



Instruction Manual

UHV Evaporator EFM 2/3/3s/4

Triple Evaporator EFM 3T

IBAD Evaporator EFM 3i

Version 3.9

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Related Manuals

EVC 300 / 300s	
EVC 100 / 100s	Power supplies for EFM
EVC 100 L	
EVC 300i	Power supply for EFM with IBAD functionality

Field of Application

The **EFM 2, EFM 3, EFM 3s, EFM 4, EFM 3T** or **EFM 3i** is an evaporator that can be used to evaporate metals and other materials from rods or a crucible in vacuum.

The UHV evaporator **EFM 2, EFM 3, EFM 3s, EFM 4, EFM 3T** or **EFM 3i** must always be used in combination with a CE-complying power supply e.g. **EVC 300, EVC 300s, EVC 300i, EVC 100, EVC 100s** or **EVC 100L**.

The EVC power supplies and the EFM UHV evaporators must always be used:

- with original cable sets which are explicitly specified for this purpose
- with all cables connected between the evaporator and the power supply
- with the evaporator in UHV condition fixed to a vacuum vessel
- with cooling water supply connected to the evaporator
- with all electronics equipment switched on
- in an indoor research laboratory environment
- by personnel qualified for operation of delicate scientific equipment
- in accordance with all related manuals.



Warning: Lethal Voltages!!

Adjustments and fault finding measurements as well as **installation procedures and repair work** may only be carried out by authorised personnel qualified to handle lethal voltages.



Attention: Please read the safety information in the relevant manual(s) before using the instrument.

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1. Description and Specifications

In the UHV evaporators **EFM 2**, **EFM 3**, **EFM 3s**, **EFM 4**, **EFM 3T** and **EFM 3i** the evaporant is either evaporated from a bar, from a rod or from a crucible. This is achieved by electron bombardment heating. The bombarding electron beam induces a temperature rise of the evaporant, causing evaporation.

The crucible-free evaporation is characterised by an ultimate degree of cleanliness with only slight increases of background pressure. From appropriate crucibles (see appendix A) low melting point, low vapour pressure or reactive materials can be evaporated. The instrument is designed for high precision sub-mono-layer up to multi-layer deposition of a wide variety of evaporants including highly refractory materials.

A key feature of the EFM is the **integrated flux monitor** (not **EFM 2**, but the flux monitor can be upgraded later). Once calibrated by a quartz thickness monitor, the flux monitor can replace the quartz balance by **continuously** monitoring the evaporation rate. Flux is measured directly, which allows a much more precise rate adjustment and much faster rate control than an indirect, i.e. temperature measurement by a thermocouple. For precision layer control, an external signal of a calibrated monitor (quartz balance or MEED oscillations) can be used for regulation with the EVC 300 series power supplies.

The beam exit column contains an ion collector which serves as a flux monitor. **At a given electron emission current (I_{EM}) and e-beam energy the ion flux measured there is directly proportional to the flux of evaporated atoms.** The ion flux is shown on the display of the EVC power supply; with the power supplies EVC 300 / EVC 300s / EVC 300i the heating power (i.e. the high voltage) can be regulated to obtain a constant growth rate. The flux monitor also operates with the shutter closed thus allowing to pre-set the evaporation rate.

Some growth processes require an ion free deposition. This requirements can be fulfilled with the optional ion suppression lens (EFM 3s/EFM 3i). The lens deflects the ionised material vapour back into the EFM. Only the neutral material vapour can reach the sample.

The EFM comes with a shutter at its outlet which can be opened and closed by a rotary drive (not **EFM 2**, but the shutter can be upgraded later). This allows precise flux adjustment prior to exposure, and exact control of the evaporation time. As an option, the shutter can be motor operated under computer remote control together with the power supplies EVC 300 / EVC 300s / EVC 300i.

The evaporation cell is enclosed in a water-cooled copper cylinder (cooling shroud). This, and the fact that just a restricted region of the evaporant is heated, facilitates to keep the background pressure during evaporation below 10^{-10} mbar. But this also depends on the target material and on the pumping speed of the vacuum system. Some evaporants and crucibles contain huge amounts of gas that are liberated on heating.

1.1. EFM 2

The single evaporator EFM 2 is the basic version of the standard evaporator EFM 3. The EFM 3 features shutter and flux monitor can be upgraded (user and factory upgrade possible) at the EFM 2.

1.2. EFM 3/4

The single evaporator EFM 3 is the standard evaporator that can be used for small samples up to 15mm diameter. The EFM 4 features a larger outlet that allows the deposition of larger samples of up to 60mm diameter, depending on the sample distance, while maintaining all properties of the EFM family.

1.3. Triple evaporator EFM 3T

The triple evaporator EFM 3T features three completely separated evaporation cells on one CF 35 flange each one similar to a single EFM 3. Each cell has an independent electron bombardment heater. It can be used for simultaneous or subsequent evaporation of up to three different materials and is therefore ideally suited to grow binary and tertiary compounds or multilayers.

The evaporation shows no material crosstalk between the three cells. Each cell has its integral flux monitor so that the flux can be measured independently and pre-set before opening the shutter. With the shutter mechanism each cell can be opened separately as well as various combinations of the cells.

1.4. EFM 3s, evaporator with ION Suppressor

The evaporator EFM 3s is designed for ion free deposition. An additional lens suppresses all ions in the material vapour. This enables the coating of delicate samples, where the ion fraction of the metal vapour disturbs the proper growth of the thin layers.

This feature is also available with the EFM 3i.

1.5. IBAD evaporator EFM 3i

The evaporator EFM 3i is designed for Ion Beam Assisted Deposition (IBAD). It features an optimized filament sample geometry, an ion-lens and an gas inlet for production of additional ions.

The filament region of the evaporator is optimised for self-ion or noble-gas ion production. The ions are created in a small region in front of the evaporant by the e-beam heating and are extracted into the ion-lens.

The ion-lens is used to focus the ionised fraction of the metal vapour or additional ions onto the sample. With the gas inlet production of additional ions of e.g. noble gases is possible. The gas is guided directly toward the filament-evaporant region to minimize the pressure rise for a given add-ion flux.

The IBAD mode of the evaporation is be used to enhance the layer by layer growth for certain systems, both for homo- and heteroepitaxy. For details see the reference and literature cited therein.

The EFM 3i can also be used to suppress the ions by exchanging the flux monitor and the ion lens; all ions are suppressed by a HV at the flux-monitor electrode and the ion-lens electrode is used for measuring the ion flux. This enables the coating of delicate samples, where the ion fraction of the metal vapour disturbs the proper growth of the thin layers.

The geometry of the EFM 3i is derived from the standard EFM 3 evaporator and features all known properties: Easy and clean evaporation.

Further properties and test results of the EFM 3i prototype are described in:

“An evaporation source for ion beam assisted deposition in ultrahigh vacuum”,
J. Kirschner, H. Engelhard and D. Hartung,
Rev. Sci. Instrum., Vol. 73, No. 11, Nov. 2002.

1.6. Specifications

Specifications EFM

e-beam energy:	about 0-1000 eV, (600-800 eV typically),
e-beam power	$P_{\max} = 300 \text{ W}$ with EVC300 ($I_{\max} = 300 \text{ mA}$)
Filament current:	typically 1.8 - 2.2 A, 2.5 A maximum
maximum bakeout temperature :	250°C
maximum crucible load:	see list in the appendix E1
cooling water flow:	> 0.5 l/min at $T \approx 30^\circ\text{C}$, max. pressure = 6 bar
temperature range with rod evaporation:	300° up to 3300°C, depends on size of evaporant (heat loss)
temperature range with Ta-, Mo- or W-crucible:	300° - 2000°C
temperature range with Knudsen crucible (stainless steel):	160°C - 800°C
rod diameter:	0.5 -6 mm, depending on maximum temperature to be reached, standard Mo clamp accepts up to 1.5 mm diameter.

