

# **Modular X-Ray Source**

# MOXS

# **Technical Manual**



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#### 1 Introduction

The modular X-ray source with optics iMOXS is a device for generating X-rays. It is possible to couple it with X-ray optics and create for this reason focused and parallelised beams. Due to the modular design and the possibility to combine it with X-ray optics, the possible fields of application are diversified. They reach from exciting X-ray fluorescence with small spots over the realisation of stray experiment to the usage in radiography. Due to its compact construction it is possible to use it in both laboratory and industrial environments.

iMOXS consists of the following modules: low-power X-ray tube, tube protective housing, revolver with interchangeable filters, shutter and high voltage and supply unit with necessary safety circuits for the operation of the X-ray source. The optics is fixed in a special mounting which is adjustable to the X-ray tube. According the request for delivery, the tubes can be supplied with different target materials. Their maximal operation parameters are 50kV and 30W (depending on target material used).

The Control and Supply Unit (CSU) is a standalone device which is operated in the local mode manually by keys and rotary knobs located on its front or correspondingly in the remote mode by a controlling PC via UPS serial interface. The built-in high voltage generator provides up to 50 kV of positive polarity for the connection with the anode of an X-ray tube. Additionally, it contains a regulated supply for the cathode filament heating current. The regulated tube current may be continuously varied from 0 up to the maximum of 800  $\mu$ A. Further on, the device is equipped with different safety loops according the requirements of the German X-ray regulation (RöV). A Windows based software with a simple user interface is a part of the package.

To operate the iMOXS by qualified personnel according the radiation protection instructions in a safe modus the modular X-ray source is already equipped with the required safety functions. They comprise for instance a shutter system, absorption filters as well as internal and external signal lamps.

The modular X-ray source can be used as a standalone device and or can be integrated in complex set-ups.

iMOXS is applied in different configurations, e.g. as iMOXS-SEM, a module for exciting X-ray fluorescence by radiation inside a scanning electron microscope or for diffractometry. In this manual these applications will be shortly described with their features after the general description.

# 2 Delivery and Installation

#### 2.1 Technical Data

# X-Ray Data:

High voltage: max. 50 kV,

adjustable in steps of 1 kV

Tube current: max. 800 μA,

adjustable in steps of 1 µA

Filament Current: max. 5 A

(depending on tube type used)

# X-Ray Tube:

Type: air-cooled metal-ceramic tube with shutter and filter revolver Anode material: Cr, Co, Cu, Mo, Rh, Ag, W

(or other on demand)

Anode spot size: 50  $\mu$ m X-Ray window: 0.1 mm, Be Tube temperature:  $\leq 60$  °C

# X-Ray Control and Supply Unit (CSU):

Height: 150 mm
Width (incl. handles) 360 mm
Depth: 300 mm
Weight: ca. 8 kg

#### **Electrical Connection Parameters:**

Voltage: 90...230 V, 50 Hz Power: max. 150 W

Connection: Safety Plug 1P+N+PE, DIN 49441

## **Optics Module (if present):**

Mounting:

Type: Polycapillary Focusing Optics

Focal spot size  $< 100\mu m$  (at E > 15 kV)

Polycapillary Parallelising Optics
Optics socket with adjustment unit

# X-Ray Source without CSU and Optics:

Height / Width / Depth: 335mm / 130mm / 250mm

Weight: approx. 5.5kg

#### **Attachment Gear (if present):**

Flange: Adapting flange to SEM probe chamber

(depending on the SEM type)

Support: Tube housing support with linear

movement stage and X-Y adjustment

#### 2 Delivery and Installation

## **Software:**

XCtrl: Control of the modular X-ray source,

Adjustment of the operation parameters by

PC via USB interface

iMOXS-Quant: Quantitative XRF-Analysis (with and

(if ordered) without standards)

**Operating Conditions:** 

Temperature range: 10 - 30 °C Recommended room 20 - 25 °C

temperature:

Relative air moisture: 10 - 80 %, non-condensing free of corrosive vapours, free of heavy dust load

# **Miscellaneous:**

In relation to the process of continuous development IfG – Institute for Scientific Instruments GmbH reserves the right to change specification without previous notice at any time.

# 2.2 Scope of Delivery

The delivery package of the CSU - Control and Supply Unit comprises the following parts:

- control and supply unit with HV generator and power supply,
- mains connection cable,
- USB serial cable for PC connection,
- Interlock cable (safety circuit),
- hardware user manual,
- tube control software on CD,
- adjustment target.

The delivery package of the modular X-ray source iMOXS includes:

- Protective tube housing with electromechanical shutter system and filter revolver,
- air-cooled metal-ceramic X-ray tube with Be window,
- HV cable for the X-ray tube,
- filament current cable for the X-ray tube,
- control cable for the tube housing (safety functions),
- absorption filter (depends on the tube type and configuration),
- X-ray tube data sheet,
- copy of type approval certificate.

If the X-source is provided with optics, an optical unit will be delivered which is composed of:

- Focusing or parallelising polycapillary optics in a mounting,
- · adjustment unit,

as well as in the case of iMOXS-SEM:

- adapting flange in accordance with the REM type (CamScan, Hitachi, JEOL, Tescan, ZEISS etc.),
- adjustable X-ray source support.

In both cases a certificate of the installed polycapillary optics is enclosed.

# 2.3 Transport and Installation

# 2.3.1 Transport

The CSU - X-ray control and supply unit is delivered in a transport box, which also contains the accessories belonging to the package ordered.

