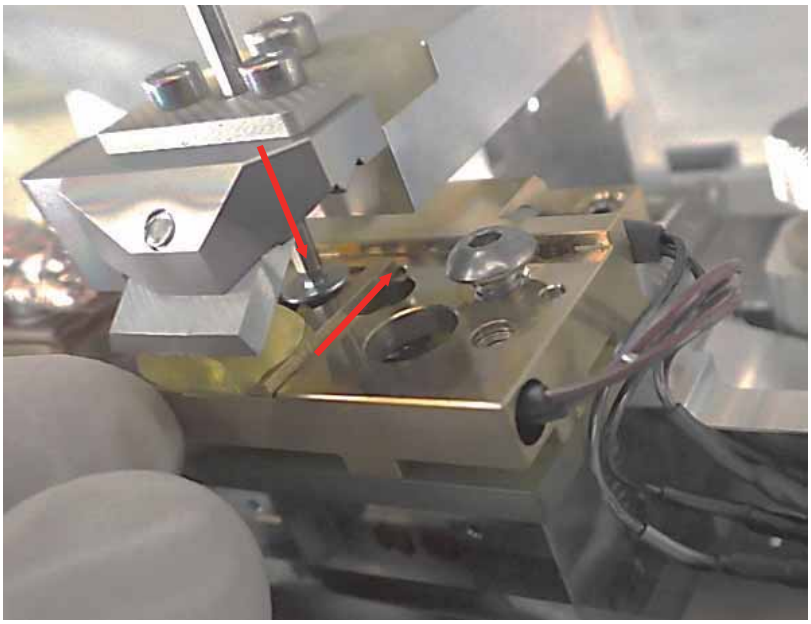
Tighten the sample holder with the hex-screws.



Before inserting the small sample holder open the centre screw until the bracket of the sample holder can be slid in.



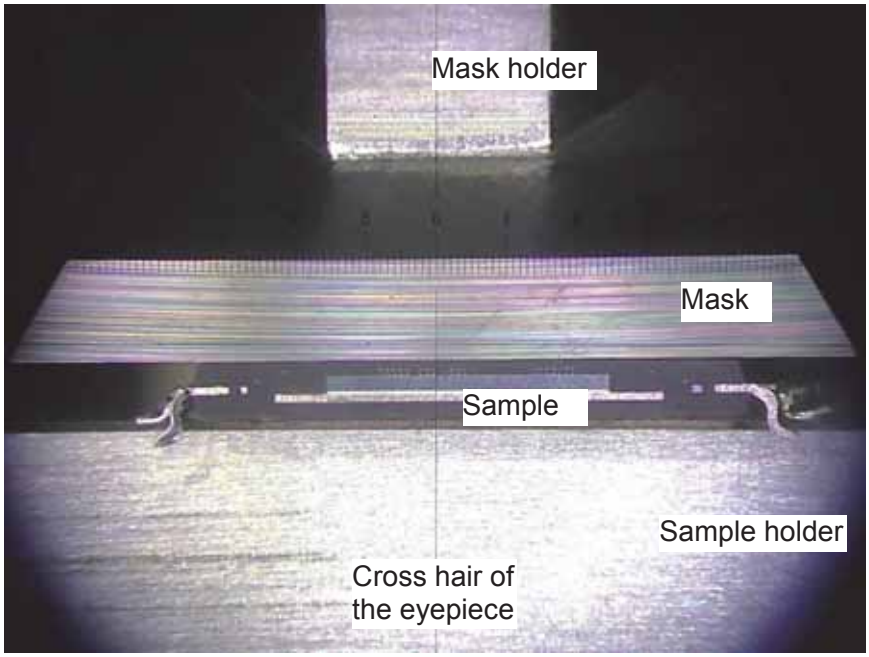
Make sure the sample will not interfere with the mask edge, use the set screw to retract. Insert the sample holder with the sample in the stage in horizontal position until the sample holder rests at the limit stop and tighten the centre screw with the hex key.



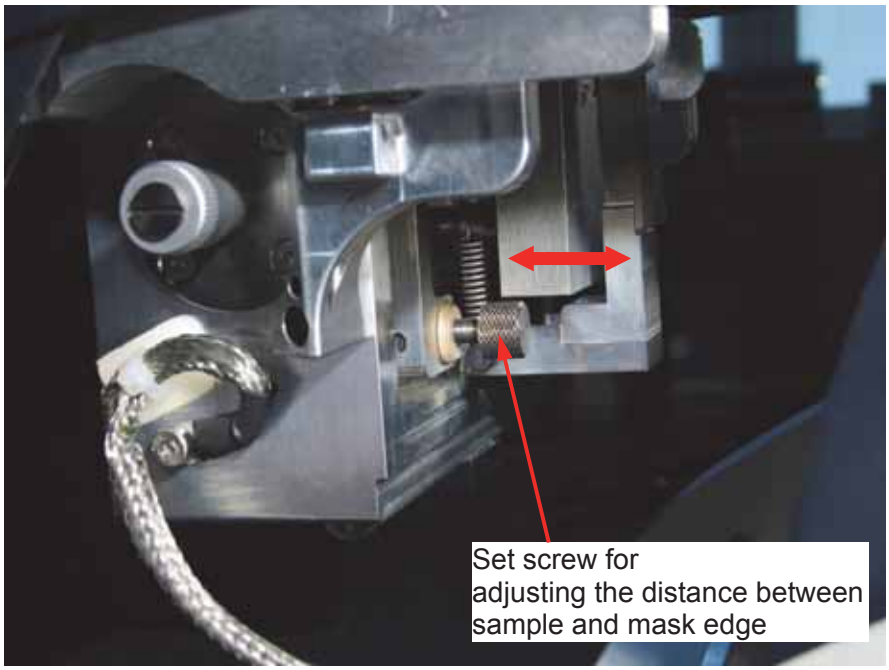
4.5 Adjusting the sample using the stereo microscope

The sample is adjusted under the stereo microscope. Set the stereo microscope in the proper position (lower red ring on the column for Leica M80 equipped with the 0.8x objective, lower black ring Leica M80 equipped with the 1.6x objective).

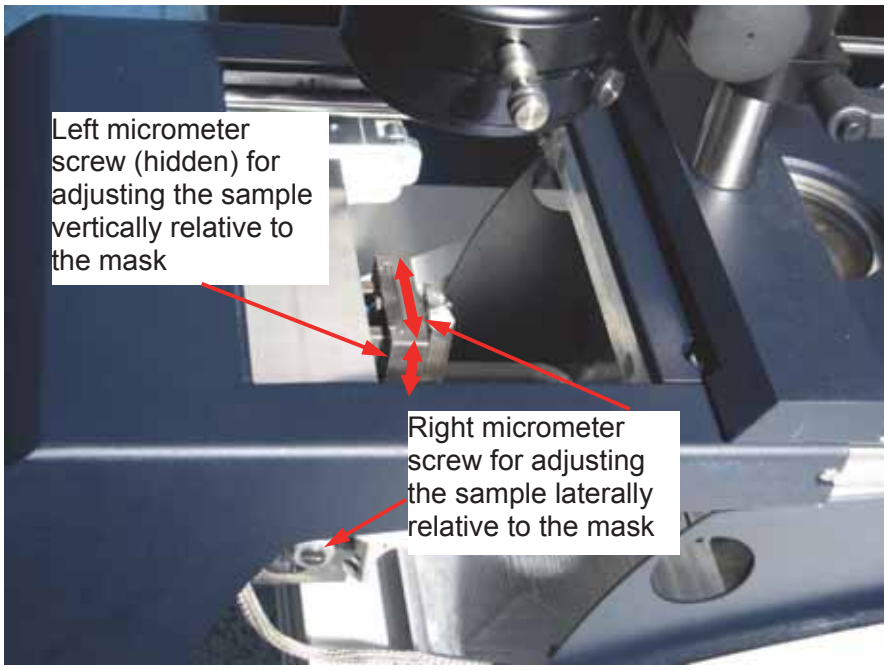
Parallelism of the sample edge relative to the mask is extremely important for the quality and evenness of the cross section. The sample must be moved as close as possible to the edge of the mask, but do <u>not apply</u> pressure against the mask edge. If the sample surface is too far from the edge of the mask, the sample surface edge becomes rounded. To avoid re-deposition on the sample surface protruded length of the sample above the mask edge should not be set above 100 µm.