Specifications EFM 2/3/3s/4

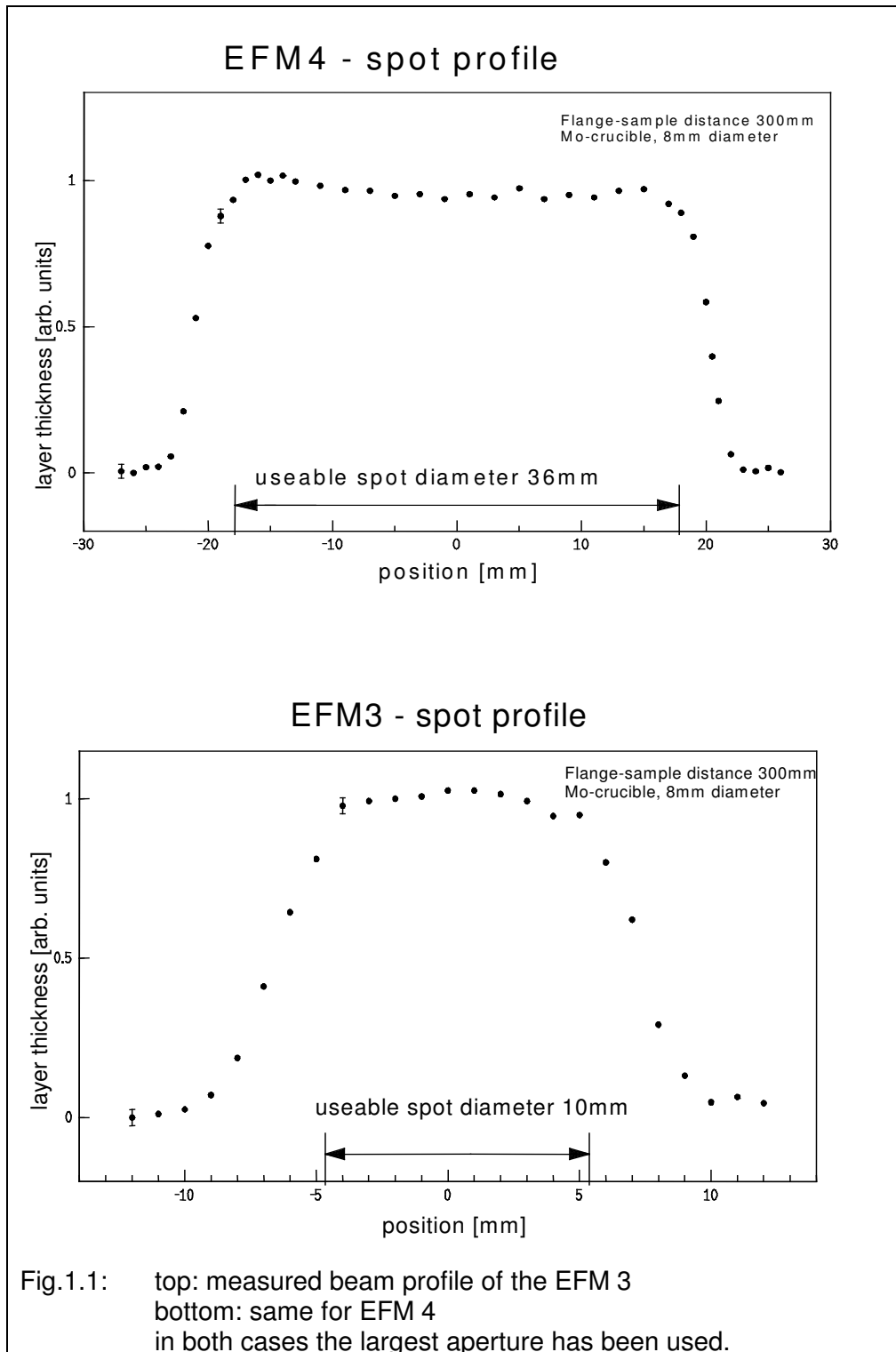
beam spot diameter at the sample	EFM 2/3/3s : 4 to 15 mm EFM 4 : 6 to 40mm, depending on distance and exit, see Fig. 1.1 and 1.2
beam divergence:	EFM 2/3/3s : $\pm 2^\circ$, $\pm 2.2^\circ$ or $\pm 2.4^\circ$, dependent on exit tube mounted. The EFM SKB contains three exchangeable exit tubes of 4, 6 and 7.4 mm I.D. EFM 4 : $\pm 2.2^\circ$, $\pm 3.6^\circ$, $\pm 5.1^\circ$ or $\pm 6.9^\circ$, depending on exit aperture. The EFM SKB contains four exchangeable exit apertures of 6, 10, 14 and 19 mm I.D..
flange to sample distance	220 - 270 mm (other length on request)
insertion depth	210mm (other length on request)
tip of evaporant to sample distance:	100-150 mm typically
end of EFM to sample distance:	10-60 mm
Weight of evaporator:	1.7 - 2.1 kg

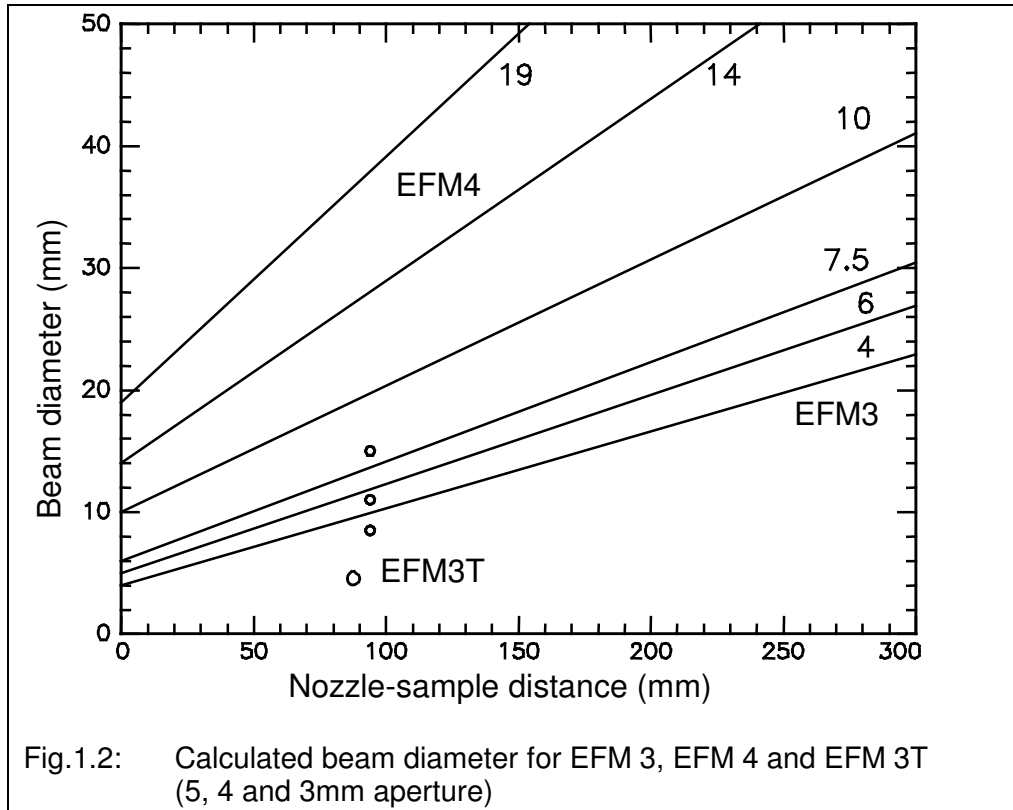
Specifications EFM 3T

beam spot diameter at the sample	15mm with 5mm I.D. exit aperture 11mm with 4mm I.D. exit aperture 8.5mm with 3mm I.D. exit aperture, see Fig. 1.2
beam divergence:	$\pm 2^\circ$, $\pm 2.2^\circ$ or $\pm 2.4^\circ$, dependent on exit apertures mounted. With the EFM 3T come three exchangeable exit apertures of 3,4 and 5 mm I.D..
flange to sample distance:	10" = 254mm
distance from the end of EFM to the sample:	94mm
selectable evaporation cells	all combinations except simultaneous evaporation out of cell 2 and cell 3 (see fig.2.7)
max. crucible diameter	8mm (see appendix E1)
Weight of evaporator:	2.9 kg

Specifications EFM 3i

beam spot diameter at the sample	see EFM 3
beam divergence:	see EFM 3
flange to sample distance	see EFM 3
tip of evaporant to sample distance:	see EFM 3
end of EFM to sample distance:	see EFM 3
max. crucible diameter	8mm (see appendix E1)
Ion lens voltage	100 .. 1100V
Weight of evaporator:	2.4 kg





1.7. Dimensions and Connections

EFM 2

Similar to the dimensions of the **EFM 3**, but without rotary and BNC feedthrough. The evaporator outlet is a little bit smaller, but has the same length.

EFM 3/3s/4

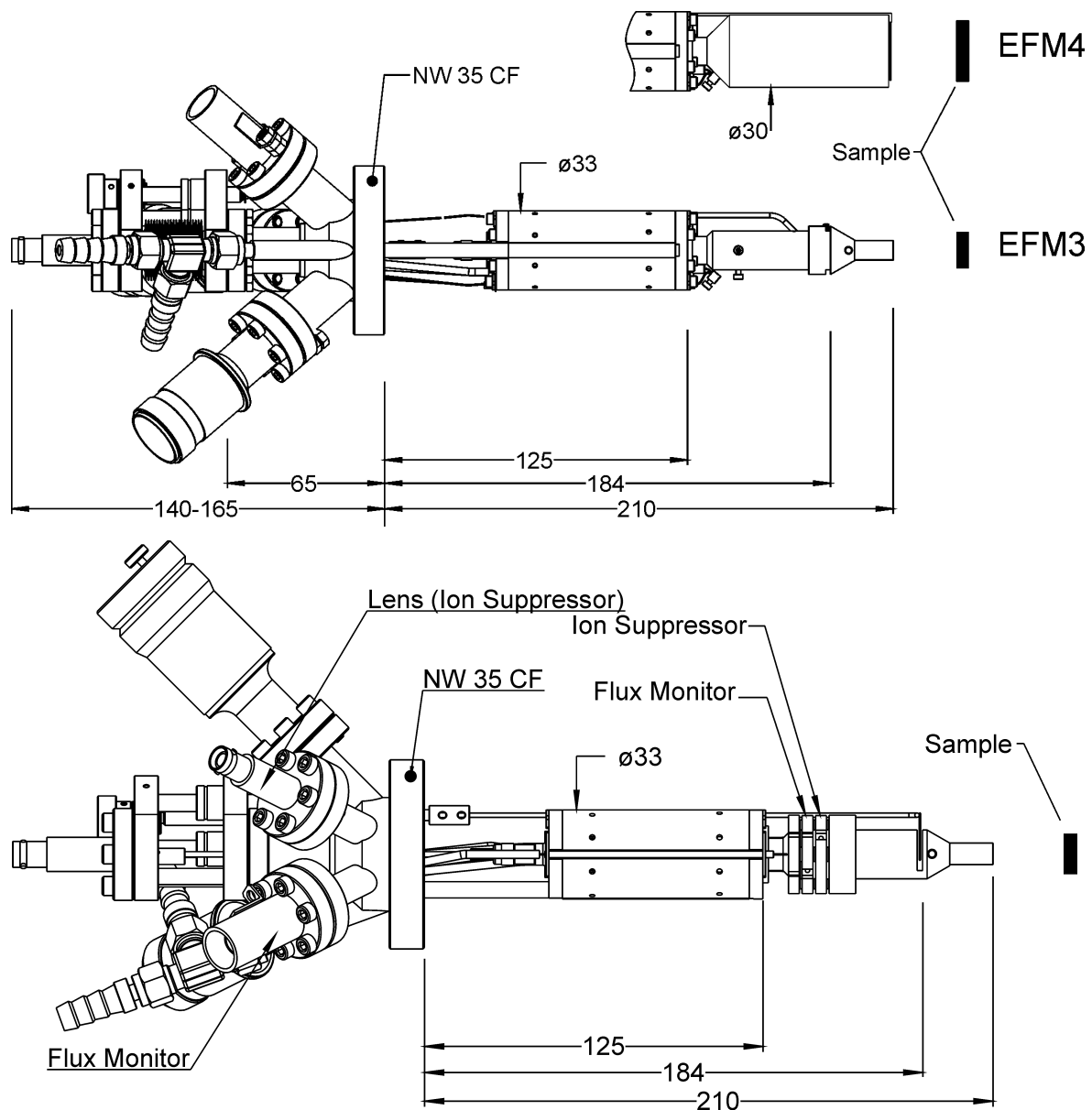


Fig. 1.3: Outer dimensions and feedthroughs of the EFM 3/4 and EFM 3s. The HV-feedthrough is mounted on a linear motion drive that moves only the evaporant. Its travel is 25mm, 50mm optional. The EFM 3s has an additional ion lens (ION Suppressor), which is connected to a SHV feedthrough.