The X-ray source is delivered in a second transport box. Further accessories are packed in both boxes.

The transport requires utmost care. During transport and storage, the equipment must be protected against moisture and temperatures below -10°C.

The total gross weight of each of the two transport boxes being below 25 kg, the equipment can be lifted and carried by one person.

#### 2.3.2 Installation

iMOXS is a laboratory equipment intended for operation in closed rooms with controlled temperature.

#### Attention!

While unpacking and installing the iMOXS never hold the mounted source at the screwed-in optics unit then because it results in deadjustment!

A simple mains outlet without special fuse protection is necessary. No water or other cooling media are required, with the exception of special requirements of the detector used.

#### Attention!

The legal regulations for operating X-ray sources must be strictly observed!

#### 2.3.3 Connections

The electric connections of the CSU such as connection to the protective tube housing, mains connection, signal connections are all arranged at the rear side. Special care has been taken to make the plugs confusion-safe. At the tube protective housing, all connections are arranged on one side independently of their being supply, control or signal connections.

# **3 Connections, Control and Display Elements**

# 3.1 Front Panel of the X-Ray Control and Supply Unit (CSU)



Front panel of the Control and Supply Unit (CSU)

The following elements are arranged at the upper left part of the front panel (see Figure 1):

- Display of High Voltage (two-digit, in kV)......(kV)
- To its right, rotary knob for high voltage setting
- Display of tube current (four-digit, in μA) ......(μA)
- To its right, rotary knob for tube current setting

In the first row below the display of tube current there are the following elements (from left to right):

PEN indicator	"Shutter OPEN" - red
ton shutter on/off	. "Shutter ON/OFF" - red
N indicator	"X-RAY ON" - yellow
ton High Voltage on	
ton High Voltage off	

In the second row below the display of tube current there are the following elements (from left to right):

"Interlock" - green/red	• LED indicator for safety interlock circuit
	• LED indicator for state of readiness
"rem" – green	• LED indicator for remote (PC) control
"loc" – blue	• LED indicator for local (manual) control
"Remote" – green	• push button for remote (PC) control
"Local" – blue	• push button for local (manual) control
ON/OFF	• mains key switch

The cooling air inlet with fan is located on the right side.

# 3.2 Rear Panel of the X-Ray Control and Supply Unit (CSU)



Figure 2
Rear Panel of Control and Supply Unit (CSU)

All the signal and control inputs and outputs as well as the cooling air outlet are located on the rear side of the Control and Supply Unit (see Figure 2):

The following elements are located from top left to right bottom:

- connector for External Safety Interlock circuit "Interlock" (Sub-D 9-pole plug socket)
- connector for external manual handling **"Interface"** (customer option) (Sub-D 25-pole plug socket)
- output for External X-ray ON signal lamp "24 V DC"
- Filament connector for tube cathode heating "Filament"
- USB-connector for PC-remote control "USB"
- connector for control leads for Tube Housing **"Tube Housing"** (Sub-D 15-pole, 3-rows, plug socket)
- High Voltage output for X-ray tube "High Voltage"
- Mains connector and mains fuse "90 260 VAC"

## **Attention!**

To prevent overheating, never cover the front and rear side of the control and supply unit. Free air circulation must be permanently secured.

#### **Attention!**

Prior to disconnecting the high voltage cable, be sure to switch off the high voltage and set the power master switch to **OFF**. It is recommended to disconnect the mains cable as well.

# 3.3 Modular X-Ray Source iMOXS

The modular X-ray source iMOXS (see Figure 3) consists of the low-power microfocus X-ray tube, which is enclosed in a tube housing with a shutter and a filter revolver.

The X-ray beam emitted by the tube is collimated and focused by the integrated X-ray optics. It is important to remember that iMOXS is delivered with a preadjusted optics module. Only in exceptional cases it may be necessary to perform slight corrections of the optics adjustment after transportation of the module (see section 4.3. below – "Adjustment of X-ray Source").

The filter wheel is a disk with mounted filters and located inside the protective tube housing. The filter revolver can be equipped with different filter materials according to the customer requirements and choice of the anode material. It is possible to integrate up to 5 filter materials. As a rule, the medial position of the wheel stays without filter. At the bottom of the adjustment unit of the optics the contained filters are indicated. The manual lever to displace the filters is also located there. It displays to the actual used filter and is used to turn the disk simultaneously.

Furthermore, at iMOXS-SEM the adapting flange is equipped with a pressure sensor which is connected to the safety interlock system.

Two LED indicators are located on the tube protective housing: the **yellow** one indicates that the high voltage is on and the **red** one points out that the shutter is open.

#### **Attention!**

When both the **yellow** and the **red** LED are switched on, X-radiation is emitted through the optics exit! X-radiation may cause severe health hazards when impinging on parts of your body!

The shutter position is controlled by micro-switches. In the case of malfunction (interrupted or shorted circuit) it is impossible to open the shutter.

The protective tube housing complies with the German radiation safety regulations. The local dosage rate does not exceed 25  $\mu Sv/h$  at a distance of 500 mm from the module.

#### 3. Connections, Control and Display Elements



**Figure 3** Modular X-ray Source iMOXS

# 3.4 Optics Module (if present)

The optics module contains the capillary optics in a metal casing, the optics socket with adjustment screws to align the optics relative the anode spot of the tube. The optics can be easily exchanged by unscrewing it from the optics socket.