Options for adjusting the distance between sample and mask:

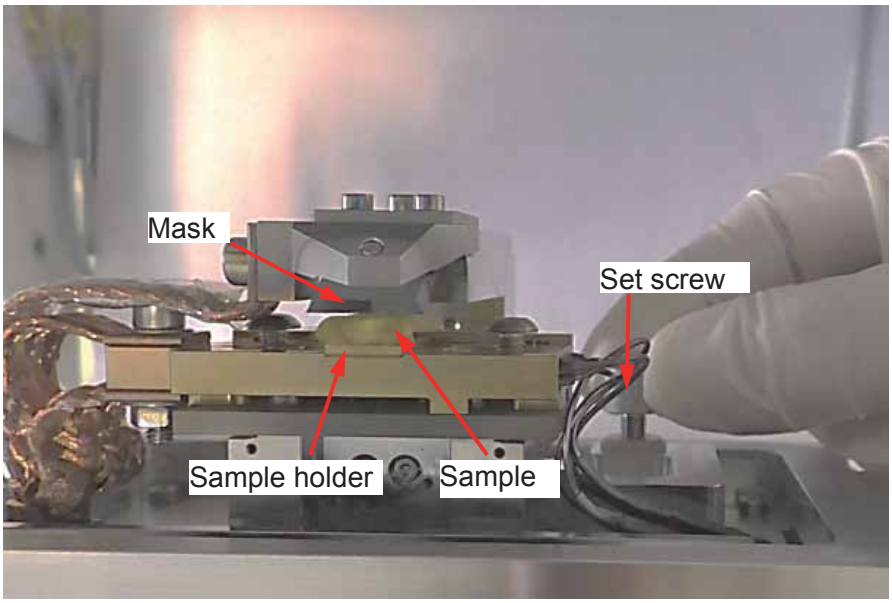


Options for adjusting laterally and vertically relative to the mask:

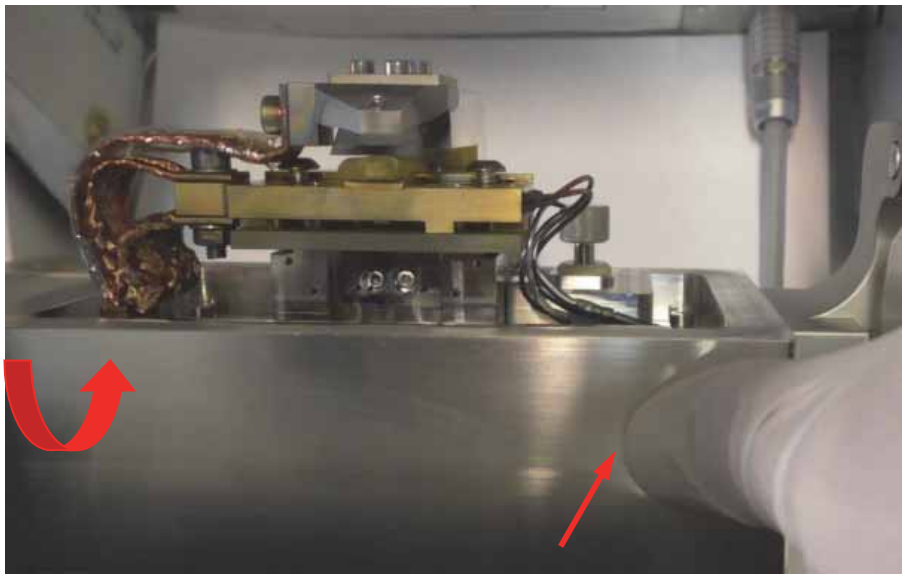


The cooling stage is tilted through 90° for adjusting the sample laterally and vertically. Adjustment is done with the micrometer screws under the light microscope until the target (area of interest) appears in the centre and level with the mask edge. The centre of the mask holder corresponds to the crossover of the ion beams.

Adjusting the distance between the sample and the mask
(cooling stage in vertical position):



To adjust the protruded length of the sample swivel the stage in horizontal position by pushing the topside of the stage housing.

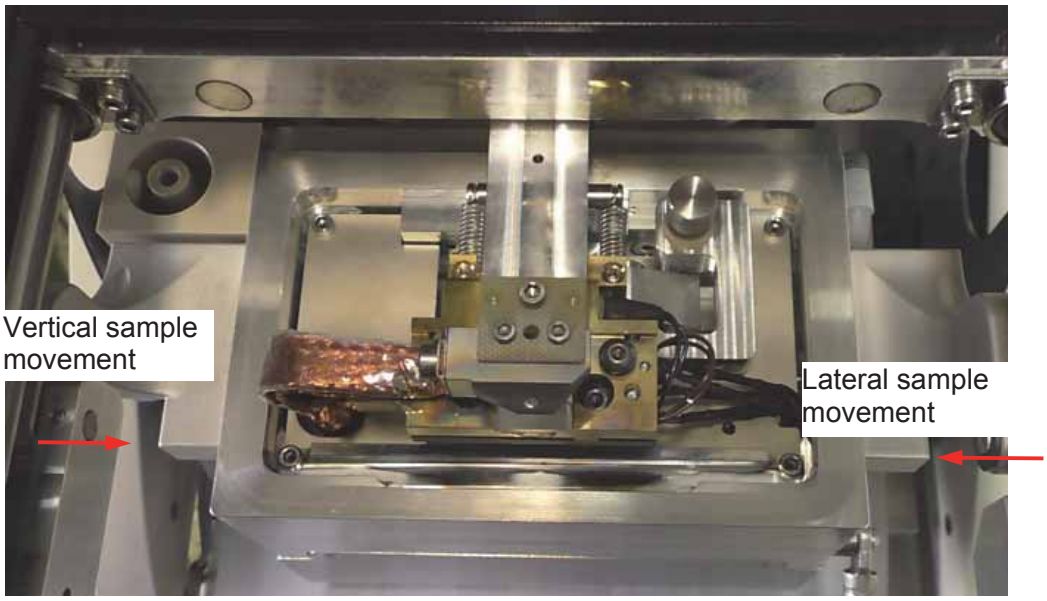


Caution!
There is a hazard when the table flange is tilted.

Personal injury can occur, e.g. trapping fingers.