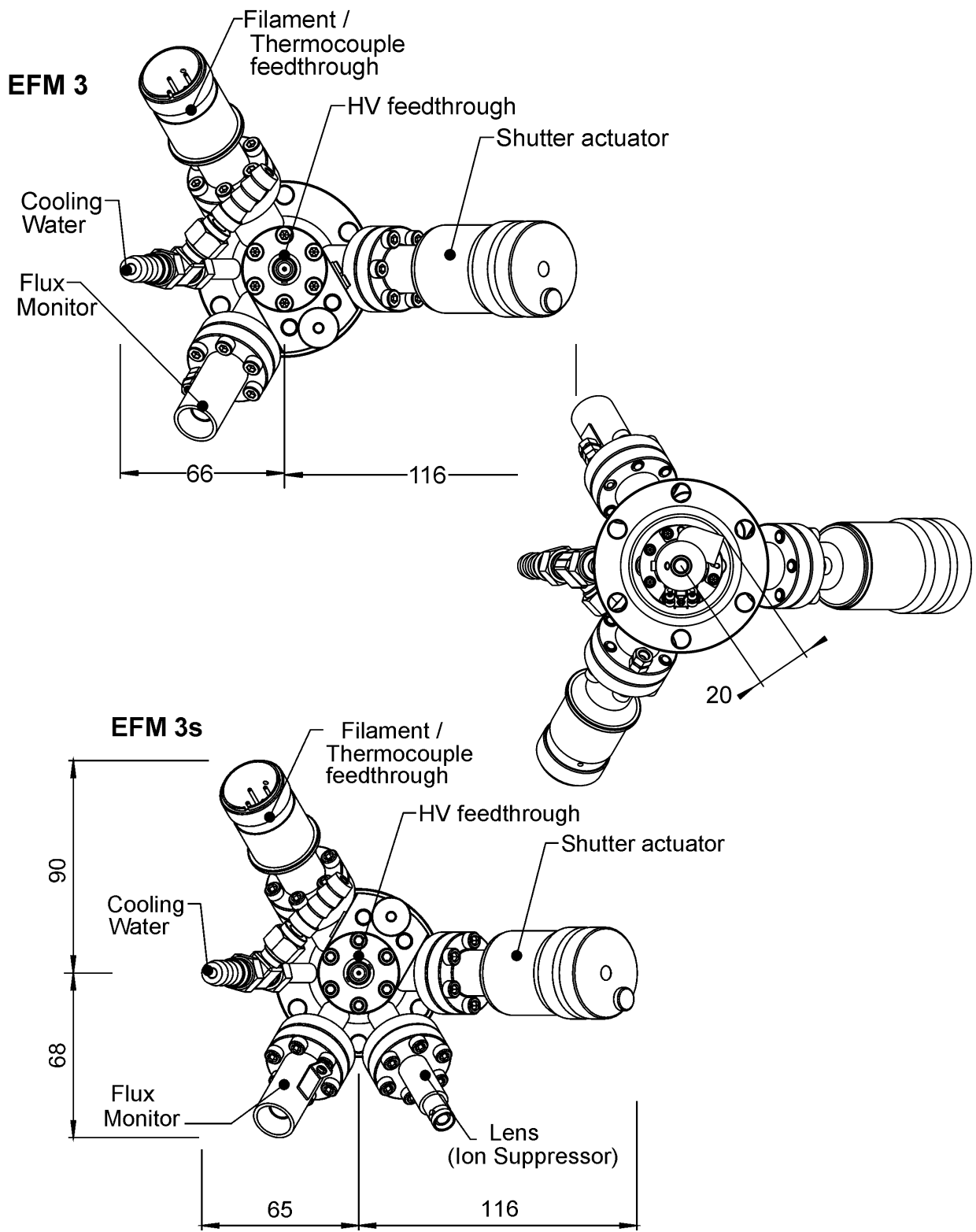


Fig. 1.3: (continued) Outer dimensions of the EFM 3 /EFM 3s from the rear view and the shutter dimensions for EFM 3 /EFM 3s. A tube of 40mm diameter is needed to fully open the shutter.

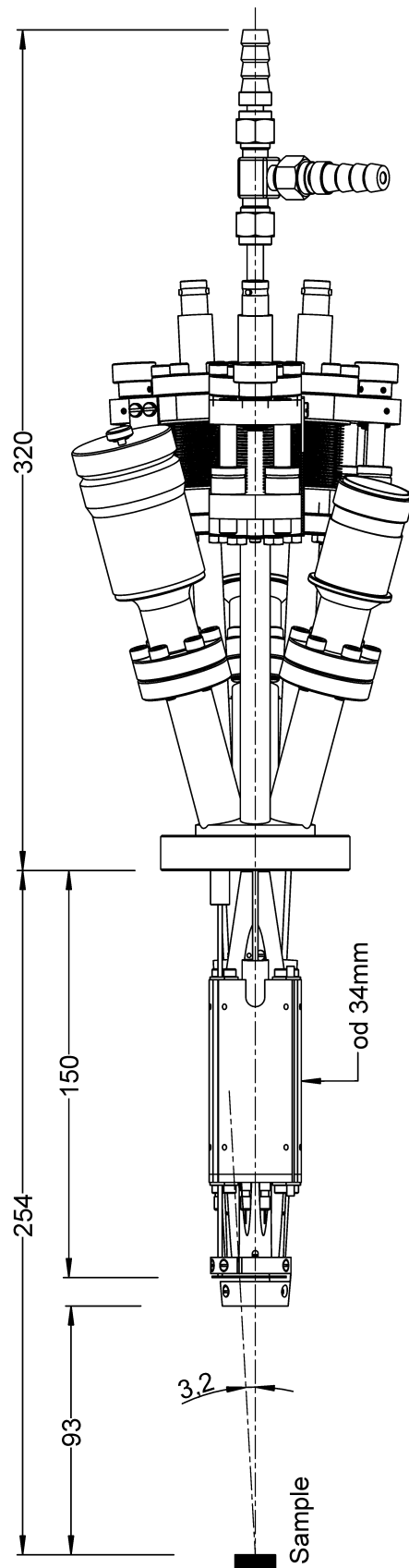
EFM 3T

Fig.: 1.4 a: Outer dimensions of EFM 3T

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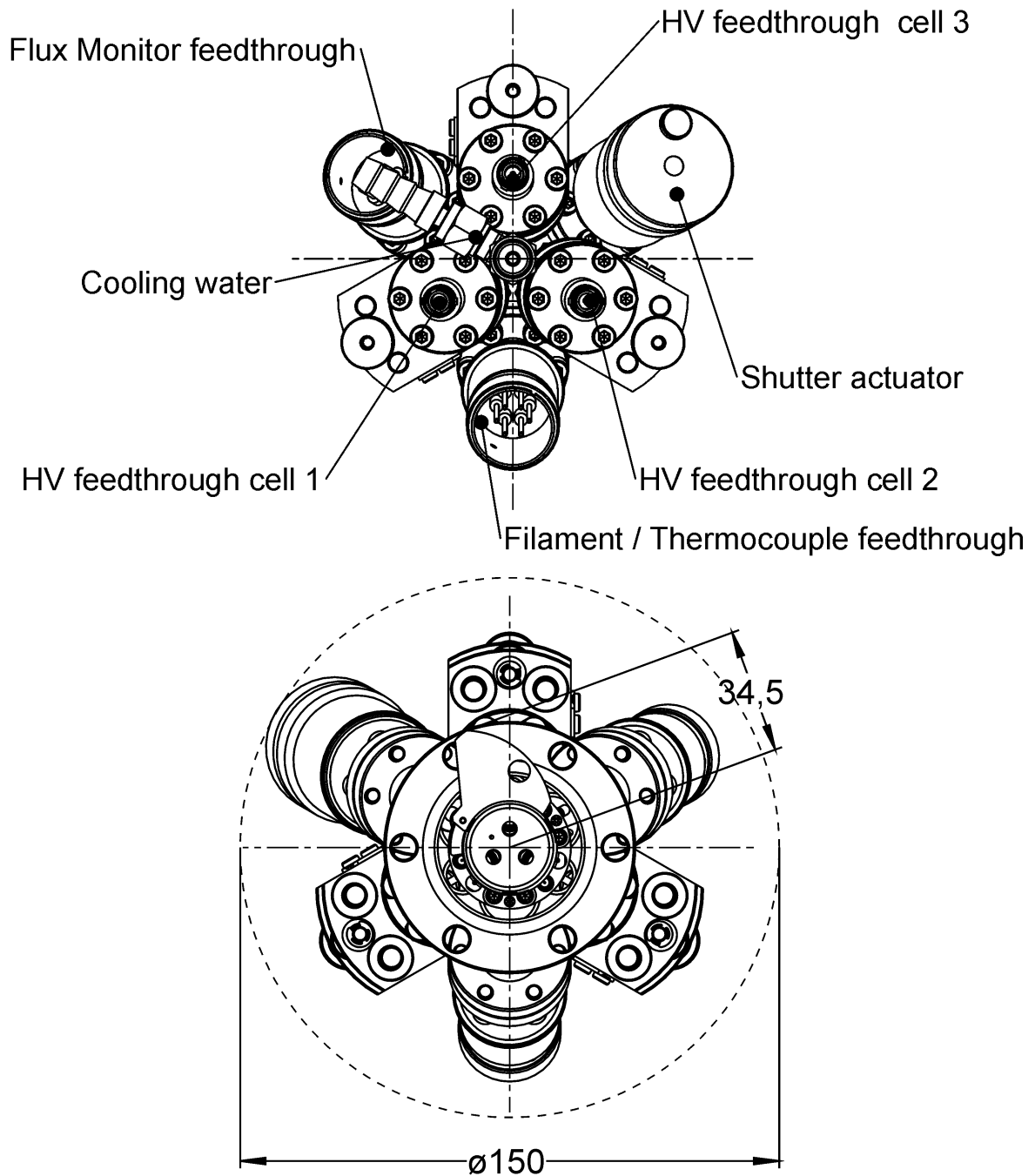