Depending on the problem under consideration, different optical elements can be installed in the module:

- $_{\odot}\,$  cylindrical monocapillaries with an inner diameter from 10  $\mu m$  to 1 mm and with a different length
- $_{\odot}\,$  elliptical monocapillaries with a focal spot size less than 5  $\mu m$  diameter and a focal distance from 1 to 5 mm
- polycapillary semi-lenses providing a quasi-parallel exit beam with a diameter of 5 to 7 mm
- focusing polycapillary optics with a variable distance between the anode spot and a sample surface providing a micro spot down to 50μm

For XRF and  $\mu$ XRF analytical applications the iMOXS is normally equipped with a focusing polycapillary optics.

The X-ray source can be operated by means of the X-ray control and supply unit (CSU) either locally (manually) or remotely (by a PC or a notebook via a USB serial interface).

The control program for operating the CSU is a simple exe-file. It is necessary only to copy it to the correspondent folder. Before using the CSU for the first time it is essential to install the required drivers.

The individual functions and their operation are described below in this section.

# 4.1 Switching-on the CSU, State of Readiness

Before switching-on the CSU you have to pay attention that all desired and required plug connectors are properly connected.

The high voltage cable and the filament current cable are permanently fixed to the tube housing and should be connected to the corresponding connectors on the rear side of the CSU. The 'Sub-D 15 pole' socket "Tube Housing" on the rear side of the CSU must be connected with the 'Sub-D 15-pole' plug on the tube housing by means of filament current cable.

The activating of the device will be made by the **mains key switch** "**ON/OFF**" downright at the front panel. It is possible only to remove the key from switched-off device. The usage of a mains key switch should guarantee that the CSU could be switched on by entitled persons only. After that the yellow push button "**HV ON**" should be actuated, the LED indicator for the state of readiness "**HV-DC**" shines green then.

After switching on the CSU is in the **state of readiness** (ready for operation). This state is characterized by the following:

- the operating mode "Remote" is active (green LED),
- the operating mode "Remote" will be noticed on the tube current display,
- the elapsed operating hours will be indicated, e.g. **H 0.2**
- all target values and displays indicate the value "0" (zero),
- the high voltage is switched off,
- the shutter is closed,
- the LED safety interlock indicator "Interlock" sends a signal about the actual state of the safety interlock circuit: green - Interlock closed, red - Interlock open.

#### 4.1.1 Installation of the Drivers

To be able to install the drivers, both the PC and the CSU have to be switched on and the USB cable connection between both devices has to be established.

#### **Attention**

The installation of the drivers requires having the administrator rights for the user.

The PC shows a message that it has found a new hardware USB <-> RG A and requests for installation.

Choose the option "Installing software from a list or selected source" and click "Continue".



Then the source for the driver has to be chosen; the delivered CD will be used as source. On the CD, via "Search", the folder USB\_ifg\_DRIVER has to be chosen, click "Continue" then.



Then you have to push the button "Continue installation" (see window "Hardware installation" below).



The software will be installed....



If the installation was carried out please push the button "Complete" to finish the procedure.



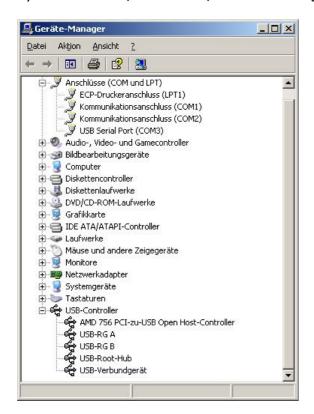
In the course of installation (second step) the message "New hardware found" will be displayed again. Now, the same procedure as described above has to be performed until the installation for USB – RG B is completed.



Further steps like described above. In the course of installation (third step) the message "New hardware found: USB Serial Port" will be displayed. Now, the same procedure described above has to be performed for the installation of the USB- Serial Port.



The correct installation can be controlled via the device manager (start, settings, system control, hardware, device manager).



The entries USB-RG A, USB-RG B (USB-Controller) and USB Serial Port (Connections COM and LPT) have to be listed there.

It is necessary to keep in mind the number of the USB serial port (COM #).

To check if this number (COM #) coincides with the corresponding entry in the file CONFIG.dat in the directory of the RG- control program, the file has to be opened with a text editor. The entry in the last row (13) must be corrected if necessary.

#### 4.1.2 User Interface and Features

To operate the CSU by means of a special Windows software, a plain user interface will be provided. Its functionality corresponds to that of the manual operation as far as possible. Additionally, it contains special supervision features, like e.g. time control for the warm-up mode of the X-ray tube with phased intervals for its conditioning.

After starting the program, a control window will be displayed (see figure 4). The actual values of high voltage and current will be displayed in red digits (immediately after starting both values must be zero).

The only activated control element is the push button "Device initialisation" then which has to be clicked by the user. After that, a dialogue window will be displayed with the question: "The last warm-up was evidently 1 day 1 hour 00 minutes ago. Would you like to make automatic warm-up?"

