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Operating Instructions

Freeze Fracture System BAF 060

BU 800 284 BE 7. Edition: 6.2003

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Inspect shipment for possible damage and notify shipper if necessary.

Compare shipment with shipping papers and notify supplier of possibly missing pieces.

Save packing material. If possible, device should be shipped in the original packing for inspection or repair.

This manual is valid from Serial-No. 742 on.

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Mount electron beam gun 2 (for Carbon coating) onto manipulator.

Press key GUN LOCK 1 (MDC 010):

- LED of key goes out.

Press key GUN LOCK 2 (MDC 010):

- LED of key GUN LOCK 2 lights up.
- Gun holder moves into loading position for gun 2.

The subsequent procedure for gun 2 follows the above description for gun 1.

4.4. Carrying out a Trial Coating

It is advisable to always carry out a trial coating prior to a freeze-etching process in order to check that the evaporation device functions correctly. In doing so, it is absolutely essential to protect the table receptor from being coated by inserting the specimen table (see section 4.2.2.).

The following requirements must be fulfilled prior to the trial coating:

Change lever AUTOMATIC/MANUAL must be in position AUTOMATIC.

The gun holder must be in its basic position (C 90°/Pt-C 45°).

Should it be in LOCK-position (LED of key GUN LOCK 1 or 2 of the MDC 010 lights green), press the respective key once. Subsequently continue to press key GUN ELEVATION SELECT until the indication GUN 1 45 deg respectively GUN 2 90 deg appears on display.

Shutter must be in position ETCH.

Press key STEP of the MDC 010-control unit. Indicate ETCH appears on display.

4.4.1. Trial coating with evaporation source GUN 1 (Pt-C)

For the operation of the control unit for the electron beam evaporation device, also refer to the EVM 030-operating manual, BU 800 205 BD, section 2.

Select the program BAF 060 with program selection in set-up menu.

- Measure 1 appears on display.
- With the arrow key the saved parameters could be shown.

Set mains switch POWER of the EVM 030-control unit to ON.

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Press key EVAPORATION GUN 1:

- LED of key flashes.
- Electron beam gun for Pt-C has been selected.

Set potentiometer VOLTAGE to 1.6kV:

- Indication of the acceleration voltage on display.

Set potentiometer EMISSION to 60mA:

- Indication of the emission current on display.

Press key HIGH VOLTAGE ON:

- LED of key lights green.
- LED of key EVAPORATION GUN 1 lights permanently.
- Voltage and emission current rise to the set values.

Press key Measure 1 on the QSG 100:

- Actual film thickness (0) and coating rate appears on display.
- Shutter moves into position GUN 1 (display MDC 010).

If an indication of an evaporation rate of approx. 0.1 nm/s and an increase in the film-thickness appear on the display of the QSG 100 and if no major arcing at the evaporation source can be observed, the evaporation source GUN 1 is ready for a coating process.

Press key HIGH VOLTAGE OFF:

- LED of key sights red.
- LED of key GUN 1 flashes.

4.4.2. Trial Coating with Evaporation Source GUN 2 (Carbon)

Interrupt the measuring of Layer 1 with keys Stop and OK on QSG 100.

- Layer 2 is activated on QSG 100. The saved parameters appear on display.
- Measure 2 appears on display.
- Shutter moves into position SHUT (display MDC 010).

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Set mains switch POWER of the EVM 030-control unit to ON.

Press key EVAPORATION GUN 2:

- LED of key flashes.
- The electron beam gun for carbon is selected.

Set potentiometer VOLTAGE to 1.9kV:

- Indication of the acceleration voltage on display.

Set potentiometer EMISSION to 90mA:

- Indication of the emission current on display.

Press key HIGH VOLTAGE ON:

- LED of key lights green.
- LED of key EVAPORATION GUN 2 lights permanently.
- Voltage and emission current rise to the set values.