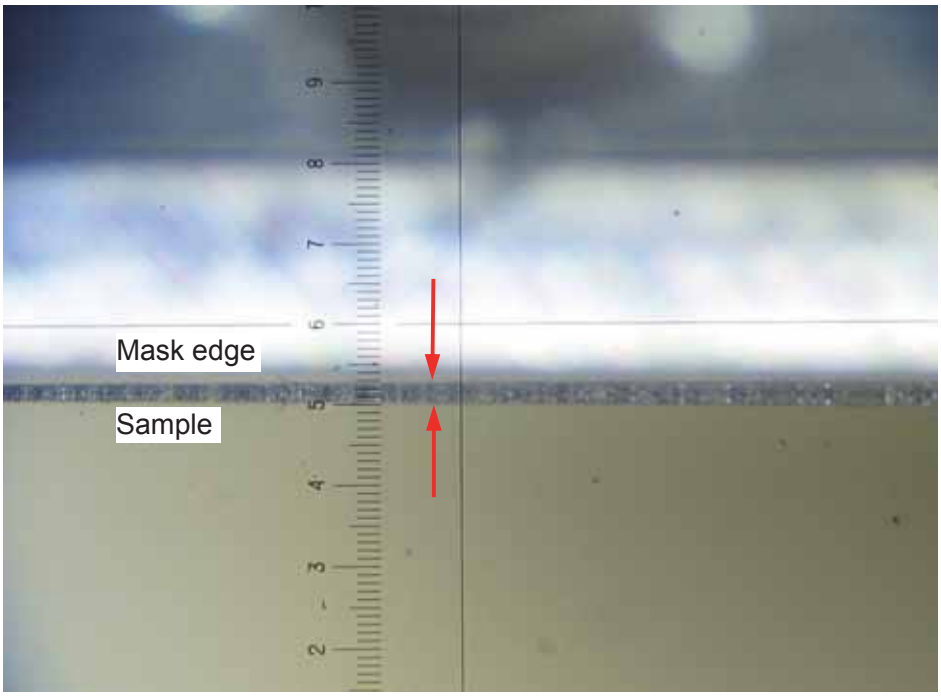
Watch your fingers when tilting the table flange.
Tilt the table slowly and carefully.

Lateral and vertical adjustment of the sample relative to the mask:
(cooling stage in tilted position)



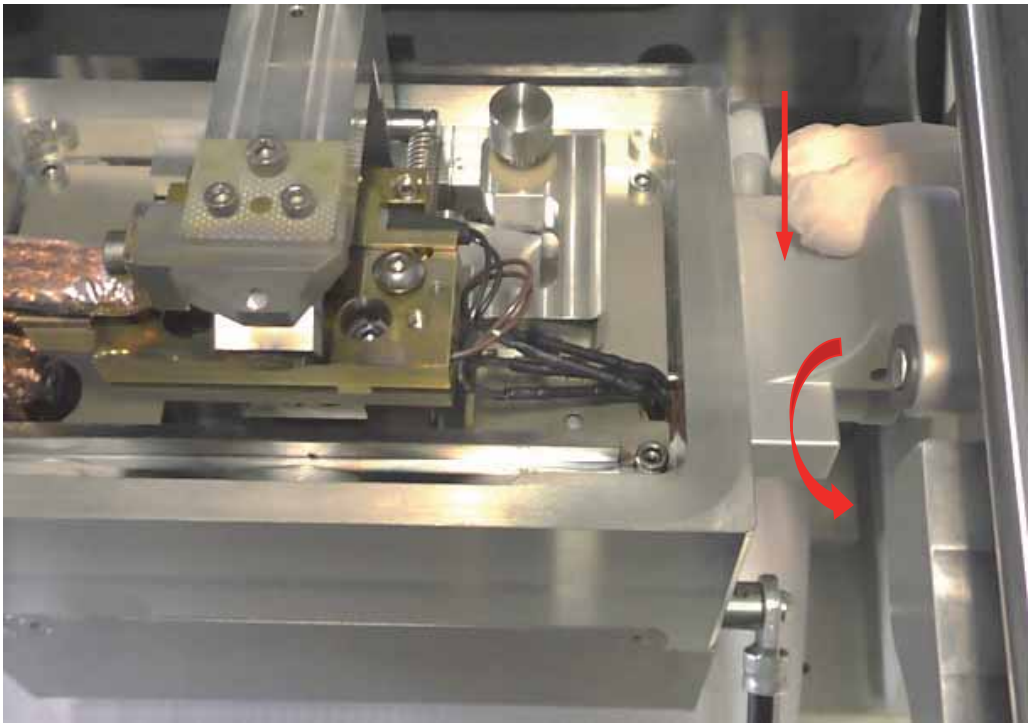
Vertical setting: for optimum results the protruded length of the sample should be between 20 µm and 100 µm. The distance can be measured with the reticule of the eyepiece of the stereo microscope.

Note: please see information about scales of the stereo microscope in the manual of the basic instrument, e.g. one scale division by using the M80 microscope equipped with the 0.8x objective equals to ~15 µm at the highest zoom setting (magnification).



4.6 Preparing for the cooling and ion beam cutting process

Swivel stage in vertical position.



Caution!
There is a hazard when closing the table flange.

Personal injury can occur, e.g. trapping fingers.

Close the table slowly and carefully.

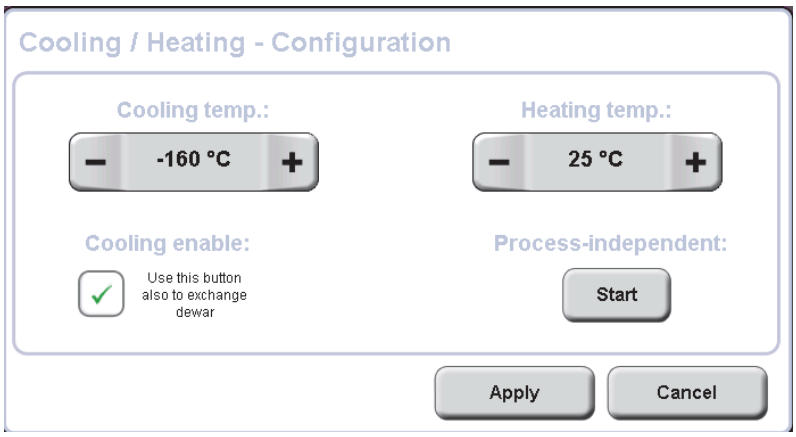
Press the Pump button on the touch screen. The status pumping symbol in the status field starts rotating. As soon as the working vacuum is reached the status symbol switches to green.

4.7 Setting the cooling temperature and activating cooling

The temperatures can be set in two different ways:

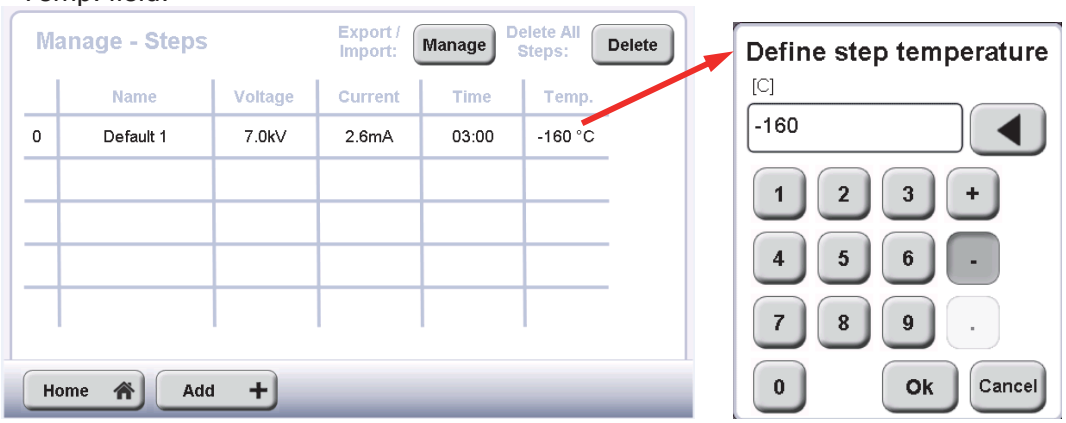
1. By pushing the symbol line with the snowflake symbol

a window appears to set the cooling temperature down to -160 °C. Furthermore the heat up temperature can be set. Warming up of the sample is automatically carried out at selected temperature under low vacuum as soon the vent button is pushed, thus avoids moisture contamination.