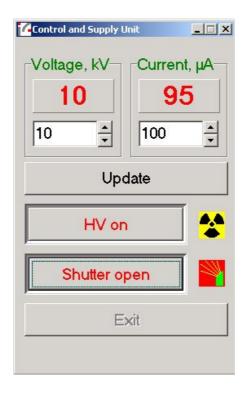


Figure 4
Control window

The user has the possibility to answer "Yes" or "No":

- 1. In the first case (Yes) the automatic warm-up procedure starts. Depending on the length of the time interval since the last warm-up the proper mode for raising high voltage und current will be chosen by the program. If the break of operation exceeds 8 weeks, the tube will be handled by the program as a newly installed unit and the complete warm-up procedure will be performed (possible duration 1.5 - 2 hours). The current operation will be described in the status bar at the bottom of the window. The actual values of high voltage and current will be displayed in red digits always. The nominal ("must") values will be displayed in the fields next to the see-saw-controller on the left. It is possible to interrupt the automatic warm-up procedure any time by actuating the push button "Cancel warm-up". After finishing the procedure completely and successfully, a corresponding entry row will be added to the log file by the program. If the log file does not exist it will be generated at first in the same directory the program was installed in. Afterwards, the push button will be deactivated and further control elements will be activated. Now the X-ray tube is ready for operation. Changing the operating conditions requires the specification of the MUST-values either by entering the values directly or by actuating the arrow buttons of the see-saw-controller with a following <Return> or a mouse click on the push button "Update".
- 2. In the second case ("No") the push button "Device initialisation" will be deactivated instantly and other control elements will be activated. The tube can now be operated by means of changing the high voltage and current MUST-values, either by entering the values directly or by actuating the arrow buttons of the see-saw-controller with a following <Return> or a mouse click on the push button "Update". Thus, the user can go directly to the tube operating mode.

After clicking on the push button "HV on" the actual values will be changed gradually to the nominal MUST-values and will reach them with some time delay. Simultaneously, on the right of the push button a radiation sign will be displayed. This process can be controlled visually by comparison of ACTUAL – and MUST-values in the control window. By clicking on the push button "Shutter" the shutter will be opened and on the right to it a sign will appear indicating the emission of X-radiation from the optics now. Actuating the shutter button once again will close the shutter.

To finish the tube operation, the shutter has to be closed. After then the high voltage has to be switched-off – however, it is necessary to wait until the actual values are zero.

After actuating the push button "Exit" the control window will be closed.

#### **Attention!**

1. We recommend always using the automatic warm-up procedure for the tube if you switch-on the tube and intend to operate it. Normally, the whole procedure does not take longer than 5-10 minutes if the tube was not in operation for 1-2 days.

2. The programmed automatic procedure simplifies the required warm-up procedure essentially. However, the program is not able to react adequately for all unexpected problems. For example, due to particularities of the tube or of the high voltage generator the current sometimes does not reach the displayed MUST-value or shows great fluctuation. In such an exceptional case the user has to halt the automatic process and to further operate the tube manually.

#### **Attention!**

After fitting of a new tube, the log file containing the data of the old tube has to be deleted or renamed! Only then the program can recognize the new tube as a newly fitted unit and perform the automatic warm-up procedure.

# 4.1.3 Error messages

Error No.	Description of Error
1	Controller program error
2	Controller function error
3	Safety relay malfunction (relay 2)
4	Safety relay malfunction (relay 4)
5	Safety relay malfunction (relay 3)
6	Malfunction of shutter switch or door switch
7	Door switch malfunction
8	Malfunction of shutter switch
9	Malfunction of shutter-LED at the tube housing
10	Malfunction of HV-LED at the tube housing
11	Malfunction of shutter-LED at CSU
12	Malfunction of HV-LED at CSU
13	Malfunction of additional lamp indicator

Several safety interlock circuits are integrated in the device. The device displays an error message if one of these circuits is broken.

If an error occurs, the shutter, high voltage and the state of readiness will be switched-off. This safety precaution ensures that in the case of a malfunction of the device it can only be put into operation again by a <u>conscious</u>, active switching-on.

To eliminate the error, the user must switch off the CSU by means of the mains key switch and unlink it from the supply voltage as a consequence.

# 4.1.4 Shift Remote / Local

The switch of the CSU control between remote operation via PC (USB connection) and manual operation (local) is effected by pushing the green button "Remote" left down at the front panel. After switching-on the mains supply, the CSU is in the state of readiness. The active mode of operation will be indicated by the green shining LED indicator "Remote".

In remote modus the state of readiness will be made by actuating the yellow push button "HV ON" resulting in the message "PC" at the display on the front panel. The device can be operated in the remote mode via the USB interface only. However, with the black push button "HV OFF" the high voltage can be switched off <u>at the device (!)</u> at any time. The LED indicators "X-ray ON" and "Shutter OPEN" show the current state of the device.

After actuating the blue push button "Local" the device will shift to the local (manual) mode. Now, the high voltage will be switched off, the shutter will be closed, the MUST-values are set to "0" and the state of readiness is deactivated. The LED indicator "Remote" is going out and the LED indicator "Local" shines blue now. For manual control the device is in the state of readiness after actuating the yellow push button "HV ON".

# 4.2 Manual Operation

# 4.2.1 Conditioning a new X-Ray Tube

X-ray tubes are vacuum electronic devices in which electrons are accelerated in a high voltage field to produce X-rays. The tube vacuum is subject to variations due to degassing and other processes. The slight long-term pressure increase observed in tubes, which are not in use cannot be fully compensated for by the getter material installed in most tubes.

Therefore, a new X-ray tube has to be conditioned with the utmost caution by a slow gradual increase of the high voltage according to the following instructions.

- To begin with, the tube has to be operated <u>without</u> cathode heating (no anode current).
- The high voltage should be increased according to the following scheme:

0 to 50%	of the maximum voltage in steps of 5 kV every 30 seconds
51% to 85%	of the maximum voltage in steps of 10 kV every 10 minutes
86% to 100%	of the maximum voltage in steps of 5 kV every 10 minutes

The tube should run at least 10 minutes at the maximum voltage.