Press key Measure 2 on the QSG 100:

- Actual film thickness (0) and coating rate appears on display.
- Shutter moves into position GUN 2 (display MDC 010).

If an indication of an evaporation rate of >0.5nm/s and an increase in the film-thickness appear on the display of the QSG 100 and if no major arcing at the evaporation source can be observed, the evaporation source GUN 2 is ready for a coating process.

Press key HIGH VOLTAGE OFF:

- LED of key sights red.
- LED of key GUN 2 flashes.
- Interrupt the measuring of Layer 2 with keys Stop and OK on QSG 100.

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It is therefore important to stick closely to the sample temperature and etching time in order to achieve a desired and reproducible etching depth.

During the etching process the subsequent coating of the samples must be prepared as follows:

- Move knife holder into the up most position, using key KNIFE ADVANCE (MDC 010).
- Check evaporation values (EVM 030) and, if necessary set them:
  GUN 1 1.6kV 60mA for Platinum-Carbon
  GUN 2 1.9kV 90mA for Carbon
  (slight deviations are possible)

Check whether program BAF 060 and Layer 1 (Measure 1 respectively) is indicated on the display of QSG 100.

Once the etching time has elapsed, start coating according to section 5.7.

5.7 Coating of specimens

Prior to coating the specimens, the change lever of the cutting system must be in position 'AUTOMATIC'.

Press key EVAPORATION GUN 1 (Platinum-Carbon) on the EVM 030:

- LED of key flashes.
- Pre-selected values for VOLTAGE and EMISSION appear on display.

Press key HIGH VOLTAGE ON:

- LED of key HIGH VOLTAGE OFF goes out.
- LED of key HIGH VOLTAGE ON lights green.
- LED of key EVAPORATION GUN 1 continues to light up.

Press key Measure 1 on QSG 100.

- Measuring of Layer 1 has been activated.
- Shutter automatically moves into position GUN 1 (should the shutter move into position GUN2, a correction according to section 5.8. is necessary).

When the pre-selected layer thickness for Platinum-Carbon has been reached:

- High voltage of the EVM 030 switches off automatically.
- QSG 100 automatically switches to Layer 2 (Measure 2).
- Shutter in the unit moves into position SHUT (indication on MDC 010-display).

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Press key EVAPORATION GUN 2 (Carbon) on the EVM 030:

- LED of key flashes.
- Pre-selected values for VOLTAGE and EMISSION appear on display.

Press key HIGH VOLTAGE ON:

- LED of key HIGH VOLTAGE OFF goes out.
- LED of key HIGH VOLTAGE ON lights green.
- LED of key EVAPORATION GUN 2 continues to light up.

Press key Measure 2 on QSG 100.

- Measuring of Layer 2 has been activated.
- Shutter automatically moves into position GUN 2.

When the pre-selected layer thickness for Carbon has been reached:

- High voltage of the EVM 030 switches off automatically.
- Indication GUN2 on the MDC 010 goes out.
- QSG 100 automatically switches back to Layer 1 (Measure 1).
- Shutter in the unit remains in the same position.

5.8. Possibly Necessary Correction of Shutter Position

Should the shutter move into position GUN 2 instead of GUN1, the QSG 100 film thickness monitor and the MDC 010-control unit are not synchronised, so that the following corrections are necessary:

Press keys Stop and OK on QSG 100.

- The shutter remains in the same position.
- The indication GUN 2 on the MDC 010 goes out.
- Layer 2 (Measure 2) is indicated on QSG 100.

Press the arrow key once. The QSG 100 changes to Layer 1 (Measure 1).

- Layer 1 (Measure 1) is indicated on QSG 100 display.

Wit the key Measure 1 the correct measuring could be continued.

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A loss of synchronisation between QSG 100 and MDC 010 results if:

- During the normal operation of BAF 060 the arrow key on the QSG 100 display has been pressed.

5.9. Removing the Specimen Table

When the samples have been coated, the specimen table with the coated samples is removed from the vacuum chamber, using the specimen table manipulator, as described in section 4.6.