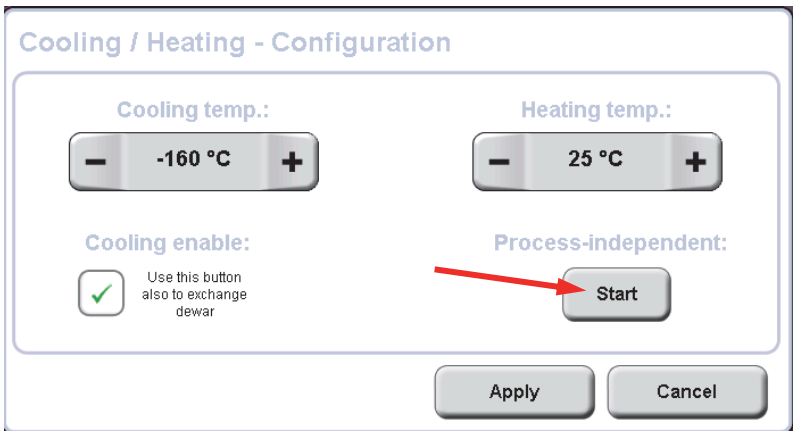
2. By pushing the symbol line for setting the sample process parameter

As soon as a step is selected the cooling temperature can be set by pushing into the Temp. field.



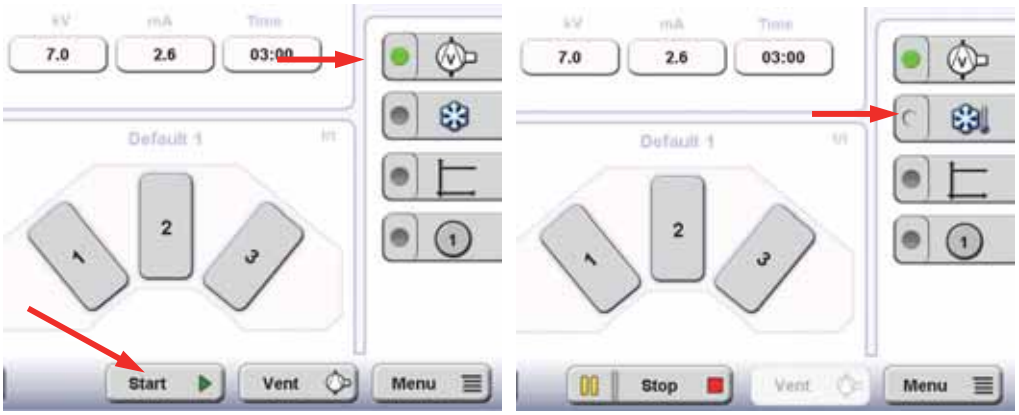
The cooling process can be activated in two different ways:

1. Process independent by pushing the Start button in the Cooling / Heating – Configuration menu.
   The cooling process starts as soon as the Start button is pressed, regardless if the vacuum is reached or the ion beam milling process has been activated. This setting is mainly used in conjunction with the vacuum/cryo transfer option. In order to keep the sample at cryo temperature even if the ion beam milling process has been stopped.

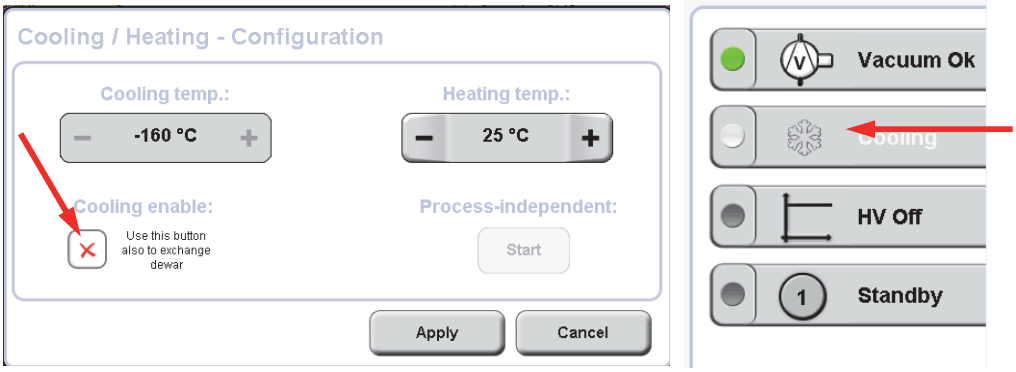


To avoid moistening of the vacuum chamber activate the cooling process if high vacuum is reached.

2. By pushing the Start button in the main menu, after the Pump button has been pushed.
   The cooling process starts as soon as the high vacuum is reached and the etching process starts automatically when the set temperature has been achieved. The cooling process stops when the etching process has been stopped. The sample will warm up, since the cooling process has been terminated.



Cooling can be disabled to perform ion beam cutting without a cooling process. Disabled cooling function will be indicated in the main menu by showing a gray snow flake symbol.



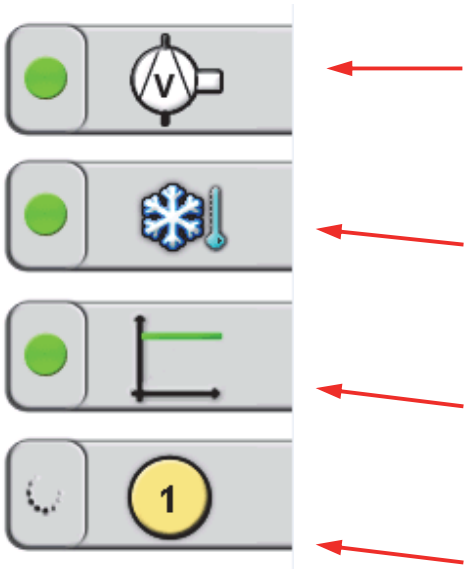
4.8 Starting the ion beam milling process

By pushing the Start button the system starts processing the sample according the set values you entered as soon as the set temperature is achieved, if cooling is enabled otherwise the milling process starts when the working vacuum is reached.

The Start button can be pushed subsequently after the Pump button was pressed and the system starts automatically as soon as the working vacuum is reached.

When a pressure in the vacuum chamber of less than 1.0E^{-4} mbar is reached, the start of the process is enabled.

Status information on the main screen:



As soon as the PUMP button is pressed the system starts evacuating the chamber down to < 1 x 10^{-5} mbar. The rotating sign indicates evacuation in progress. As soon as the working vacuum is reached the sign turns green.

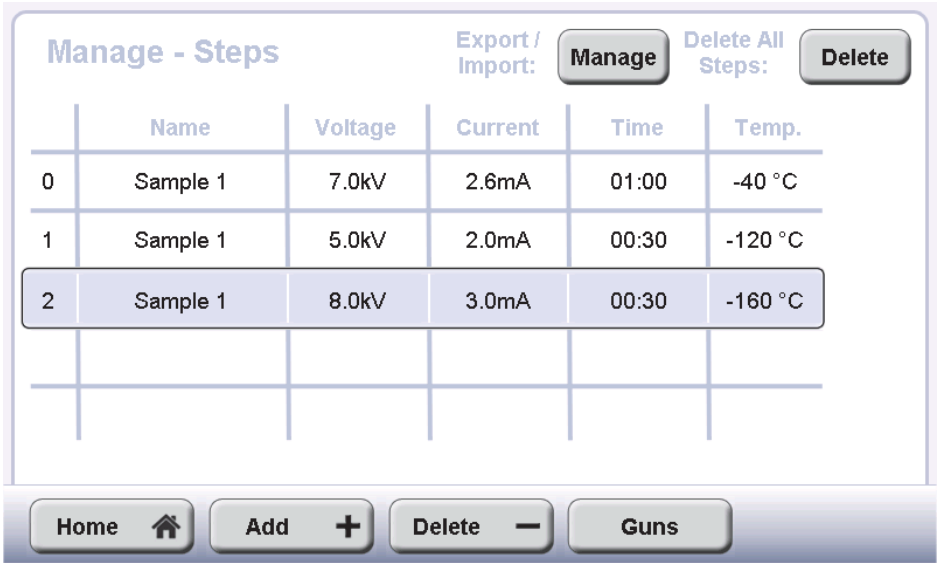
The symbol indicates the cooling process in progess. The set temperature is achieved when the sign turns to green.