- In case of a breakdown, conditioning should be resumed from the previous step and carried out according to the present scheme until the tube runs at the maximum voltage without breakdown.
- As soon as the tube runs smoothly at the maximum voltage, the high voltage should be reduced to 50% of its maximum value and the current should be increased to its corresponding maximum value for this voltage according to the recommendations of the manufacturer (see also data sheet of the tube used).
- Subsequently the high voltage should be increased in steps of 5 kV up to the maximum value with the 2 minutes duration at each step. At each step the anode current should be maintained at its corresponding maximum value for this voltage according to the recommendations of the manufacturer (see the table below exemplary for a Rh-anode!).

HV, kV									
I <sub>A</sub> , mA	0.6	0.7	0.8	0.68	0.55	0.4	0.3	0.2	0.1

In the manual mode the operator is personally responsible for performing the conditioning procedure of a new X-ray tube or an X-ray tube with a downtime of more than 8 weeks!

In the computer assisted mode (remote mode), optional automatic conditioning can be performed (see section 4.1.2).

# 4.2.2 Manual reconditioning of used X-ray tubes after an operating pause

**Attention:** An X-ray tube should be conditioned after its transportation or when its period of non-use is more than 8 weeks!

The X-ray tube may be conditioned also manually in dependence on its actual downtime:

- 50% of the maximum voltage may be applied immediately
- The anode current should be increased to its corresponding maximum value according to the recommendations of the manufacturer (see also data sheet of the tube in operation).
- Subsequently the high voltage should be increased according to the following scheme (at each step the anode current should be adjusted to its corresponding maximum value according to the recommendations of the manufacturer):

Downtime of the tube	1 - 2	2 - 8	1 - 2	2 days	2 – 8
	hours	hours	days	_	weeks
				2	
				weeks	
Increase of the HV in steps					
of 10 kV within intervals of:	3	10	30	1	2
	seconds	seconds	seconds	minute	minutes

# 4.2.3 Setting Nominal (MUST) Values for Voltage and Current

The nominal value of the high voltage can be manually set by the **[kV]** rotary knob on the front panel in steps of 1 kV up to the maximum value of 50 kV (depends on the X-ray tube used).

The nominal value of the tube current can be manually set by the **[\mu A]** rotary knob on the front panel in steps of 1  $\mu A$  or 10 $\mu A$  up to the maximum value of 800  $\mu A$  (depends on the X-ray tube used).

As long as the high voltage is switched off, the nominal values of the high voltage and tube current are displayed by the two displays on the front panel. Clockwise (respectively counter clockwise) rotation increases (respectively decreases) the regulated values.

#### Attention!

Be sure not to exceed the maximum values of the high voltage, current and power authorized for the given tube type when operating the X-ray tube.

## 4.2.4 Switching On High Voltage

After setting the required ("must") values, the high voltage will be switched on automatically. Simultaneously, the two displays for high voltage "kV" and tube current "µA" switch from preset to actual (instantaneous) values.

A requirement for switching on the high voltage is a closed interlock loop, indicated by the green **Interlock** LED. If the interlock loop is open, the **Interlock** LED is red. High voltage cannot be switched on before the interlock loop is closed once again. If the control and supply unit is used for some other purpose without external safety switches and without tube housing, the sockets "tube housing" and "interlock" on the control and supply unit rear panel should be closed using the corresponding dummy plugs.

After pressing the "HV ON" button, the yellow signal field "X-Ray ON" lights up. The current flow through the signal elements is continuously monitored. In the case of a defect (power interruption or short circuit) of the signal elements, the high voltage cannot be switched on, or would instantly be switched off in the moment the defect occurred.

At first, the high voltage is increased gradually up to the preset value by an internal ramp function. Subsequently, the tube current is increased also to its preset value. Due to the internal control function of the control and supply unit, this process can take several seconds (2...5 s).

The actual values of high voltage and tube current can be regulated manually in the "X-Ray ON" mode. Both control displays ("kV" and " $\mu A$ ") always show the instant actual values.

When the yellow signal field "X-Ray ON" lights up, the direct voltage of 24 V (maximum permissible power 5 W) is passed on to the exit "24V DC" (External X-ray ON Lamp). This exit is intended for an optional signal lamp that can indicate the actual state of the "X-ray ON" control at some distance away from the tube housing.

#### Attention!

With the high voltage switched on, the X-ray tube emits ionising radiation! Without adequate protective measures it can cause serious health damage to the personnel in the room!

#### **Attention!**

As a rule, the high voltage should be switched on only when the X-ray tube is connected with the control and supply unit. As an exception, the control and supply unit may be used for other purposes with the tube housing and with the interlock circuits shorted by dummy plugs. In this case, operating the control and supply unit becomes potentially dangerous because all its safety functions are put out of action!

#### **Attention!**

A new X-ray tube, as well as a tube that was out of operation for some time, must be conditioned. This procedure includes a slow, gradual increase of the high voltage (see sections 4.1.2 and 4.1.3).

## 4.2.5 Switching Off High Voltage

The high voltage can be switched off manually by the black "HV OFF" push button. Simultaneously the current is switched off and the shutter will be closed if it was open.

The yellow "X-Ray ON" indicator extinguishes announcing the state "OFF". The auxiliary 24 V DC voltage at the exit "24V DC" (External X-ray ON Lamp) on the CSU rear panel is switched off.