Fig. 1.4: Outer dimensions and feedthroughs of the EFM 3T. The lower drawing shows the max. space needed for the shutter. If no simultaneous evaporation is needed 25mm is sufficient (Shutter Position C in Fig. 2.7). All HV-feedthroughs are mounted on linear motion drives that move only the evaporant (25mm travel)

EFM 3i

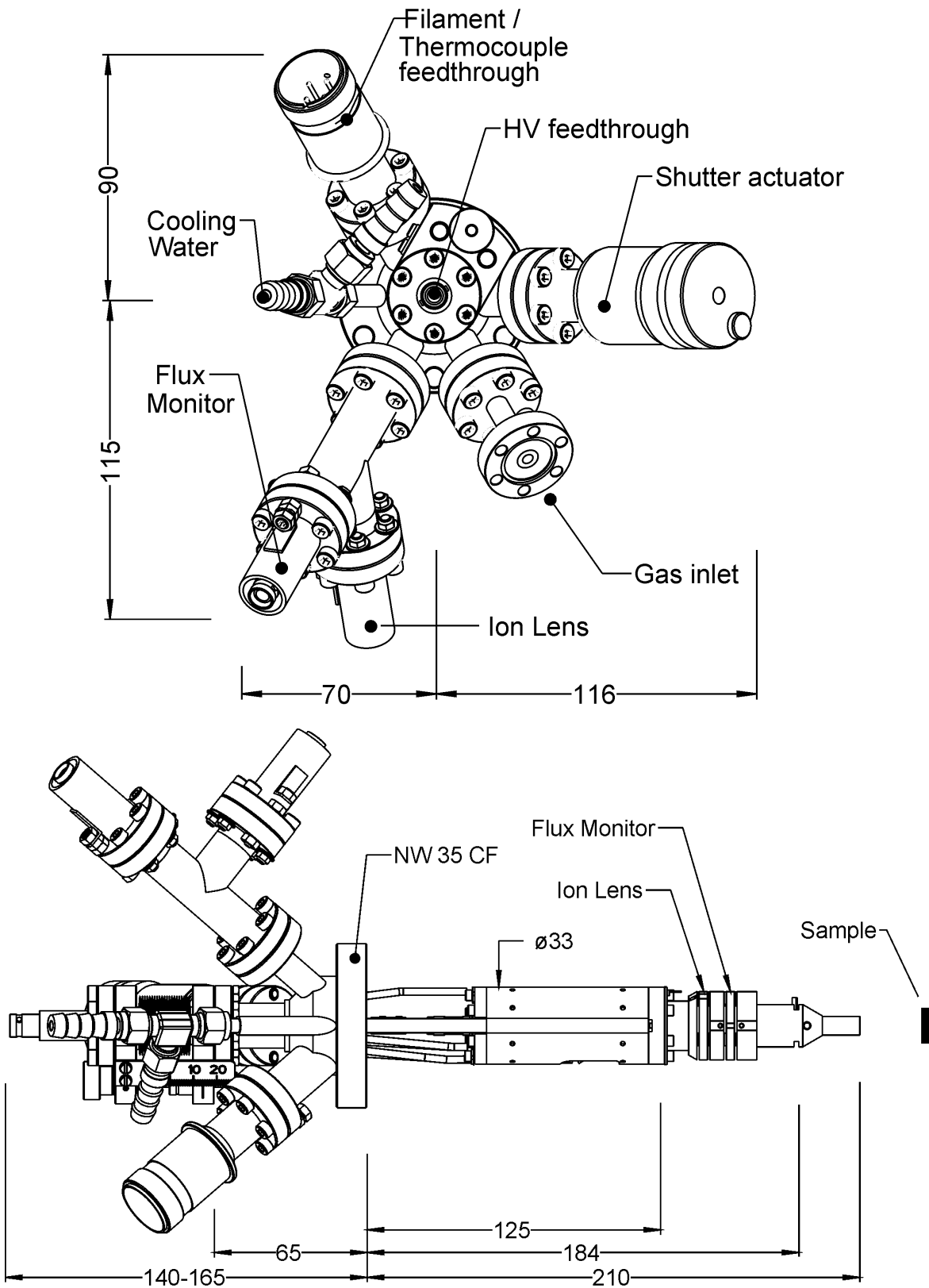


Fig. 1.5 Outer dimensions and feedthroughs of the EFM 3i. The HV-feedthrough is mounted on a linear motion drive that moves only the evaporant. Its travel is 25mm, 50mm optional.

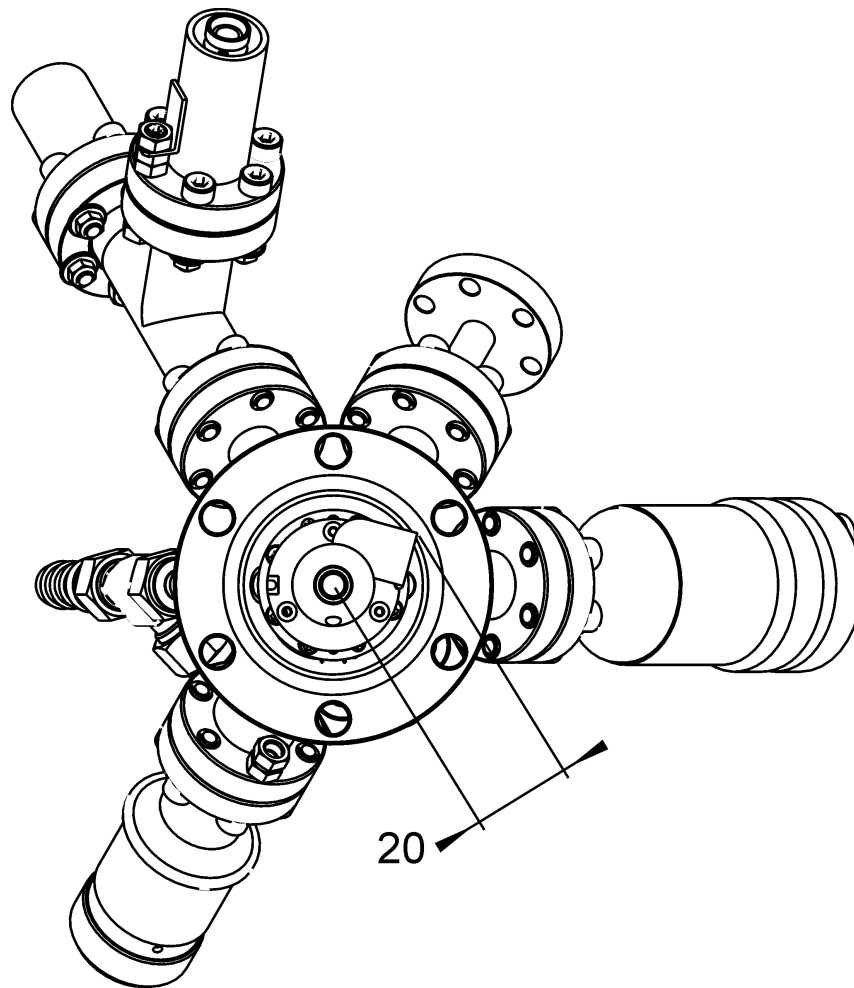


Fig. 1.5: (continued) Outer dimensions of the EFM 3i shutter. A tube of 40mm diameter is needed to fully open the shutter.

2. Installation

The important outer dimensions for installation of the EFM-evaporators are shown in fig. 1.3-1.5.

For the EFM 2/3/3s/4/3i a chamber tube size with an inner diameter of at least 34 mm is sufficient for pumping. The maximum tube length allowed is 180 mm (200mm for EFM 4), otherwise the shutter cannot be fully opened.

The EFM 3T needs a tube of 35 mm diameter; the tube length should not exceed 145 mm, otherwise a tube diameter of 70mm is needed to fully open the shutter of 50mm to open the individual cells (see. fig. 1.4).

For special length (optionally available) correct the dimensions for the insertion depth ordered.

For operation with a rod the EFM can be mounted in any orientation, although caution should be applied when aiming downwards. At temperatures above the melting point the evaporant may dislodge and fall out of the crucible used. When in operation with a crucible a minimum incline of 20° with respect to the horizontal (flange to sample) is recommended. Many materials wet the crucible surface, in this case nearly horizontal mounting is also possible. For changing the evaporant (rod or crucible) a free space of about 200 mm at the HV-feedthrough side of the EFM is required.

The best direction to evaporate is along the sample normal. Then the evaporated layer will be most homogenous. At other angles the evaporant spot looks elliptical and the density of deposited atoms is different along the small and the long ellipsoidal axes.