5.10. Floating off an Cleaning the Specimen Replicas

Remove specimen carriers with coated samples from the specimen table and float off the replica from the sample by carefully dipping the specimen carrier into a suitable solvent in a flat angle tilted position.

NaClO (eau de javel) or CrO3 (30%) are suitable for animal tissue and cell suspensions.

H2SO4 (70%) is suitable for plant tissue and cell suspensions.

A 50:50 mixture of chloroform-methanol is suitable for samples with high grease content.

A replica of an animal or plant tissue-specimen is often only removed from the tissue block, after the tissue material has completely dissolved.

When the replica has been cleaned from the sample material using the suitable solvent, the replica is rinsed three times in double-distilled water and lifted onto a carrier grid.

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Trouble	Cause	Elimination
QSG 100 film thickness monitor		
No display appears on the QSG 100 film thickness monitor after the mains switch of the unit has been operated.	Mains switch at rear of the QSG 100 has not been switched on.	Switch on mains switch.
	Automatic cut-off F5 in the unit has switched off: switch doll points down.	Switch on automatic fuse F5: switch dolly points upwards.
	Switch of the multipoint connector in the unit is switched off: switch doesn't light up.	Switch on multipoint connector.
On the QSG 100 display appears NO QUARTZ instead of coating rate.	Connecting line between QSG 100 and quartz holder is interrupted.	Check connecting parts with control buzzer for current flow or short circuit.
	No quartz crystal has been inserted.	Insert quartz crystal into quartz holder.
EVM 030		
There is no display on the unit, after the mains switch POWER has been operated.	There is no connection voltage on the mains inlet X1.1 of the unit.	Check mains supply: Connection cable, multipoint connector or fuse F5.

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8. DESCRIPTION OF THE UNIT

8.1. Scope of delivery

The scope of delivery of the basic BAF 060 unit contains:

- Unit housing with rack slide-in system for the reception of the required control- and display devices.
- Vacuum chamber (inner diameter 400mm) with connection openings for the high vacuum pumping station, lock chamber and various current-, rotary, and cooling agent feedthroughs, as well as front door with built-in sight glass.
- Lock chamber for introducing specimen table and evaporation sources.
- High-vacuum pumping station with TMH 261 turbo molecular-drag pump, MD4 membrane pump, vacuum angle valves DN16 ISO-KF and DN25 ISO-KF, insertable gate valve DN63, UPC 010 pumping station control unit with integrated vacuum measuring device for three TPR 010 pre-vacuum- and one IKR 050 high vacuum measuring gauge.
- Microtome system with specimen stage, cutting system, rotary shutter and protection sheet.
- LN2-dewar with level sensor, built into the vacuum chamber, for cooling the specimen table.
- Knife/Shutter cooling with direct contact to liquid nitrogen.
- MTC 010 Temperature control unit and MDC 010 motor control unit for the microtome system.
- Selectable manual operation for cutting movement.
- EVM 030 Electron beam evaporation device with two EK 030 evaporation guns.
- QSG 100 quartz crystal film thickness measuring device with integrated oscillator and QSK 060 quartz holder.
- Safety device with force-activated safety disconnector on the vacuum chamber door, position indicator at the lock valve and appropriately programmed switchover points to monitor the pressure via the vacuum measuring system.
- Accessories set, containing:
    - 1 Specimen table loading system
    - 1 Manipulator for specimen tables
    - 1 Manipulator for evaporation sources
    - 1 Adapter for knife manipulator
    - 1 Binocular microscope, 10-fold
    - 1 LN2 connection line
    - 1 Nitrogen gas-connection line (yellow)
    - 1 Compressed air-connection line (blue)
    - 1 Specimen table for 3mm specimen carriers