# 4.2.6 Open/Close Shutter

The shutter at the tube housing can be opened by pressing the red **"Shutter"** button on the front panel of the CSU, both at switched-on and switched-off high voltage.

When the shutter is opened, the red indicator "Shutter OPEN" lights up. The current through the signal elements is continuously monitored. In the case of an error of the signal elements (power interruption or short circuit), the shutter cannot be opened or is closed in the moment when the error occurs.

Hitting the red "Shutter" push button once more closes the shutter at the tube housing. The signal indicator "Shutter OPEN" extinguishes.

# 4.2.7 Filter Changer

The filter changer is a disk wheel with mounted X-ray filters which are put up in the tube housing. The placement of filters on it (filter choice) depends on the material of the anode used and the special wishes of the customer. It is possible to install up to 4 different materials at the wheel. As a rule, one position on it stays without filter.

It is possible to read off the built-in filter materials at the base of the optics unit. There is located the lever to shift the filter also. The lever points to the actual filter and serves simultaneously for turning the wheel.

# 4.2.8 Switching Off the X-ray Source

The CSU will be switched off by actuating the **mains key switch** "**ON/OFF**". However, before switching off the mains supply the shutter should be closed and the high voltage should be shut down to zero.

It is not advisable to switch off the mains supply "hot" with running X-ray tube because abrupt changes of the high voltage may have an adverse effect on the life cycle of the X-ray tube.

Also, before switching off it is recommended not to disconnect the cables of the tube protective housing and the tube to the CSU to discharge the high voltage to the ground safely.

The shutoff of the mains supply means a **total reset** for the CSU, meaning that for instance all nominal (must) values are cancelled.

After switching on the mains supply again, the CSU is in the state of readiness (see 4.1).

# 4.3 Alignment of the Optics

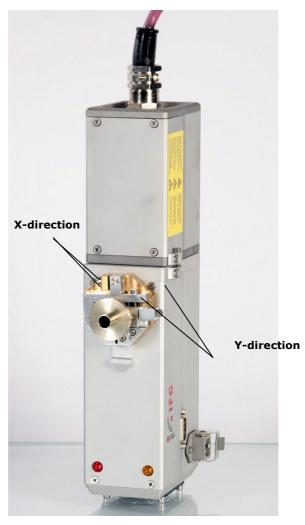
Prior to delivery, the optics unit attached to the tube housing is pre-aligned in such a way that its first focus matches the anode spot of the X-ray tube. As a rule, no additional adjustment of the optics is required.

When (as an exception) it is necessary to adjust the optics relative to the anode spot, one should observe the following instructions:

The focal distance optics-anode (Z axis) is <u>firmly</u> fixed by the optics holder. The optics unit is equipped with a built-in adjustment device which allows an alignment of the optics in X- and Y-direction by using the two corresponding screws which are fixed by a locknut (see figure 5). The alignment consists in measuring the fluorescence signal from a homogeneous sample (pure Cu) while the optics is moved relative to the X-ray source and finding the position with a maximum count rate.

#### **Attention!**

In contrast to the above-mentioned procedures, any other changes at or inside the tube housing as well as at the cooling element may lead to optics misalignments, which can be properly corrected by the manufacturer only! The consequences of such changes are not covered by the IfG guarantee!



**Figure 5**Modular X-ray source iMOXS

# 4.4 Alignment of iMOXS-SEM (if present)

The source module is flange mounted to a SEM. The source is mounted on a special support including adjustment device (see figures 6 and 7), which allows the fine adjustment of the X-ray beam in X and Y direction by screws to match it with the electron beam spot on the SEM sample so that the regions for XRF and EPMA coincide. The support includes also a manually adjustable linear stage **2** for retraction of the entire source with the optics from the vicinity of the sample in the SEM. A vacuum-sealed connection to the coupling flange of the SEM is realized via the flange adaptor, custom-built for different types of SEM according to the customer's order.

The high voltage cable and the filament current cable are permanently fixed to the tube housing and should be connected to the corresponding connectors on the rear side of the CSU. The 'Sub-D 15 pole' socket "Tube Housing" on the rear side of the CSU must be connected with the 'Sub-D 15-pole' plug on the tube housing by means of filament current cable.