Adjustment:

If the sample itself can be adjusted a port aligner is not required. The optional port aligner allows a tilt of $\pm 3^\circ$. If using a port aligner the inner diameter of the chamber tubing should be larger than 35 mm and the tube length should be as short as possible, at best shorter than 50 mm. Only then the full tilt of $\pm 3^\circ$ can be used.

The glowing filament of the EFM makes a light spot at the sample (shutter opened). The illuminated area is identical with the actual deposition area. A viewport looking towards the sample helps to align its position.

First degassing of a new EFM:

A new EFM should be mounted first without a rod or with an empty crucible. After bakeout (see sec. 4.1) first degas the EFM (see sec. 4.2) then degas the crucible (see sec. 4.3).

2.1 Water Cooling

The water hose (8 mm i.d.) is attached to the hose nipples (8 mm o.d.) and must be secured by means of screw clamps. Quicklock water hose connectors are available optional. Prevent forces pulling on this connection by employing the necessary carefulness. For bake-out, the water hose has to be removed. The straight port should be used for inlet, the radial port for outlet. A water flow rate of more than 0.5 l/min at a maximum temperature of 30 °C is recommended.

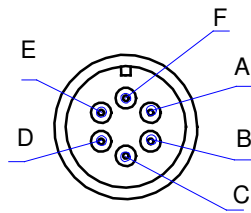


Fig. 2.5 : Flange connector for Filament and Thermocouple from atmospheric side:

- F) Filament cell 1
- A) Filament cell 2
- B) Filament cell 3
- C) Thermocouple CuNi (neg.)
- D) Thermocouple NiCr (pos.)
- E) Filament GND

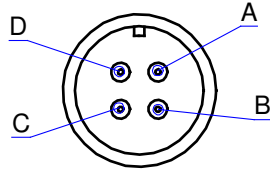


Fig. 2.6 : Flange connector for Flux monitors from atmospheric side:

- A) Flux monitor cell 1
- B) Flux monitor cell 2
- C) Flux monitor cell 3
- D) not connected

Checks

Check the resistance between the filament pins F, A and B and pin E at the 6 pin feedthrough. It should be $R < 1 \Omega$. None of these pins should have contact to ground.

Thermocouple pins C and D to ground: $R < 2 \Omega$.

Check that there is no contact between all flux monitor pins (4 pin feedthrough) and ground.

To check short-circuit safety of the HV-feedthrough proceed as described in the last section.

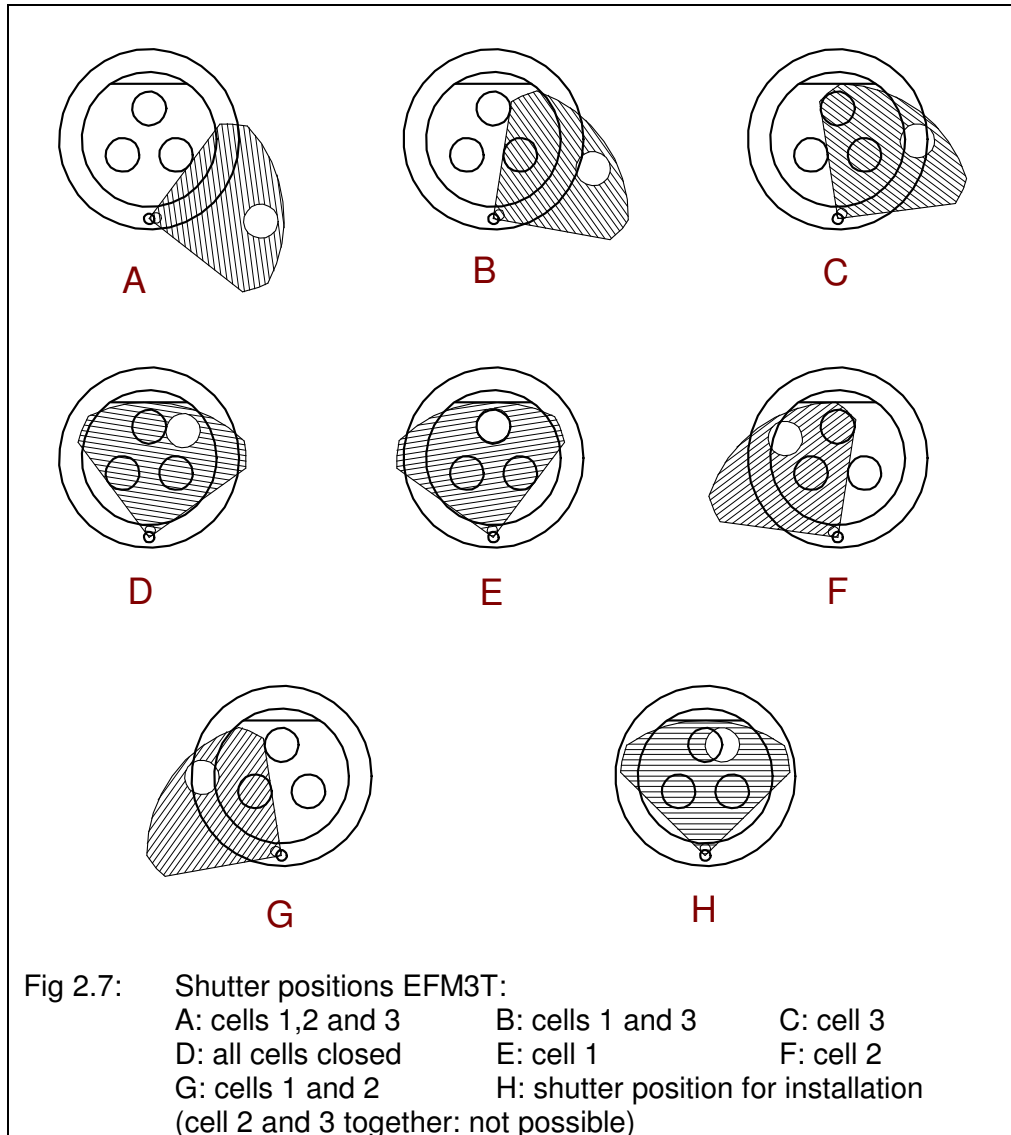
Note this new design of the filament contact since May 2004, older EFM 3T don't use the pin E (6 pin feedthrough). Instead the filament GND is connected to the evaporator cooling shroud.

Old version of EFM 3T (grounded filament): up to and including SN 0110-2004, SN 0112-2004 and all 4 digit SN up to SN 0310

New version of EFM 3T (isolated filament): SN 0111-2004, SN 0113-2004 ff.

2.4. Shutter Positions EFM 3T

The triple evaporator EFM 3T has a multi-position shutter that is used to open the cells selected. All combinations (except cell 2 and 3 together) can be chosen. Fig. 1.4 shows the positions of the shutter. See the testsheet for the corresponding position at the rotary feedthrough. The reading of the rotary feedthrough should be checked before installation.



2.5. Gas inlet of EFM 3i

The EFM 3i is equipped with an additional port for gas inlet into the filament/sample region where additional ions are produced.

A leak valve can be mounted onto the CF 16 flange of the gas inlet. Do not remove the CF 16 spacer, which supports the gas capillary. If the inlet is not used, it is not necessary to separately pump the inlet line. Close the CF16 flange of the spacer with a blind flange. That is also sufficient for UHV operation.

3. Mounting and changing the evaporant

3.1. Mounting the evaporant in EFM 2, EFM 3, EFM 3s, EFM 4 and EFM 3i

For initial mounting and/or later changing of the evaporant the rear CF 16 flange (HV feedthrough see fig. 1.3 and 1.5) is used. Loosen this flange and cautiously pull the support of the evaporant out of the furnace body. A \varnothing 2 mm molybdenum rod serves as a mounting post.

Fitting a Rod Evaporant

The evaporant should be spot welded to the molybdenum post or the Mo-barrel connector delivered with the EFM may be used to support the rod (see fig. 3.1). Always use extremely clean material.

Good dimensions for evaporation material are bars of 1.5-2.5 mm diameter. Optional Mo-barrel connectors that support larger rods are available (see App. E). Two or three twisted wires with a diameter of 1 mm or less are also possible. Only for materials with very high evaporation temperatures smaller diameters can be used to reach the maximum temperature with lower heating powers.

With "soft" materials such as gold, good results can be obtained if the rod evaporant is mechanically supported by an appropriate spiral of a non-alloying metal. For gold wire of 1 mm dia. a supporting tungsten spiral (made e.g. from 0.25 mm wire) can be used. For easier handling use a tungsten crucible.

The total length of the evaporant must not exceed 50 mm (see fig. 3.1), or there would be a danger of short-circuiting the tip of the rod to the housing. A typical length of evaporant (to be on the safe side) would be 45mm. The tip of the evaporant should not intersect the plane defined by the filament wire.

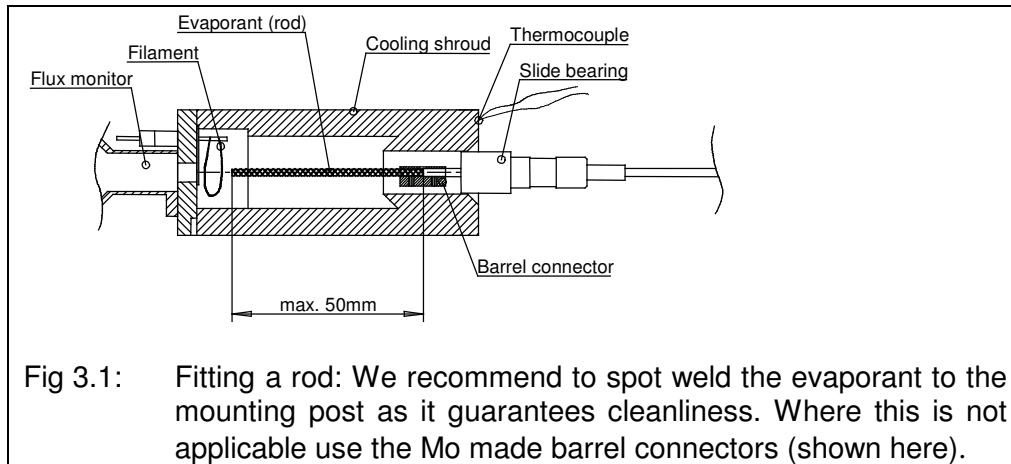
Centre the rod before inserting into the evaporator

For inserting the evaporant set the linear feedthrough to maximum length (fully counter-clockwise), otherwise the filament wire may be destroyed whilst inserting. Carefully feed the slide bearing through the centre of the rear opening into the copper cooling body. The evaporant bar must not be bent.

