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Issued by:

Leica Mikrosysteme GmbH
Hernalser Hauptstrasse 219
A-1170 Vienna
Leica EM TIC 3X Rotary 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 Rotary 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 Rotary 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 Technical Documentation.

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

In order to ensure the safety of service technicians and operators, and to prevent any damage to the Leica EM TIC 3X Rotary 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).**

**WARNING**

Burn hazard. Hot surface inside. Allow to cool before servicing the ion source.

**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 Rotary Stage

#### 1.1.2 Name and address of the manufacturer

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2. **Product description**

2.1 **Field of application and proper use**

The **Leica EM TIC 3X Rotary Stage** is used for precise processing of samples for subsequent examination with an electron microscope (EM) or light microscope (LM). For this purpose, an ion beam polished area or a cross section (optional item) 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.

Ion beam polishing is used to improve a mechanically or chemically polished surface of the sample e.g. to remove fine scratches, abrasive material and smearing artefacts. **To achieve the highest surface quality using ion beam polishing it is mandatory to pre-prepare the sample surface to a high quality e.g. mechanically polishing with fine grain size (<0.5 \( \mu \)m) of the abrasive material.**

The sample thickness should not exceed 12 mm. Furthermore the sample should be parallel within 0.5 mm in order to prepare a uniform surface area.

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 to change.

The **Leica EM TIC 3X Rotary Stage** must not be used beyond the limits specified in the chapter “Limitation”.

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. Motor for rotation or oscillation
2. Rest for sample holder
3. Set screw for setting the ion beam incident angle
4. Rotation sensor
5. Clamping screw for sample holder
6. Guiding mechanism for lateral movement
2.3 Safety information

2.3.1 General instructions

Danger!
The Leica EM TIC 3X Rotary 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 Rotary 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 not be operated except by trained and authorized personal.
- Repairs may only be made by authorized staff of Leica representatives.
- If the Leica EM TIC 3X Rotary 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 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 CO₂ fire extinguisher must be used.

Technical Service must be consulted before resuming work with the system.
3. Installation and setup

3.1 Transportation and storage conditions

The Leica EM TIC 3X Rotary 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 at temperatures between >15°C < 30°C <80% RH (no condensation). 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 Rotary 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.
4. Operating instructions

4.1 Introduction

The ion beam polishing 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 positioned on one of the holders and inserted in the rotary stage. An ion beam incident angle can be set from 0° - 48° (depending on the sample size → see Limitation). To achieve a uniform polished surface the sample can be rotated or oscillated. For large area polishing a lateral movement of the sample can be activated.

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.
3. 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!

4. Slide the preventer until the bearing is completely visible.
5. Move the complete stage to the left.

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

7. 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.
8. Insert the other stage in the vertical position.

9. Hinge the stage on its two bearings.
10. Slide the preventer to cover the bearing completely before you make any other connections.

11. Swivel the stage into its horizontal position, slightly retract the flap damper and connect to hinge point.
12. Connect the plug of the stage.

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

*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. Initialization is performed in the setup menu.*

*Initialization must be performed without a sample inserted in the rotary stage! During the initialization process an automatic lateral movement of the holder mechanics is made to its limit switch (12.5 mm). Hence an already inserted large sample can collide with the ion source mechanics and damage to the holder mechanics may occur.*

### 4.3 Specifications for ion beam polishing

- Max. sample diameter: 38 mm
- Max. Ion beam prepared area: Ø 25 mm
- Max. sample height: 12 mm
- Lateral movement: +/- 12.5 mm
- Lateral speed: 0.1 to 2 mm/sec
- Incident angle: 0° to 48° (1.5° increments)
- Rotation speed: Low (~4 rpm), Medium (~7 rpm), High (~10 rpm)
- Oscillation: 20°, 45°, 90°, 180°, 360°
4.4 Limitation

To perform ion beam polishing of large areas a lateral movement of the sample up to +/- 12.5 mm can be selected. Due to the size of the vacuum chamber and ion gun design of the Leica EM TIC 3X the range of this movement is limited by the sample size.