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8.2. Front View of the Unit

1 Unit housing
2 Vacuum chamber
3 Lock chamber
4 Holder for binocular microscope
5 UPC 010 Pumping station control unit
6 MTC 010 Temperature control unit
7 MDC 010 Motor control unit
8 QSG 100 Quartz crystal film thickness monitor
9 EVM 030 Electron beam evaporation control unit
10 Mains switch
11 LN2-Connection
12 Hand wheel for manual cutting operation

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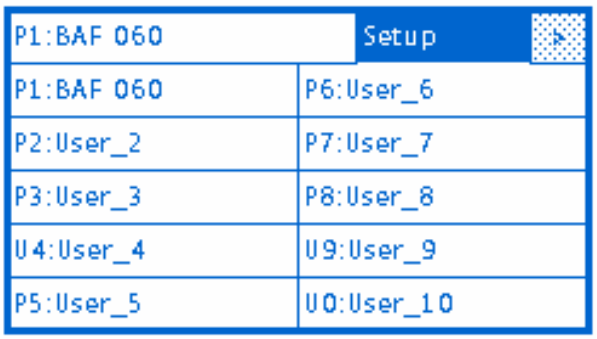
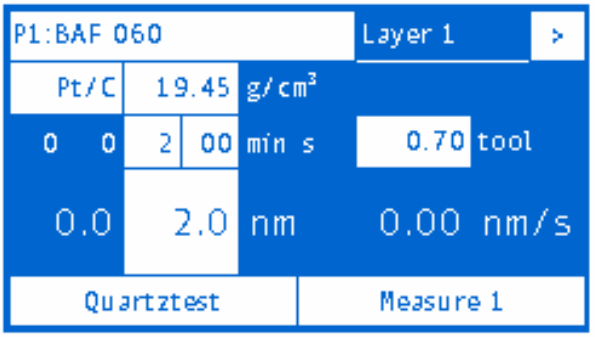
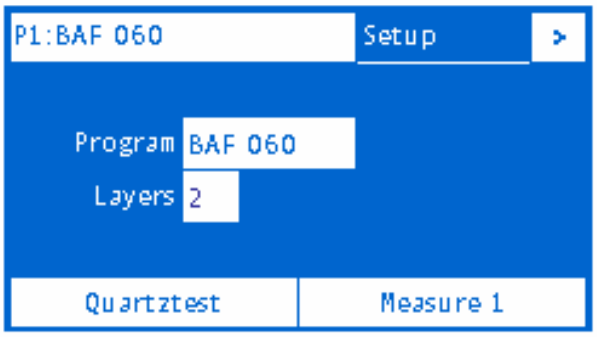
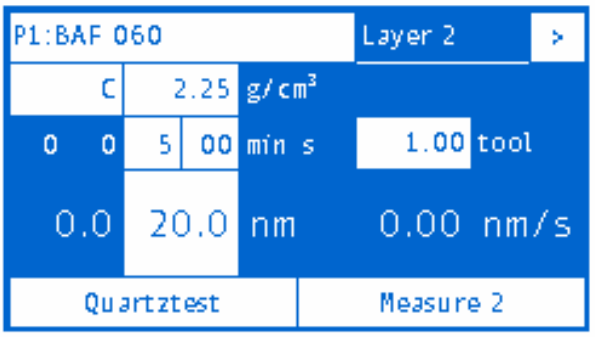
8.8. QSG 100 Quartz crystal film thickness monitor









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	Description Teil	Item Pos.	Order No. Bestell-Nr.	S	Reference Bemerkungen
1	Vacuum chamber	1			BU 800 284 E/2
1	Manual cutting system	2			BU 800 284 E/4
1	Load lock chamber	3			BU 800 284 E/13
1	Evaporation system	4			BU 800 284 E/14
1	Loading manipulator for el. beam guns	5	BU 018 691 –T		BU 800 284 E/17
1	Loading manipulator for specimen tables	6	BU 018 693 –T		BU 800 284 E/18
1	High vacuum pumping system	7			BU 800 284 E/19
1	UPC 010 Pumping system control	8	BU S07 250		
	MTC 010 Temperature control unit	9	BU S06 750		
	MDC 010 Motor control unit	10	LE 01121 LN		
	QSG 100 Film thickness and rate monitor	11	LE 03349 VN		
	QSK 060 Quartz measuring head	12	LZ 03440 VN		