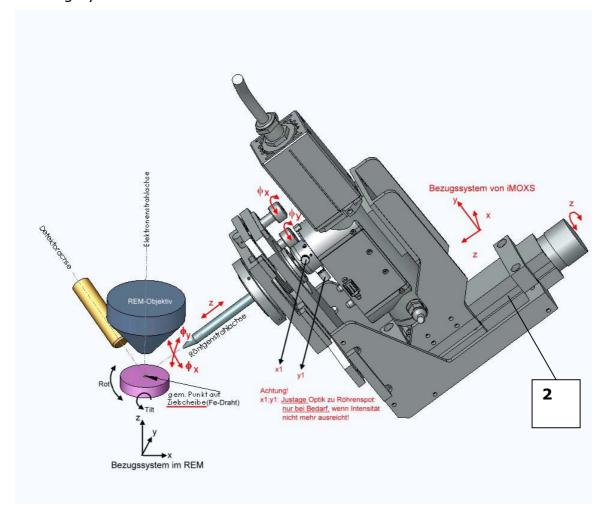


Figure 6

Schematic representation of the possibilities for iMOXS adjustments

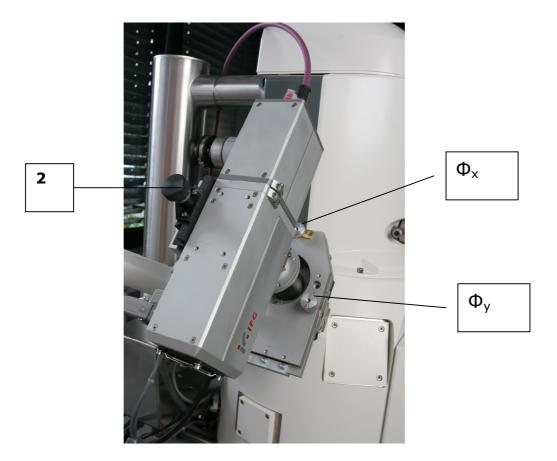


Figure 7

iMOXS adjustments at SEM

# 4.4.1 Adjustment of the X-Ray Spot relative to the Electron Beam Spot

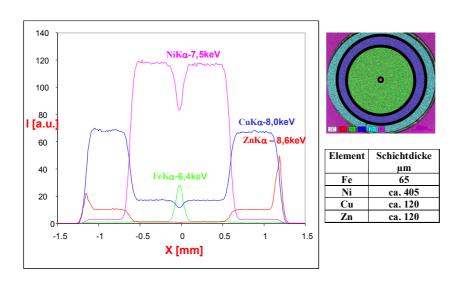
The aim of this adjustment is to guarantee that excitation of the fluorescence radiation with an X-ray beam takes place exactly on the same spot where the nondeflected electron beam hits a sample.

A special target is provided to make the fine adjustment of the iMOXS easier. This target is a disk with concentric rings made from different elements (Figure 8). The target is fixed on an aluminium substrate. The following elements are arranged on the target (from the edge towards the centre): zinc, copper, nickel and in the middle an iron wire with the diameter of 65  $\mu$ m. In this sequence, the energy of excited characteristic radiation decreases when the X-ray spot of the primary beam is shifted towards the centre of the target, i.e. positions of the fluorescence lines in the spectrum move to the left. The target should be fixed on a sample holder. Then the sample holder should be placed into its normal operational position under the electron beam.

For preparing the adjustment procedure it is important that the target is positioned at the proper working distance in the SEM. This distance should be put on record. Besides, the non-deflected electron beam should hit the centre of the target. The adjustment procedure can be controlled with a secondary electron image or by means of ESMA analysis.

The target has to be positioned under the electron beam in such a way that the iron core is focused with an enlargement of about 3000 in the middle of the SE-picture.

When the positioning of the target is accomplished, the electron beam is switched off and the target is excited with the X-ray beam. Further adjustment consists in finding the maximum intensity of the Fe K-line with the help of the screws  $\Phi_{\mathbf{x}}$  and  $\Phi_{\mathbf{y}}$  (see Figure 6).



**Figure 8**Target for adjustment of the spot position with distribution of elements (LineScan)

# 4.4.2 Adjustment of the optics focal distance relative to the sample surface

The procedure described in section 4.4.1 guarantees that the X-ray beam and the electron beam meet in the centre of the target when the target is at the proper working distance. At the next step the optics focal distance should be adjusted (see Figure 8). This adjustment can be performed by moving the X-ray source along the optical axis with the help of the linear stage **Z** (see Figure 6) or **2** (see Figure 7).

At first the intensity of the Fe K-line should be measured and put on record. Then the source should be shifted in 1 mm steps towards and from the target (attention: the source must not bump into the SEM equipment). At every step the source position is adjusted to the maximum intensity of the Fe Ka-line or to the largest (Fe Ka / Ni Ka) - ratio respectively through its x-y-shifts with the help of  $\Phi$  and  $\Phi$  screws and the measured intensity of the Fe-line is put on record.

The source position with the highest intensity of the Fe Ka-line corresponds to the proper focal distance of the capillary optics.

It is recommended to perform a fine adjustment with the step of  $\pm$  0.5 mm in the vicinity of the position with the maximum intensity of the Fe-line.

When the adjustment of the focal distance is accomplished, one should repeat the procedure of section 4.4.1 in order to confirm that the measurement spot has not changed.

The optimal position of the source should be marked on the stage and fixed with a stopper.

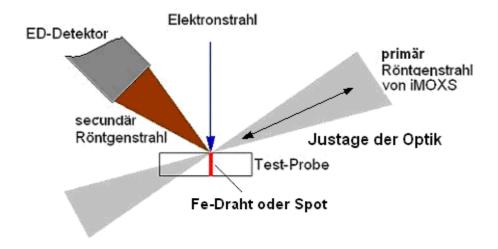


Figure 8

Schema for adjustment of the exciting X-rays in SEM

#### **Attention!**

In contrast to the above-mentioned procedures, any other changes at or inside the tube housing as well as at the cooling element may lead to optics misalignments, which can be properly corrected only by the manufacturer! The consequences of such changes are not covered by the IfG guarantee!

#### **Remark:**

As an alternative to the adjustment target, one can use also a thin wire on a substrate (e.g. quartz or wax) for adjustment the optics focal distance.

# 4.4.3 Checks of adjustment stability at the user

The intensity of the Cu Ka-line from a homogeneous sample made of pure Cu gives a good idea of the total alignment of the iMOXS. Therefore such tests should be repeated regularly. It is recommended to measure the fluorescence signal from a Cu sample once a week under the same conditions, i.e. the same temperature of the device, the same position of the sample on the table, the same position of the table, etc. The measured intensities of Cu Ka-signal should be put on record (e.g. inserted into an Excel spreadsheet and graphically presented as a trend chart).