Sample size: Lateral movement range:

Ø 38 mm +/- 3 mm
Ø 35 mm +/- 5 mm
Ø 33 mm +/- 6 mm
Ø 30 mm +/- 8 mm
Ø 28 mm +/- 9.5 mm
Ø 25 mm +/- 11.5 mm
<Ø 20 mm +/- 12.5 mm

The ion beam incident angle can be set from 0° - 48°. An angle between 6° - 15° is used for ion beam polishing. Higher angle settings are applied if contrast enhancement should be achieved. Due to the dimensions of the vacuum chamber the range incident angle range depends on the sample size.

Sample size: Incident angle range:

Ø 38 mm 0° - 12°
Ø 35 mm 0° - 13°
Ø 33 mm 0° - 14°
Ø 30 mm 0° - 15°
Ø 28 mm 0° - 16°
Ø 25 mm 0° - 18°
Ø 23 mm 0° - 36°
<Ø 20 mm 0° - 48°

Setting the lateral movement and incident angle parameters beyond these limits can damage the mechanics of the rotary stage.

High voltage (>8 kV) and long etching time (>10 h) settings may affect the performance of the motor for rotation. Long term processes (>10 h) with high energies (>8 keV) lead to high heat impact on sample and sample stage, which may affect the rotation speed. Hence, the rotary stage should be cooled down at least for 30 minutes before continuing with the same settings. A cooling step can be automatically performed by using the program function in the load-up menu (see chapter 4.11)
4.5 Sample holders of the Rotary Stage (ion beam polishing)

Several sample holders are available and delivered with the rotary stage:

- for sample thickness of 0 – 3 mm
- for sample thickness of 3 – 6 mm
- for sample thickness of 6 – 9 mm
- for sample thickness of 9 – 12 mm
- for SEM stub with Ø 3.1 mm and sample thickness of 0 – 3 mm
- for SEM stub with Ø3.1 mm and sample thickness of 3 – 6 mm

Note: SEM stub holders are especially useful in conjunction with the pre-preparation instrument Leica EM TXP. The sample can remain on the SEM stub from the mechanically polishing step (Leica EM TXP) via ion beam polishing (Leica EM TIC 3X) to eventually coating and finally SEM examination.
4.6 Mounting the sample onto the holder of the Rotary Stage

Choose the holder suitable for the sample and its thickness.

The sample holder diameter is 20 mm. To position the sample close to the rotation center, concentric ring marks on the holder are used for orientation. If the sample is larger than Ø 20 mm the holder should be orientated using forceps in the following manner:

Place double-sided sticky copper tape onto the bottom side of the sample. Note: when rotating the sample this surface should be parallel within 0.5 mm to the polished side of the sample.

Take the holder with forceps, center and fix the holder onto the sample.
4.7 Adjusting the sample to the eucentric height

Use the loading tool and insert the intermediate ring of the sample holder. Insert the ring with the thread in front of you.

Clamp this ring with the clamping screw.

Insert the sample holder with the sample. The flat side of the sample holder should be orientated to the thread.
By turning the adjustment screw the sample height can be set in line with the rim of the loading tool.

Clamp the sample holder using the Allen key.

Remove the sample with the intermediate ring by opening the clamping screw.
4.8 Inserting the sample in the Rotary Stage

After venting and opening the stage flange insert the sample in the holder of the rotary stage so the flat portion of the intermediate ring shows in the direction of the clamping screw. If necessary open the clamping screw.

Make sure the sample is located correctly in the holder mechanics.

Clamp the sample holder using the Allen key.
4.9 Setting the incident angle and lateral movement parameter

The incident angle is set using the adjustment knob on the left side of the rotary stage.

Set the incident angle in accordance to the limitation of the sample size.

For ion beam polishing, values between 3° and 15° are most commonly set. The angle setting can be observed in the upright position of the stage.
Once the incident angle is set, close the rotary stage flange and set the lateral movement, if necessary to perform uniform sample preparation on large area of interest.

For doing so push the rotary stage symbol to open the Load-up menu.

Load-up menu, push Stage button in the Manage – Steps menu and select **Standard** holder for ion beam polishing stage settings.