	Spare Parts for / Ersatzteile zu			BAL-TEC AG
	BAF 060 Freeze Etching System		**BU P01 250**	**BU 800 284 E/1**

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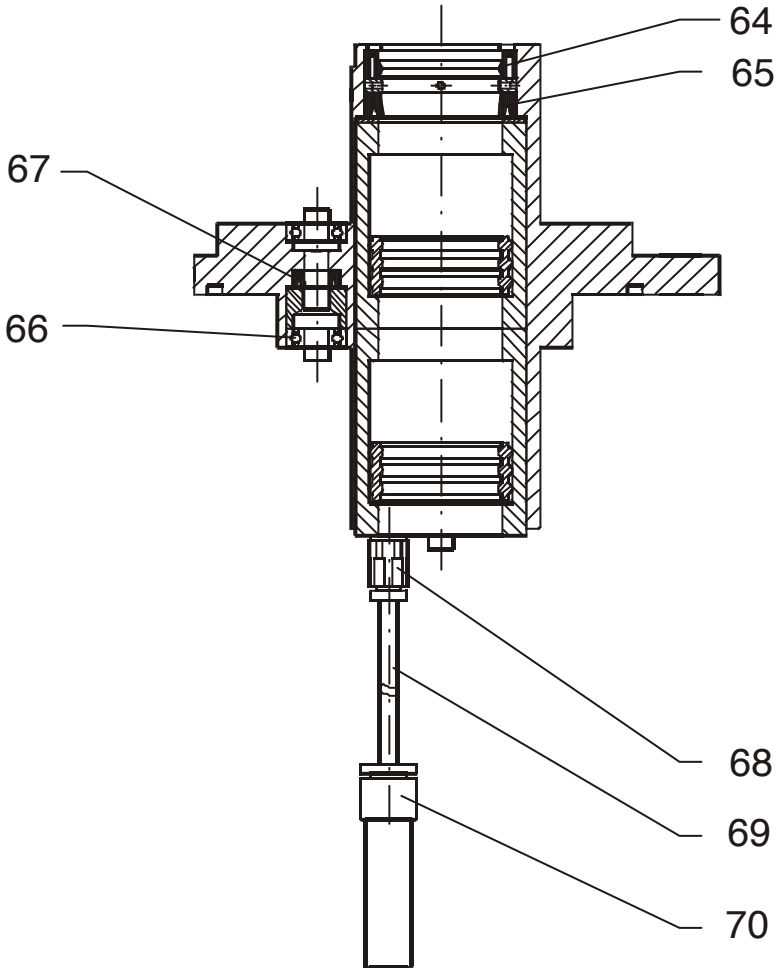
	Description Teil	Item Pos.	Order No. Bestell-Nr.	S	Reference Bemerkungen
1	Shaft seal Viton, Ø 33 x 6mm	64	B 8010 096 26		
1	Shaft seal, Ø 25 / 33 x 5.5mm	65	LE 03107 KN		
1	Shaft seal, Ø 5 / 10 x 4mm	66	LE 03106 KN		
2	Grooved ball bearing, Ø 5 / 13 x 4mm	67	LE 00839 KN		
1	Connecting coupling, Ø 4mm, M5	68	LE 01280 KN		
1	Connecting tube, Ø 4 x 205mm	69	LE 01283 KN		L = 1.5m
1	Reducing coupling, Ø 10 / 4mm	70	LE 01282 KN		
1	O-Ring Viton, Ø 88.57 x 2.62mm	71	LE 01025 KN		



	Spare Parts for / Ersatzteile zu			BAL-TEC AG
	Feedthrough flange cpl.		**LE 00851 MN**	**BU 800 284 E/1**