As soon as the set voltage is reached the sign in the HV field turns from a rotating circle (in progress) to green.

Once the ion etching process is started, the sign in the Load-Up line turns to yellow (in progress). It switches to green as soon as the process is finished.

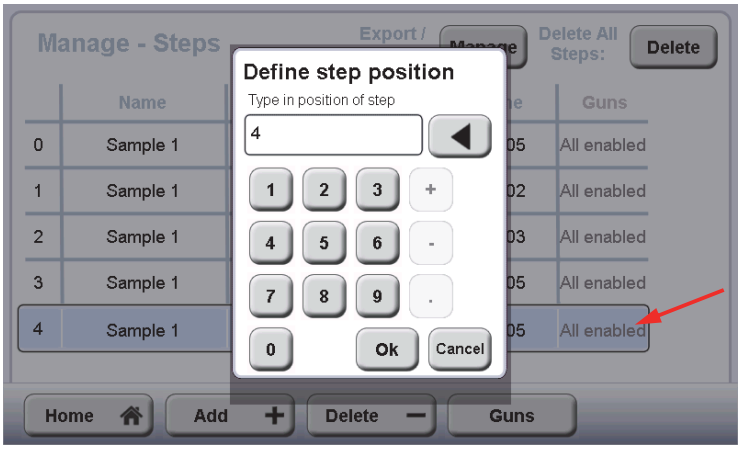
4.9 Programming

Before starting the ion etching process the sample name and gun settings can be edited and further processing steps can be added. Press the Load-Up button  and choose the following options:

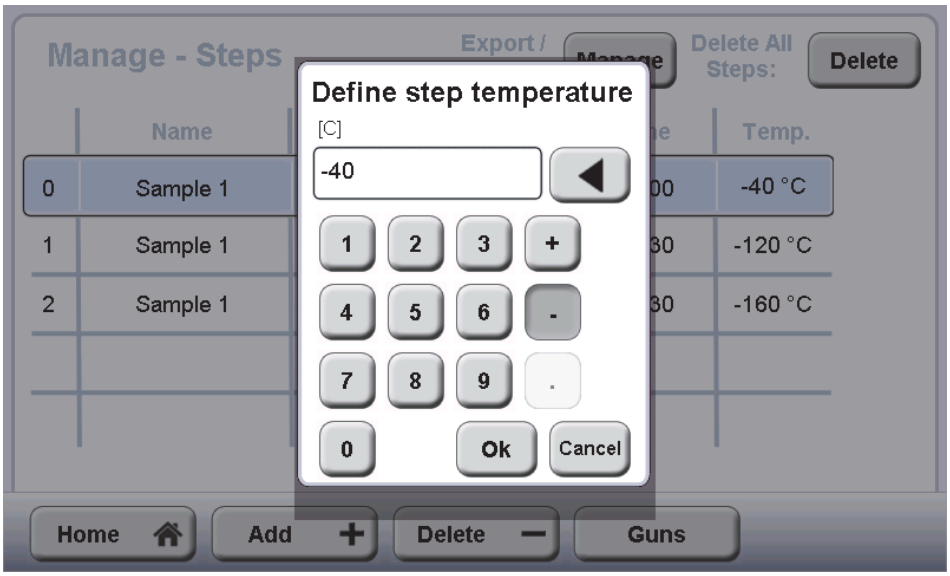


Add +: up to 50 additional processing steps can be added

When pushing the step line the steps can be re-arranged in different order by pushing into the position indication field. Furthermore, the sample or the guns can be edited by pushing into the desired field of the selected step line.



The temperature of each step can be set individually by pushing in the temperature field of the selected step line.



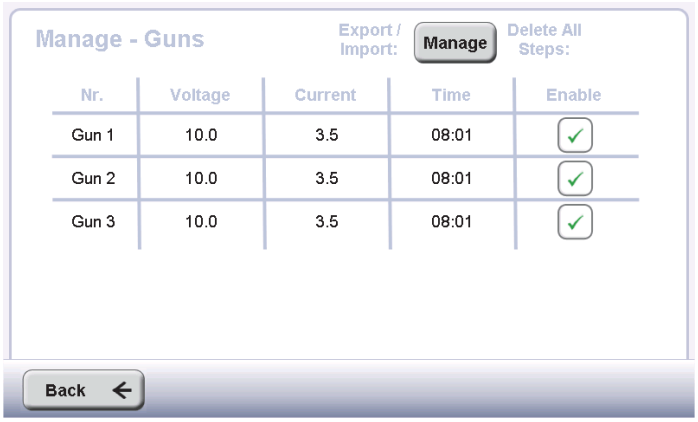
Delete - : the selected step can be removed

As soon as the recipe consists of more than one step the sign in the Load-Up line will change to a multiple step indication. The amount of steps is indicated in the sample information line of the main window (e.g. 1 sample, 3 steps). The last programme is active for the next sample process, even when the parameters on the main screen are changed. This change will affect the first step only. The instrument will continue according to the programme.

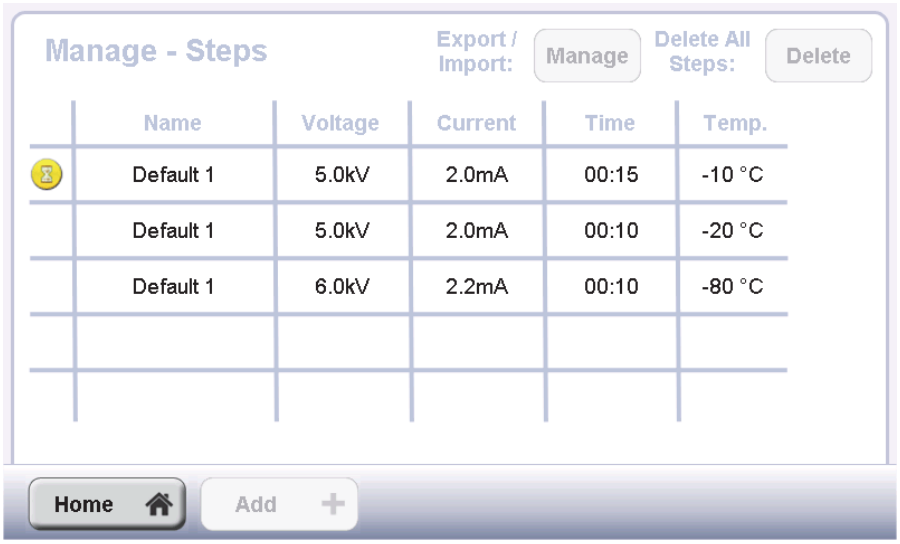
To delete the programme push the Delete button in the Manage – Step menu. Every step will be deleted except the first step.

Guns edit:

The gun parameters can be set differently for each step by pushing into the desired field of selected gun line. A star symbol beside the values (kV, mA, Time) in the parameter input line appears in the main window when selecting different gun parameters.



The progress of the recipe can be recalled by pushing the Load Up line: yellow sign = in process, green = finished process of the step.



4.10 Stopping the ion beam milling process

The milling process stops automatically when the pre-set processing time has elapsed. However, it can also be paused at any time by pushing the pause symbol or stopped by pushing the Stop button. Depending in which way the cooling process has been activated cooling the sample will be terminated or will remain after the ion beam milling process has been stopped.

4.11 Venting the chamber

Once the process is finished the system stops cooling if activated via the Start button in the main menu. To remove the sample the “Vent” button must be pushed. The sample will be heated up to the set value. The heat up process is symbolized with a sun symbol instead of the snowflake symbol. As soon as the set heat up process has been completed, venting the chamber will be performed automatically.