Check the concentric position of the rod through the beam outlet (with the shutter being open and a suitable illumination) and correct when necessary.

We recommend a careful ex-vacuum alignment of the evaporant to the centre (with about \pm 1 mm accuracy). Note that the HV-flange and the end-flange of the linear motion feedthrough both carry a mark for reproducible positioning.

Tighten the mini flange. Check that there is no short circuiting of the evaporant to the filament or to the cooling shroud using an ohmmeter. Resistance between HV and ground should be $> 2 \text{ M}\Omega$.



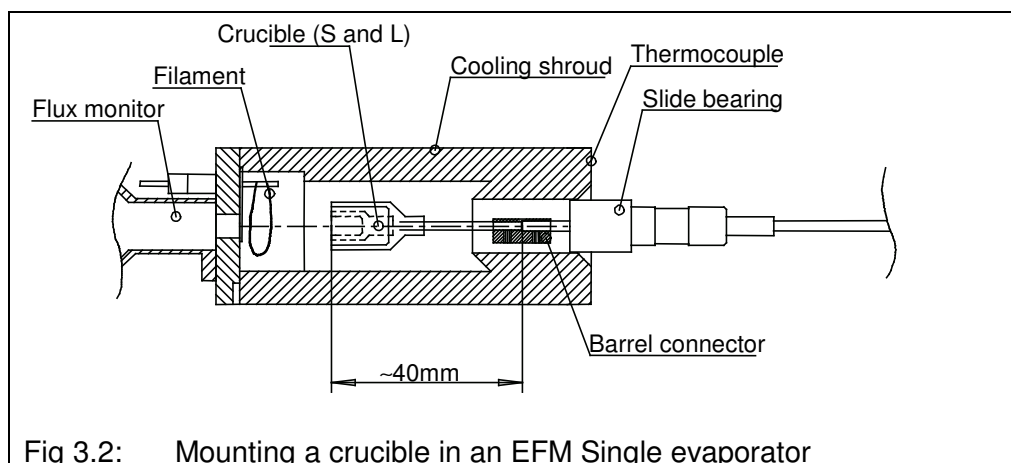
Mounting a crucible

Available crucibles are made of tungsten, molybdenum, graphite, tantalum, Al_2O_3 , pyrolytic boron nitride (pBN), zirconium oxide (ZrO_2), beryllium oxide (BeO), or stainless steel with nozzle. For sizes of the crucibles see section E.1. The EFM 3i is restricted to crucibles with a maximum outer diameter of 8 mm. A crucible is mounted using a molybdenum barrel connector as shown in Fig. 3.2. Check the length of the crucible (40 mm). Note that the HV-flange and the end-flange of the linear motion feedthrough both carry a mark for reproducible positioning.

Check the concentric position through the beam outlet (with the shutter being open and a suitable illumination) and correct when necessary.

We recommend a careful ex-vacuum alignment of the crucible to the centre (with about ± 1 mm accuracy). Note that the HV-flange and the end-flange of the linear motion feedthrough both carry a mark for reproducible positioning.

The crucible can be refilled through the mini CF flange "HV" at the rear end. A new crucible should thoroughly be degassed (see 4.1 and 4.2) prior to initial loading with evaporant material.



3.2. Mounting the evaporant in EFM 3T

For initial mounting and/or later changing of the evaporants the three rear mini CF flanges (HV-feedthrough shown in Fig. 2.3) are used. Loosen one of these flanges and cautiously pull the support of the evaporant out of the furnace body. The evaporant, either a rod or a crucible, is held by a molybdenum barrel connector that also serves as the slide bearing.

Fitting a Rod Evaporant

The evaporant can be spot-weld to the 1.5 diameter molybdenum rod that is delivered with the evaporator or alternatively can directly be inserted into the Mo barrel connector. For the EFM 3T only the standard Mo barrel connector is available.

For mounting the rod into the evaporator proceed as described in sec. 3.1. referring to fig. 3.3.

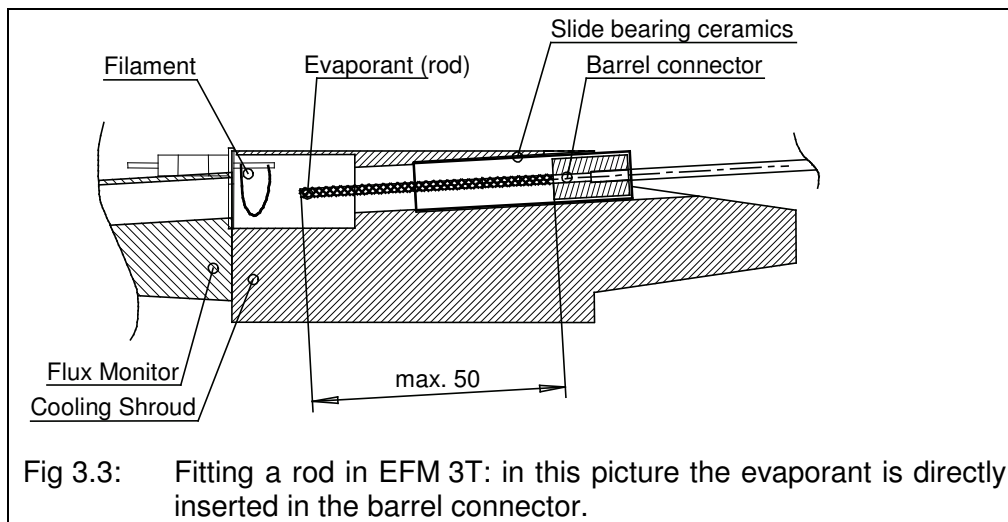
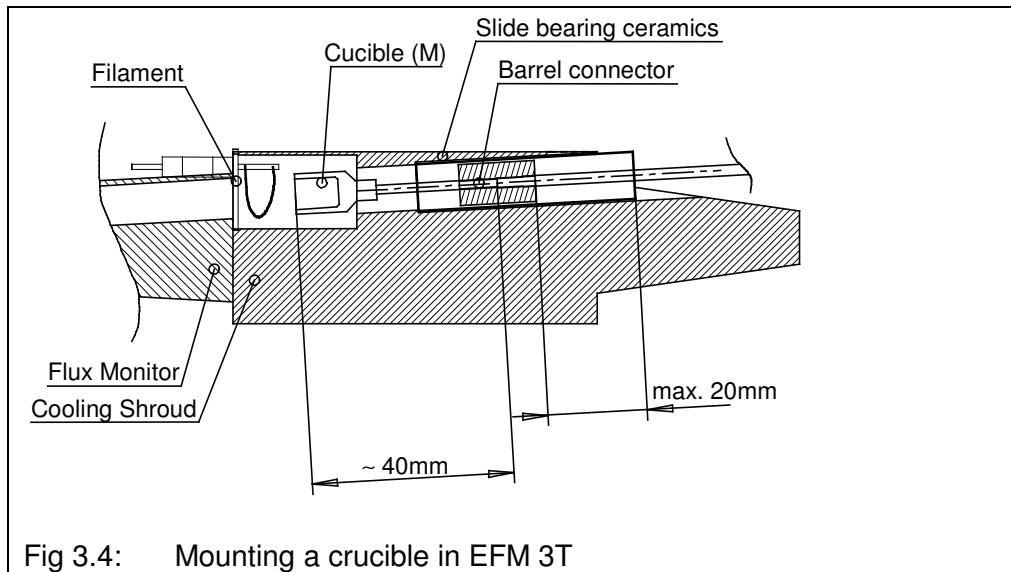


Fig 3.3: Fitting a rod in EFM 3T: in this picture the evaporant is directly inserted in the barrel connector.

Mounting a crucible

All available crucibles are listed in section F.1. All crucibles with a maximum outer diameter of 8 mm can be used with the EFM 3T. A crucible is mounted on a 1.5mm dia. molybdenum post which is directly inserted into the Mo-clamp that also serves as a slide bearing (Fig. 3.4).

For mounting of the crucible into the evaporator proceed as described in sec. 3.1. referring to fig. 3.4.



3.3. Position of evaporant for IBAD

For ion beam assisted deposition with the EFM 3i the position of the rod or crucible is more important than for normal operation. This is both true for the axial alignment and the position of the linear motion. The axial alignment must be optimised before mounting the evaporator as described before. The position of the linear motion can be set during evaporation.

The filament-evaporant space is optimised for effective ion production for the ion extraction and transport to the sample. The evaporant/filament structure then acts like a “saturated diode”. The properties depend crucially on the correct position of the evaporant.

The positioning of the evaporant is described in ref. [7]. Proceed as follows:

- Move the evaporant forward. The top of the evaporant should be beyond the filament plane.
- Apply a HV of 600-800V; start to heat the evaporant with filament current regulation. Wait until a constant emission current stabilizes (e.g. 5-10mA).
- Retract the evaporant using the linear motion until the emission current has dropped to 75% - 80% of the initial value.

For rod evaporation the linear motion must be used to compensate for the shortening of the rod during evaporation!

This procedure also gives a good evaporant position for normal evaporation!