Check if the origin is correctly set in the center of the rotation by observing the apex of the holder rest which should be coincident with the center mark of the lateral movement mechanics. For better observation use the stereo microscope.
Push the arrow button in order to move the sample holder in desired direction. As soon the center has been reached push the 0 button to set the zero position.

Push the right arrow button as long as the sample is close to the right ion gun. Read the X shift value. Set this distance for the lateral movement of the sample during the process using the + or - button.

Select speed of the lateral movement and sample rotation (disable oscillation). Push the test button to check the complete rotation and travel range making sure that the sample not collide with the ion source mechanics.

Now the sample can be processed. Please refer to the manual of the Leica EM TIC 3X for further information regarding gun parameter setting.

Due to the high quality of mechanical pre-preparation (polishing) the sample process time usually takes about 30 to 60 minutes, depending on voltage setting.

4.10 Preparing sample for oscillation process

Sometimes rectangular samples or samples with preferred directions do not need to be rotated during the process. An oscillating function can be used for such samples.
To orientate the sample for oscillation please proceed as follows:

The holder should be mounted onto the sample in such a manner that the flat portion of the holder is orientated perpendicular to the area of the sample which needs to be prepared at an oscillation angle.

The portion of the sample nearer to the ion beam gun is opposite to the flat portion of the holder.

Set the eucentric height with the loading tool as mentioned above.

Insert the sample into the rotary stage and clamp it with the Allen key.

Enter the Rotary Stage configuration menu by pushing the load-up button and set the oscillating angle (20°, 45°, 90°, 180° or 360°). Usually, an angle setting of 45° or 90° is used.
An offset of the origin of the lateral movement can be performed (e.g. for accurate positioning a sample detail).

In order to set the area of interest in the cross over of the ion beams proceed like following:

1) Set the cross hair of the eyepiece reticle to the center mark of the lateral movement mechanics.
2) Move the sample detail in desired direction using the arrow button (sample detail coincident with the cross hair).
3) Push the 0 button to set origin.

Select speed of the lateral movement and sample oscillation (enable oscillation). Push the test button to check the complete oscillation and travel range making sure that the sample not collide with the ion source mechanics and the area of interest is set in the ion beam path.

Now the sample can be processed. Please refer to the manual of the Leica EM TIC 3X for further information regarding gun parameter setting.

Due to the high quality of mechanical pre-preparation (polishing) the sample process time usually takes about 30 to 60 minutes, depending on voltage setting.

### 4.11 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 selected step line.

![Image of step line with options](image1)

**Delete - : the selected step can be removed**

*By selecting a low kV or even 0 kV a cooling step can be inserted to avoid heavy influence by heat impact.*

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 program is active for the next sample process, even when changing the parameters on the main screen. This change will affect the first step only. The instrument will continue according to the programme.

![Image of step line with options](image2)

To delete the program 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.
4.12 Holder for cross sectioning (optional item)

The Leica Cross Sectioning holder (CS holder) for the Leica EM TIC 3X Rotary Stage is used for precise processing of samples for subsequent examination with an electron microscope (EM) or light microscope (LM). For this purpose, an ion beam cross sectioned area of a sample is created with the Leica EM TIC 3X Triple Ion Beam Cutter system in conjunction with the Rotary Stage.

Ion beam cross sectioning is used for roughly pre-prepared samples which subsequently will be inserted and aligned in the Cross Sectioning Holder of the Rotary Stage.

For detailed information please refer to the manual of the cross sectioning holder.
5. Maintenance and service

5.1 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!

5.2 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.

5.3 Cleaning the Rotary 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. Carefully apply an oil free airstream to remove loose particles. Avoid blowing the particles inside the stage.
Make sure that the flange surface is clean and not contaminated, otherwise vacuum can’t be achieved. Clean this surface with isopropanol only.

5.4 Ion beam protection and storage

The stage is equipped with three ion beam protection parts to avoid damage of the flange surface. These protective parts have to be replaced as soon as they become 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). When inserting the stage please set the incident angle above 30°.

5.5. Servicing and repair by Customer Service

The Leica EM TIC 3X Rotary 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.