To avoid humidity inside the chamber, the chamber should be kept under vacuum even when the system is not in use. Please press “Pump” even when no further sample needs to be processed. You may switch off the mains switch when a long break is expected (longer than three months). The vacuum is kept at a certain level.

In order to prevent the surfaces becoming contaminated during venting, and particularly while the surfaces are heated, it is recommended to vent the system with nitrogen. To do this, connect a nitrogen gas bottle (or a fixed nitrogen supply line) to the venting valve connector on the back of the system instead of the air filter (see instrument manual). Using nitrogen for venting helps to improve the vacuum conditions and prolongs the service life of the ion source.

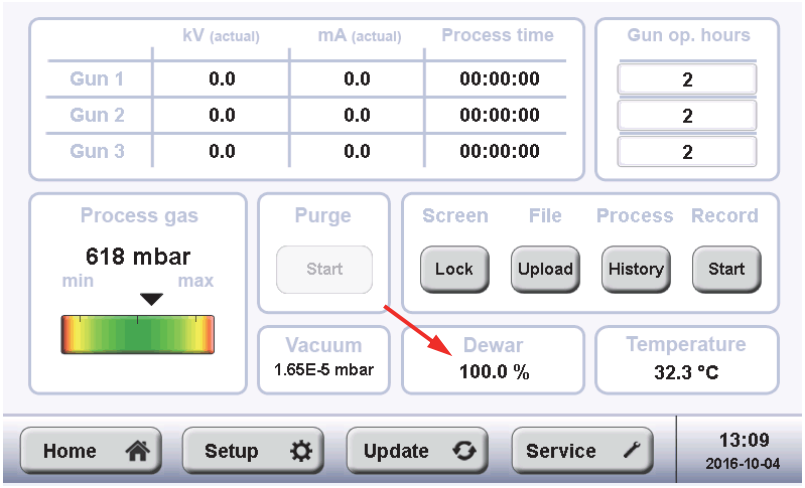
4.12 Unloading the sample

To unload the sample, proceed as follows:

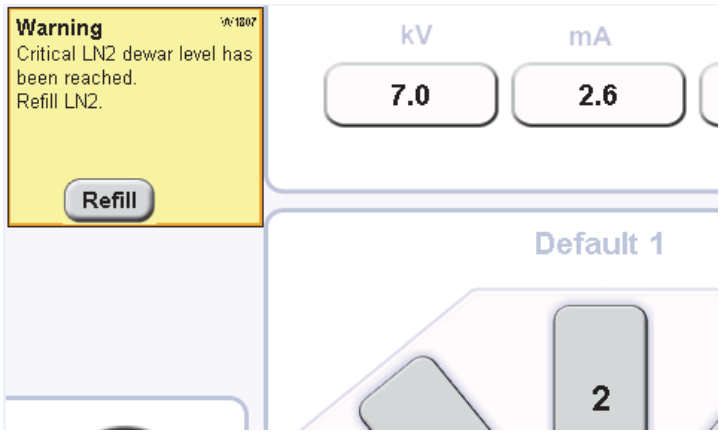
1. Vent the system.
2. Pull the table flange out.
3. Move the sample holder away from the mask to avoid damaging the sample when you pull the sample holder out (slightly retract sample using the set screw).
4. Pull the sample holder up and out of the system.
5. If necessary, detach the sample from the sample holder for subsequent examination.
6. If you are not going to load a new sample immediately, close the table flange and pump the system.

4.13 Exchanging the Dewar

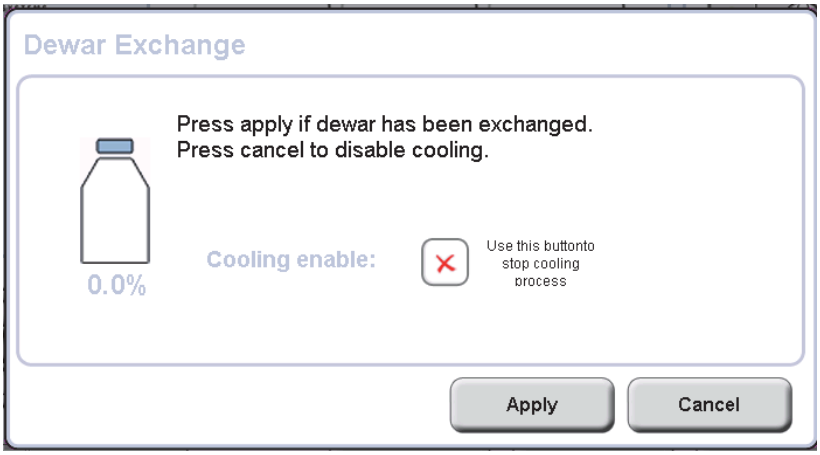
The Dewar level is indicated in the menu:



Warning messages will appear as soon the LN_2 Dewar becomes empty:

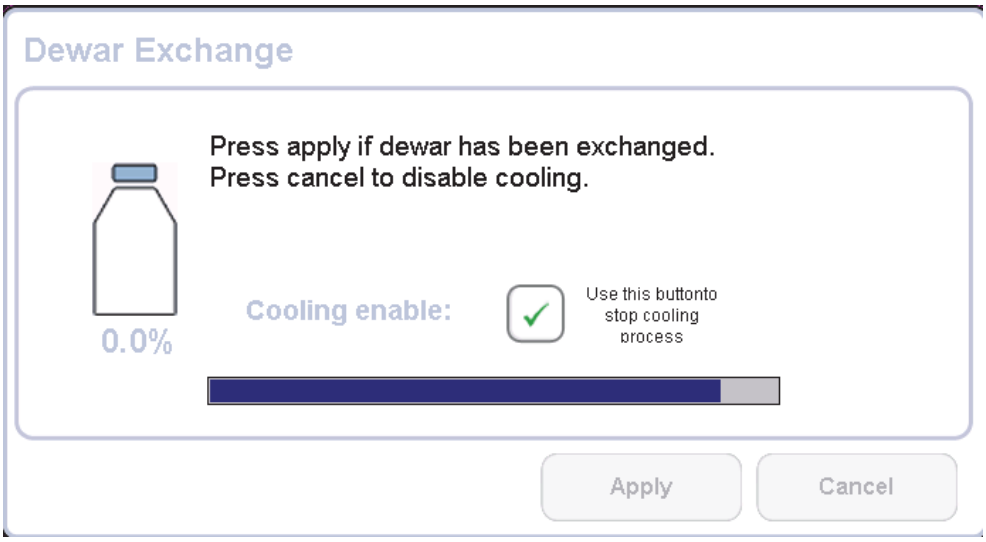


Push the Refill button to stop the cooling process prior exchanging or refilling the Dewar.



For exchanging the Dewar position a second full Dewar close to the empty Dewar. Carefully remove the LN_2 pump and insert it carefully in the full Dewar. Watch the frozen stiff LN_2 tube to prevent it from braking. This process should be done uninterrupted, otherwise the valves of the LN_2 pump could freeze. In this case the complete pumping system needs to be warmed up by a blow-dryer at ~40 °C.

As soon the Dewar has been exchanged or refilled push the Apply button and enable the cooling process.



5. Maintenance and Service

5.1 Maintenance of the Cooling Stage

5.1.1 Safety measures during maintenance and service

Caution!
Danger may be caused by servicing and cleaning the system incorrectly.

This may cause personal injury and damage to the system.

Maintenance operations on the Leica EM TIC 3X Triple Ion Beam Cutter system must only be performed by specially trained technicians or a Leica-trained service engineer!

5.1.2 General instructions for maintenance and cleaning

There is danger from external elements (dust, dirt, etc.).

Contamination, particularly of the interior (pressurized) surfaces, may cause the vacuum system to malfunction.