4. Operation

A thorough bake-out and degas of both the evaporator and the evaporant (respectively the crucible) is required to obtain a good background pressure and clean evaporation.

4.1. Bakeout

The EFM evaporators are fully bakeable to 250° C. Remove all cabling and the water hoses for bake-out. Before bake-out the cooling water should be blown out of the evaporator.

4.2. Degassing the evaporator

Degassing is recommended both after bake-out of the UHV system and especially after changing the evaporant. During degassing the pressure should not exceed 5×10^{-6} mbar. During degassing the water cooling must not be used otherwise the temperature of the cooling shroud can not exceed 100° C. Before degassing the cooling water should be blown out of the evaporator.

The temperature of the instrument should be checked with the built-in thermocouple, which is in contact with the outer side of the copper cooling shroud (see fig 3.1). It must not be higher than 300° C. When using an EVC 100/300 power supply, the temperature is displayed at the power supply, the EVC 100L power supply indicates only the temperature below or above the preset overtemperature of 300° C (see corresponding manuals). The accuracy of the temperature measurement is only $\pm 10^\circ\text{C}$ (due to contact potentials), as it is a monitor for degassing only.

It is recommended to open the shutter during degassing. If necessary, the shutter may also remain closed, e.g. if the sample cannot be removed from the outlet. The cell is then degassed through the rear ventilation boreholes.

For degassing use the following procedures. The first version is not available with the EVC 100L power supply:

Degas with filament heating (EVC 100/300)

- Set the high voltage to zero. Otherwise the e-beam voltage would heat the evaporant.
- Slowly increase the filament current up to about 2 A. Take care that the filament protection is set to 2.5 A at the power supply EVC 300/300s/100/100s.
- The copper cooling shroud gradually heats. It will take 1 to 2 hours until a temperature of 200 to 300° C is reached.

Note that due to the smaller filament and larger mass the **EFM 3T** will heat up slower if a single filament is used. It is recommended to degas all cells to be used. All three filaments can be used simultaneously if the power supplies are available.

Degas with evaporant heating (EVC 100L, EVC 100/300)

- Turn the high voltage to about 200V.
- Gradually increase the current until emission starts (typically at 1.8 A filament current). Set to an emission current of about 5 mA (1 W heating power).

- If no emission can be obtained with the standard filament currents, cautiously move the crucible, rod with the linear motion towards the filament. Take care not to touch the filament with the crucible, because of the danger of destroying the filament.
- The temperature of the cooling shroud will rise faster during degassing than without a crucible.
- Degas the evaporator until the desired shroud temperature is reached. The overheat protection of the EVCs will switch off the heating power, when exceeding the overtemp temperature (preset 300 °C for all EVC)

If a low temperature melting target is used, this degas procedure cannot be used. If necessary, degas the evaporator with an empty molybdenum crucible before installing the target

This procedure can also combine the degas of an empty crucible and the evaporator. First degas the evaporator without cooling, then degas the crucible with water cooling (see sec. 4.3).

4.3. Degassing a crucible

New crucibles should generally be degassed without filling, prior to the evaporation experiment. Especially during initial bakeout of a new crucible, very high impurity fluxes may occur. For degassing a crucible proceed as follows:

- Degas the evaporator (see sec. 4.2)
- Turn on the cooling water.
- Start heating the crucible as described in sec. 4.5 with a low heating power of about 5 to 10 W (2 to 5 W for small crucibles).
- Watch the pressure in the main chamber, in order to prevent internal HV break through. The pressure should not exceed $5 \cdot 10^{-6}$ mbar during degassing.
- **Watch the Flux monitor reading, if available!** A decreasing flux vs. time characteristics indicates that impurities are degassing, whereas a constant or increasing flux shows that bulk material is evaporating.
- Slowly increase the heating power. Take care not to exceed the maximum allowed temperature. The crucibles may not be degassed with the maximum allowed temperature if the evaporation temperature needed is quite low.



Above the limiting temperature (cf. table E1) the crucible material will start to evaporate or to chemically decompose

The different crucibles can be degassed with different maximum heating powers due to their maximum allowed temperature and size (compare with Fig. 5.1 and table E1).

For standard size W, Mo and Ta crucibles you may apply up to 150 Watts heating power, where Mo itself will start to evaporate weakly from the crucible. For small size crucibles use only 40 Watts for degassing. If the purity of the crucible material is insufficient it may need cleaning procedures as referred to in sec 4.4.

For PBN and the oxide crucibles only considerably lower heating powers may be applied, depending on the size of the crucibles. E.g. for a medium size crucible a degassing power of less than 30 W is recommended.

4.4. Degassing the evaporant

The procedure for degassing the evaporant is the same as for evaporation (see Sec. 5).

For degassing the evaporant it is recommended to begin with the linear motion set to its maximum length. Continuous water cooling is required during operation. The pressure should not exceed 10^{-6} mbar.

It is recommended for the first degas of a new evaporant to let the the shutter open (although it works with the shutter closed, too).

Start heating the evaporant until a small flux is obtained (typically some nA for rod evaporation or some 10 nA for crucible evaporation). Gradually increase the heating power to obtain the evaporation temperature needed; whilst doing this maintain observation of the pressure rise in the vacuum chamber and control the flux monitor.

Note: Gas or contaminations desorbing from the evaporant and the inner walls of the cooling shroud are indicated by the flux monitor. Such impurities show a decreasing flux vs. time, whereas the evaporant material yields a stable or increasing flux. This initial degassing can take several hours. Generally the cleanness of evaporants will improve even after several days of operation

If the purity of your evaporant material ("as bought") is insufficient, it may be worthwhile to apply a combination of thermal and chemical cleaning procedure. Refer, e.g., to :

R.G. Musket et al., "Preparation of Atomically Clean Surfaces of Selected Elements: A Review", Appl. Surf. Sci. 10 (1982) 143-207.

Although these procedures are applied to surfaces, many of them, such as heating in oxygen in order to remove carbon impurities, will also work well for the depletion of bulk material.

4.5. Evaporation

After sufficient degassing start with evaporation:

- Turn on the water cooling.
- Set the HV to about 600V to 800V and the filament current to about 1.5 A
- Slowly increase the filament current until emission starts and the flux monitor indicates an ion flux (positive polarity) in the 1 nA range.
- If no emission can be obtained it may be necessary to correct the evaporant position. If the sample is retracted too far, the space charge of electrons is not extracted from the filament and no emission current is shown (Note that the "emission-current" is the HV-current). Cautiously move the crucible or rod with the linear motion towards the filament. Take care not to touch the filament with the evaporant, because of the danger of destroying the filament. The position of a crucible needs not to be varied after that procedure whereas a rod shortens with evaporation and needs to be repositioned (see below).

- Slowly increase the heating power to obtain the flux needed. Wait always until the flux monitor reading stabilises. In case of a sudden increase of the flux monitor current immediately reduce the emission current.
- Especially when using crucibles the warming up time makes the reaction time slow. Take your time to find the correct settings for a new material!

The ion current is proportional to the evaporation rate and the emission current. At a given HV and sample position this current is directly proportional to the flux of evaporated atoms.

The operational conditions depend strongly on the evaporated material and on the choice of rod or crucible evaporation. Typical operational parameters are given in Appendix A. A typical value for the evaporation rate is about one monolayer per minute.

For crucible operation Fig. 4.1 shows typical temperatures reached with given heating powers. Together with the parameters given in App. A the needed heating power can be estimated.

For low-temperature materials such as C₆₀ or organic materials a small high voltage of only 100V or a few 100V may be appropriate. It may be necessary to increase the high voltage to 900V or 1000V for high-temperature materials such as tungsten or generally for rod-evaporation due to electrooptical properties of this geometry. Owing to space charge effects there will be a drastic increase in the emission current with the same filament current and thus in the heating power that can be obtained.

With small evaporation rates of about hundred Å per day one filling may last for months. Of course, rod-evaporation (diffuse in all directions) is less effective than crucible-evaporation (more directional) and also the evaporant volume of a rod is comparably small. However, since there is no crucible involved, the cleanliness of rod-evaporation is generally higher if one uses very clean material. In certain cases (e.g. Fe) crucible evaporation can be cleaner, because it is possible to clean the evaporant by melting it entirely.

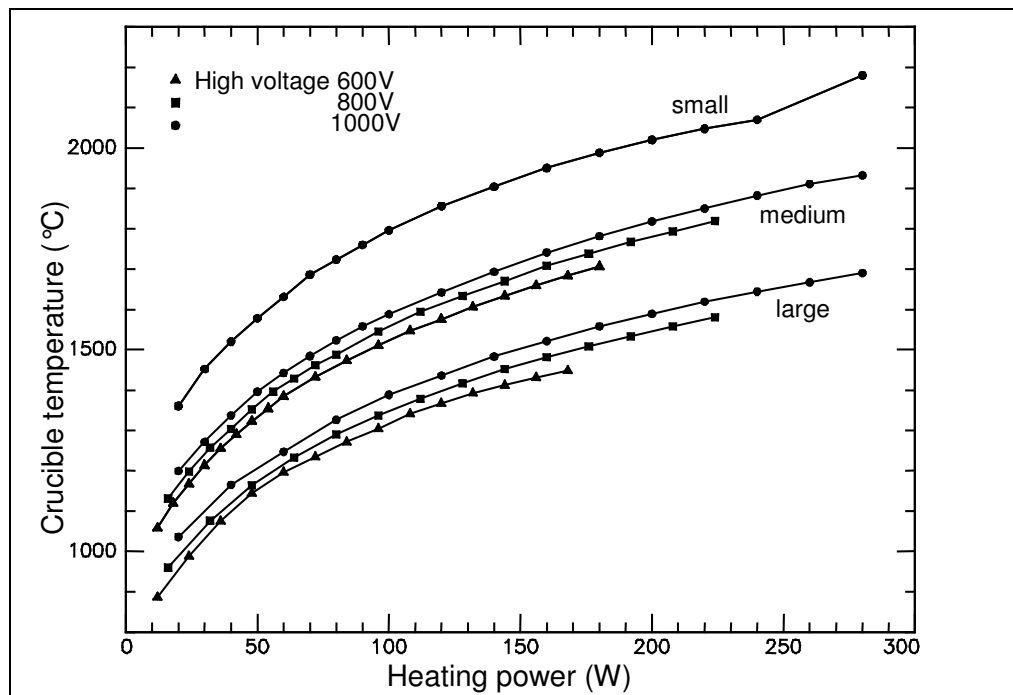


Fig. 4.1: Measured crucible temperatures of different crucible sizes with three different e-beam energies. For this measurement Mo-crucibles were used.