The results obtained show if the proper alignment of the iMOXS remains valid or if some adjustments are necessary.

# **5 Function of Safety Circuits**

# 5.1 External Safety Interlock

By means of an external safety interlock, hereinafter: "interlock", such features like e.g. switch off of the high voltage during ventilating the sample chamber of a SEM, contacts at radiation protection shields or doors, thermal circuit breaker, proximity switches, emergency stop switch or similar can be involved in a safety concept to assure radiation protection.

On the rear panel of the control and supply unit CSU unit there is a 9-pole plug socket "Interlock". Using it an interlock is realized in connection with a SEM or a similar application (vacuum), respectively. For this reason, the plug socket is connected with a vacuum sensor which is installed at the SEM adapter flange of the iMOXS source.

If the operator attempts to ventilate the SEM without previous closing of the shutter during activated high voltage, then the high voltage is automatically switched off and the shutter is closed by the CSU. However, the CSU stands further in the state of readiness: the adjusted values of voltage and current remain stored. To work with iMOXS (release of the interlock) becomes possible only if the SEM is evacuated again (door closed).

To prevent the high voltage switch-off during the ventilation of the SEM, the shutter has to be closed beforehand. In this case the high voltage remains switched on. An attempt to open the shutter in the ventilated state of the SEM leads to an instant interlocking of the mode of operation of the device: the high voltage will be switched off and the shutter remains closed. To work with iMOXS (release of the interlock) becomes possible only if the SEM is evacuated again (door closed).

But if the device functions are locked <u>and</u> in addition error messages are displayed at the CSU, the CSU has to be switched-off completely by means of the mains key switch, the cause of fault has to be identified and to be cleared. Only by this the state of readiness of the CSU can be re-established.

#### Attention!

Please avoid any contact of the external interlock with ground. This may destroy the interlock circuit in the CSU.

# 5.2 Description of the safety and supervision features

With a maximal permissible current of  $800\mu A$ , the X-ray tube will generate X-radiation up to an energy of 50keV. However, it is permitted to operate the tube with a maximal power of 30W only.

All necessary constructional measures were met in order to avoid any hazard of the service personnel by X-rays.

There are two direct connection cables to the X-ray tube, the coaxial high-voltage cable to "HV" and the cathode current supply "filament". Additionally, all security-relevant connections and signals are led to the tube protection housing over the cable "tube housing".

Before start-up of the iMOXS it is necessary to consider the operational stipulations (implementation of the legal X-ray regulations) of the radiation protection officer (see also chapter 6).

The safety precautions comprise the following safety circuits:

- A locking mechanism directly in front of the X-ray tube, the so-called shutter, which is controlled by two independent micro switches.
- Two current monitored indicators: one for the activity of the X-ray tube "X-Ray ON", the other for the opened shutter "Shutter OPEN", both at the front panel of the CSU
- Two current monitored LED indicators (red, yellow) at the tube protection housing: the yellow one signalises the state "X-Ray ON", the red one the state "Shutter OPEN".

The safety systems are linked with each other in multiple ways:

- It is possible to switch on the high voltage for the X-ray tube only if the internal and external interlock circuits are closed.
- There is the LED indicator "X-RAY ON" at the CSU front panel whose current is supervised. If it failed, no sufficient warning of the personnel would be given and for this reason the high voltage of the tube would be switched off immediately or could not be switched on, respectively.
- Furthermore, there are two indicators "Shutter OPEN" and "HV ON", both at the CSU front panel and at the tube protection housing whose currents are supervised, too. If only one of them failed, no sufficient warning of the personnel would be given. For this reason the shutter would be closed then or it would be impossible to open it, respectively.

It is only possible to switch on (again) the high voltage of the tube if all indicators are fully functional (again) and the safety circuits are closed.

# **6 Radiation Protection and Operational Approval**

iMOXS is an X-ray radiator which is type-approved by Bundesamt für Strahlenschutz (German X-ray protection authority): Approval sheet No. BfS 06/07 R RöV (German X-ray regulation) from February 28, 2007), PTB (Physikalisch-Technische Bundesanstalt / German National Metrology Institute) - Test report-No. 6.32-T241 from February 09, 2007. This means in particular that the local dose rate in a distance from 1.0 m of the accessible surface amounts to less than 3  $\mu Sv/h$ .

The initial operation of iMOXS is allowed only after examination of the entire plant by an expert. The initial operation of iMOXS has to be reported to the relevant administrative body (according to §4 Abs.1 Nr.1 RöV) including the required documents (see §4 Abs. 2 RöV) two weeks in advance at the latest. Among other things, an inspection report of an expert has to be presented and a radiation protection officer has to be appointed who has the requisite qualification. The users who supervise and self-dependently conduct the operation of the radiator must have the requisite qualification according RöV and the corresponding qualification guide line.

Please have in mind that the mentioned regulations are valid for Germany only. You need to consider the national safety regulations applicable for your country. Operating errors, e.g. an opening of the shutter with missing sample or direct presence of persons at the tube protective housing with opened shutter, create potential health hazard by radiation damage!

During the construction and implementation of the security concept special importance was attached to the prevention of danger for the health of the service personnel. Thus, all safety functions operate independently of attached connected PC and possible program errors. The wiring of the iMOXS source and its safety circuits was made in such a way that a rough program error or a program crash at the PC does not impair security. All safety circuits work wirebound.