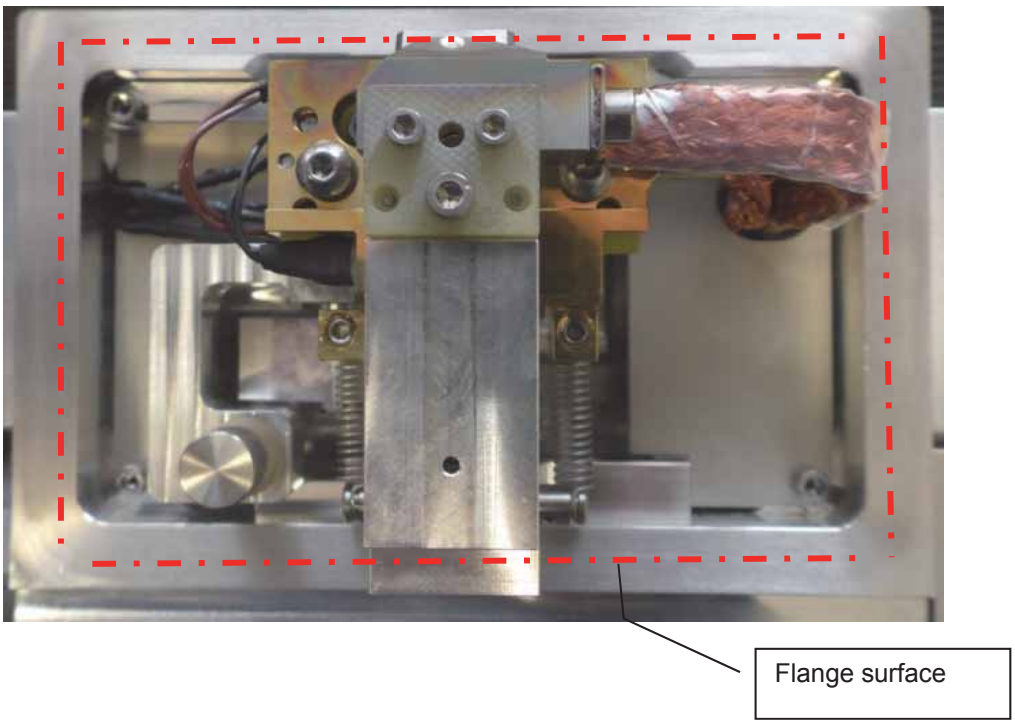
When working on the interior of the Leica EM TIC 3X Triple Ion Beam Cutter system, it is essential to observe the principles of vacuum hygiene. Gloves must be worn when disassembling and assembling components in the vacuum area, and also for all adjustment work.

All work must be carried out in a clean, dust-free environment.

5.1.3 Cleaning the Cooling Stage

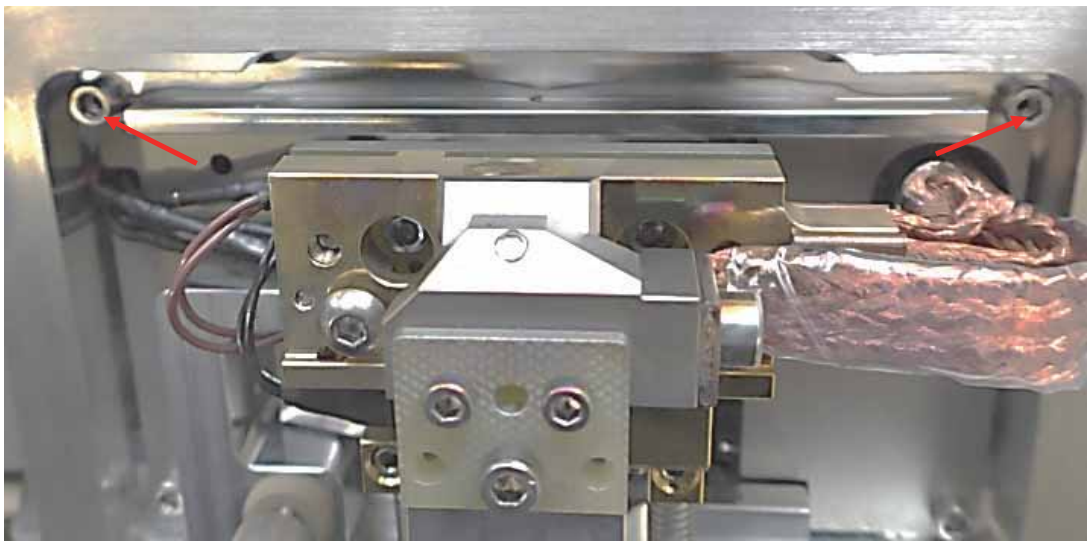
As deposition of the sputtered material will be noticeable after several sample processes, the stage should be cleaned using oil-free compressed air. For this cleaning step the stage has to be removed from the instrument and carefully apply an oil free airstream to remove loose particles. Avoid blowing the particles inside the stage.

Make sure the flange surface is clean and not contaminated, otherwise vacuum can't be achieved. Clean this surface with isopropanol.

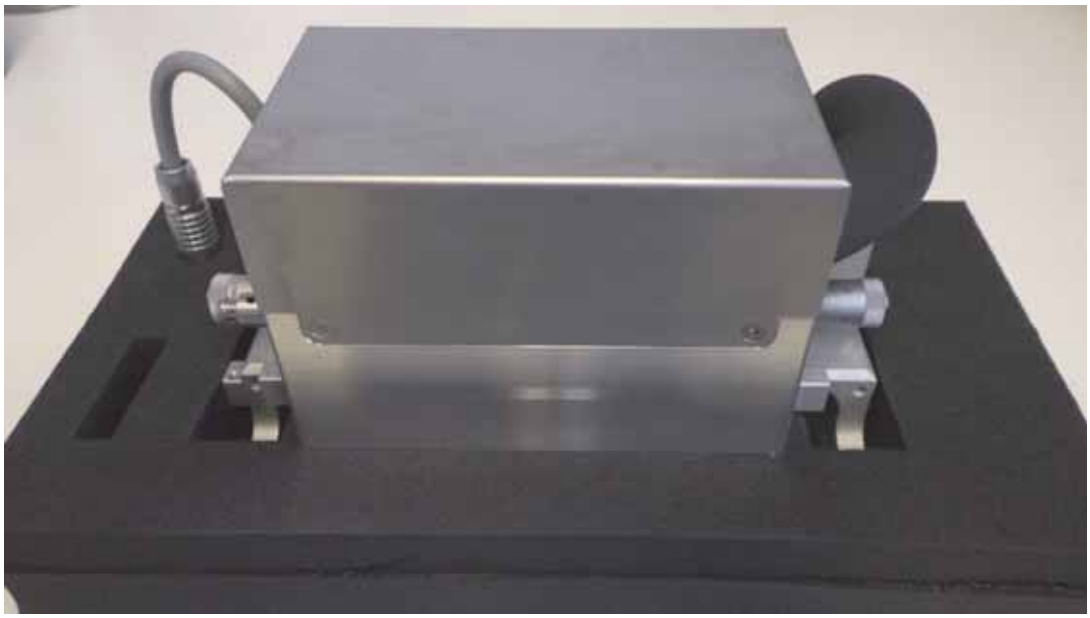


5.1.4 Ion beam protection and storage

The stage is equipped with an ion beam protection shield to avoid damage of the flange. This protective part has to be replaced by removing the two hex keys as soon as it becomes eroded to such an extent that deep indentations are visible.



For storage, use the foam storage insert delivered with the instrument (it is a part of the packaging).



5.1.5 Replacing the mask

Assembly	Task	Cycle
Mask	Replace	Every 10hrs

The mask is susceptible to wear because of its exposure to the ion beam, and must therefore be replaced periodically. The degree of wear depends on the process energy used and the process duration. The quality of the mask (straight, sharp, clean mask edge with no unevenness) is very important for the quality of the cutting process.

Therefore, the mask should be inspected before every cutting process. If any dirt or deposits are on the mask, they must be wiped off with acetone.

Caution!

There is danger from the sharp edges of the mask.

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

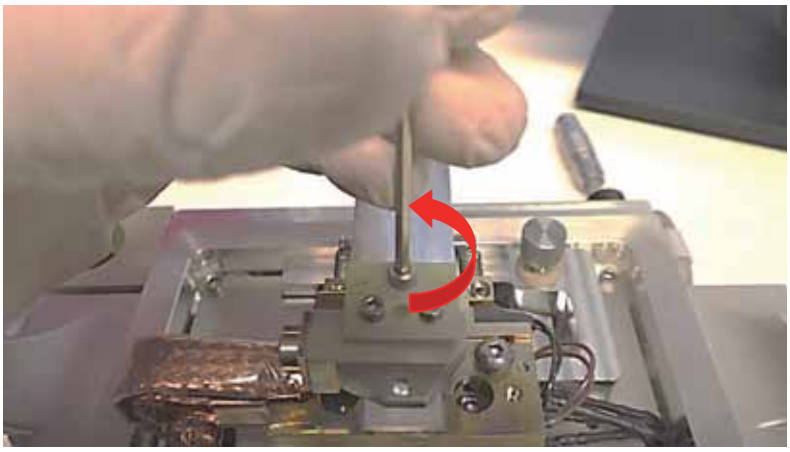
Be careful when working on the mask!

The mask should be replaced when:

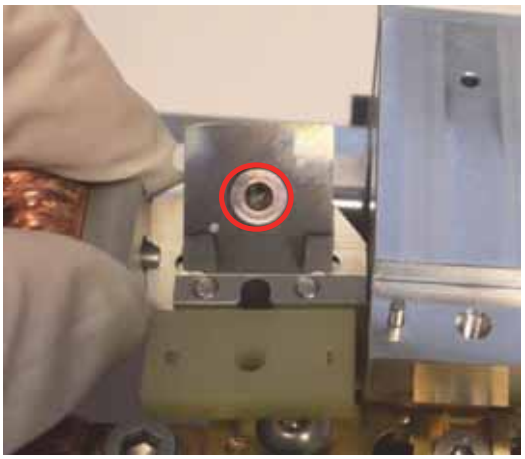
- dirt and deposits on it cannot be removed,
- the mask edge becomes rounded,
- there are unevenness, nicks, cracks in the mask edge,
- the cutting process causes concave wear of the mask edge,
- the mask edge is no longer parallel to the mask mounting
- each mask has 4 edges and can be used four times by rotating it through 90°

It must be ensured that the mask edge is parallel to the mask mounting. This is ensured at installation by the shape of the mask holder and the counter-sunk screw.

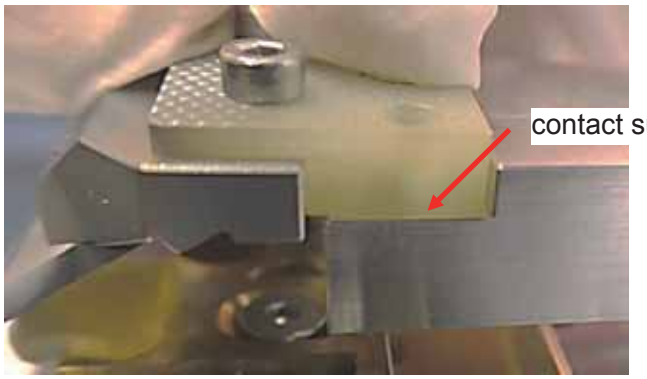
1. Remove mask holder with a hex key, size 2.5.



2. Detach the mask with a hex key, size 2.0 and take the mask off. Turn the mask 90° (fresh mask side) or insert a new mask. Mount the mask onto its holder. Make sure that the lower mask edge rests onto its support on both sides.



3. Mount the mask holder onto the cooling stage, make sure that the isolating part firmly contacts the counterpart. Fasten the mask holder with a 2.5 Allen key.



5.1.6 Maintenance of the valves of LN_2 pump

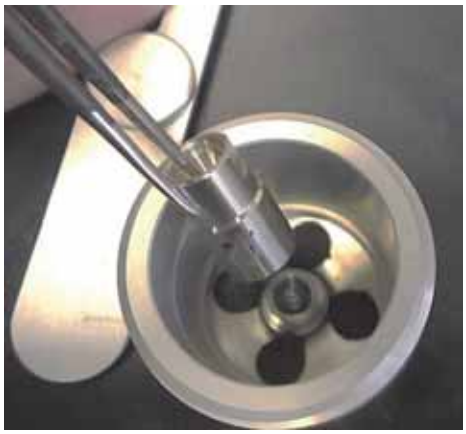
Assembly	Task	Cycle
LN_2 Pump	Clean valves	- if cooling rate becomes too slow - if low temperatures can be achieved

Humidity and particles in the valves can cause freezing and sticking of the valves during operation. To prevent this the valves of the LN_2 pump can be easily removed and cleaned.

1. Removing the upper valve

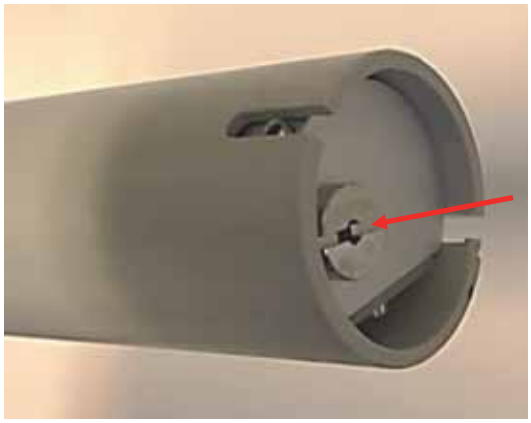
   Open the shutter by pushing the lever and remove valve housing using a 8 mm flat spanner.
   Attention: the ball is freely moveable and can easily drop out. Keep the pump in upright position.

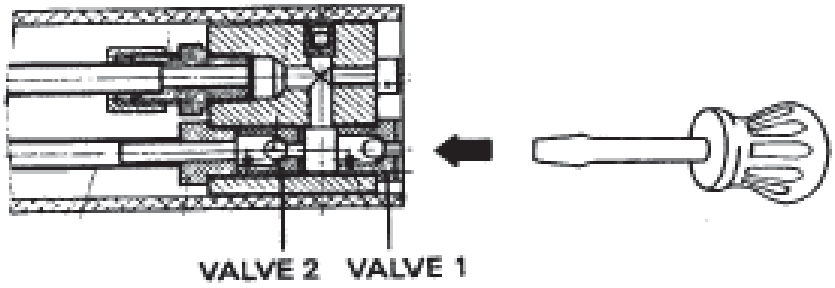
   Remove the valve housing and ball with tweezers.



2. Removing the lower valves

   Remove both valves with an appropriate screw driver. The second steel valve is located underneath the first valve.





3. Clean the valves with isopropanol in an ultrasonic bath and let them dry after the cleaning procedure.

upper valve

lower valves

4. Before inserting the lower valves, check that the ball inside the valves is freely moving.

5.2 Servicing and repair by Customer Service

The Leica EM TIC 3X Cooling Stage is covered by a WARRANTY in accordance with 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.

Caution!
There is danger due to overloaded or defective components.
This may cause personal injury and damage to the system.
The environmental conditions that were agreed contractually and determined at the time of installation must be maintained.

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. If in doubt, please contact our Customer Service department.

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Leica Mikrosysteme GmbH | Vienna, Austria
T +43 1 486 8050-0 | F +43 1 486 8050-30

www.leica-microsystems.com