During the initial heating nearly all rods will melt at the tip thereby forming a ball of typically 2-3 mm dia. held by surface tension. Thus, a repositioning of the evaporant rod may be necessary immediately after the initial heating (melting of the tip) and subsequently after evaporation of typically several thousand Å. With extended use of a bar or rod evaporant, it will gradually shorten in length. This is recognisable by the fact that with a given emission current the high voltage has to be increased in order to obtain the same flux. In this case move the evaporant forward using the linear motion and control the parameters after each rotation of the actuator. This may be done keeping the evaporator running with the shutter closed for on-line control.

4.6. Evaporation with active flux regulation

This option is available for the power supply EVC 300/EVC 300s/EVC 300i.

Due to thermal drifts of the evaporator the flux of evaporant material will generally also show temporal variations. The Autoflux option eliminates such drift effects because the flux is actively regulated by means of a feedback loop between the flux monitor and the high voltage of the electron beam heater.

For operating the flux regulation proceed as follows:

1. Start the evaporation as described above. Wait until the desired value of flux current is obtained approximately. It is advantageous to start with a high voltage of about 600 - 800 Volts that the possible variation of the high voltage is large enough.
2. Press the AUTO-button. The presently flux is kept constant by means of altering the heating power via the high voltage.

For details see the corresponding manual of the power supply EVC 300x.

NOTE: For low-temperature evaporation in the few-hundred-°C-range a lower voltage can be appropriate. In any case, take care that the high voltage stays away from its upper limiting value of 1000V.

The Autoflux option may not work as described with ion beam assisted deposition. The focussing condition of the ions onto the sample and also the collected ion current at the flux monitor changes if the high voltage applied to the target changes, because then the mean ion energy changes as well. A reproducible flux regulation may then not be possible.

4.7. Ion beam assisted deposition with EFM 3i

There are two different modes for ion beam assisted deposition using

- self ions that are created from the metal vapour itself
- add-ions of the additional gas, e.g. noble gas atoms, that are provided by the gas inlet flange with a leak valve.

The ions are then focussed onto the sample using the ion lens; the flux monitor measures the remaining fraction of the . Proceed as follows:

- Connect the ion lens (SHV-feedthrough **Lens**) to the lens voltage at the EVC300i

- Connect the flux monitor (SHV-feedthrough **FM**) to Flux IN at the EVC300i.
- Start with evaporation as described. Allow additional gas for ion production as necessary.
- Set the lens voltage to 75%-80% of the high voltage applied to the evaporant to maximize the ion current reaching the sample. Watch the sample current.

Pulsed ion beam

A pulsed ion beam can be obtained by switching the lens voltage from its optimum focusing condition (75-80%) to its minimal value (ca. 10V). Then the ion beam is not focused and only a very small fraction of the initial ion flux reaches the sample.

4.8. Suppression of ions during evaporation with EFM 3s

The ion suppressor lens (EFM 3s) can be used to prevent all “self ions” created in the evaporation region from reaching the sample. For this operation mode switch on the ion suppressor (“SUPR ON” with EVC 300s). A voltage of about 1200 V is applied to the suppressor electrode to prevent all ions passing the flux monitor.



Attention: The ion flux may differ from the value measured with “SUPR OFF”, because of the deflected ions.

4.9. Suppression of ions with EFM 3i

The ion lens (EFM 3i) can also be used as ion suppressor to prevent all “self ions” created in the evaporation region. For this operation mode the ion lens and the flux monitor electrodes use are interchanged. A voltage of about 110% of the e-beam voltage is applied to the flux monitor electrode to prevent all ions leaving the flux monitor. The ion flux is measured at the lens electrode.

Proceed as follows:

- Connect the ion lens electrode (SHV-feedthrough **Lens**) to Flux In at the EVC 300i
- Connect the flux monitor electrode (SHV-feedthrough **FM**) to Lens at the EVC 300i.
- Set the lens voltage to about 110% of the heating voltage.
- Start evaporation; all ions are suppressed (no sample current measurable).



Attention: The ion flux may differ from the value measured with “SUPR OFF”, because of the deflected ions.

5. Maintenance

In all cases the power supply must be switched off and disconnected from the evaporator. The evaporator is removed from the chamber.

5.1. Cleaning

When evaporating only small amounts of material (e.g. from monolayer production), no cleaning of the cooling shroud or shutter is required on changing the evaporant. The cooling facility keeps contamination negligible.

There is very little maintenance necessary on the EFM evaporator. However it may be occasionally necessary to change a broken filament or inspect and replace a ceramic insulator which has become coated with conducting material. Also, after prolonged use all inner parts including the flux monitor at the front of the oven may become covered with material, such that mechanical cleaning becomes necessary (small metal brush or sandpaper recommended). After evaporation of large amounts of material the inner region of the copper cooling shroud should be cleaned after removal of the top flange (procedure see 5.2) using a small brush or similar device. Especially, thin sheets of loose evaporant must be carefully removed from the inner region.

EFM 2/3/3s/4/3i/3T

The ceramic insulators of the slide bearing need inspection (see fig. 3.1) if a resistance of $<1\text{M}\Omega$ is measured between the HV-feedthrough and ground.

Every filament is isolated from ground potential by two small ceramics which may also be contaminated by the evaporant. This can gradually lead to leakage currents. If no sufficient emission current can be reached, the filament need to be replaced.

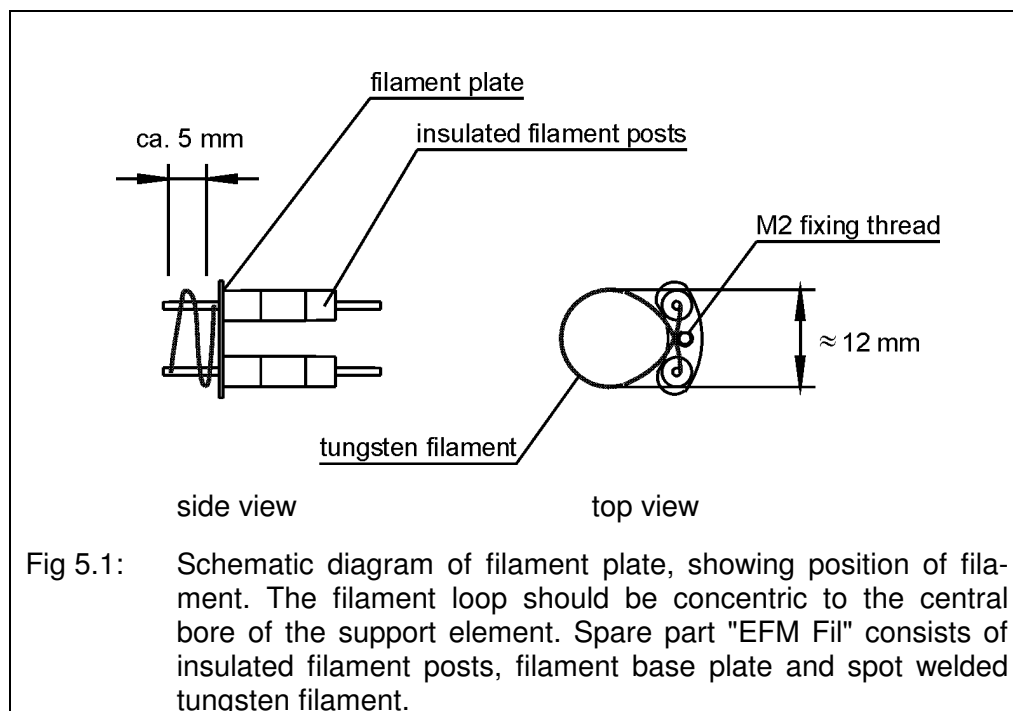
5.2. Filament Exchange

An excessive heating current, strong mechanical strain (shock, impact) or normal wear after long-time usage may destroy the filament. For replacement either a new filament with holder may be obtained from the manufacturer or the repair/replacement may be done on-site with a precision spot welder by spot welding a new filament. The filament wire is W99/Th1, \varnothing 0.125 mm. It can be ordered e.g. at Goodfellow Ltd. (Cambridge), Part Number W 145300. In the following, the procedure for both is given.

Changing the filament of EFM 2/3/3s/4:

Demounting the filament holder:

1. Loosen the two collets which connect the current leads to the ceramic feedthroughs of the filament holder at the front of the cooling body (Allen wrench 1.5 mm).
2. Loosen the 8 M3 bolts of the front copper flange on the cooling body. Note that the front copper flange can be tight after extended use. It may be necessary to use a small screw driver or a razor blade to loosen it. Move the shutter and both current leads with their holding plates some mm away from the front flange. The flux monitor (stainless steel cylinder at the front of the furnace) may remain screwed to the copper flange. Its flexible cable connection can also stay attached.
3. Cautiously remove the copper flange from the furnace body until free access is gained to the filament. Avoid strong forces being applied to the flexible cable connection.
4. Loosen the filament plate from the copper flange by loosening the M2 holding screw.
5. This applies only for the case of do-it-yourself repair of the filament:
Spot weld a spiral filament (tungsten of 0.125 mm thickness, length = 50-55 mm, preferably thoriated wire) to the holding bars of the filament holder. The tungsten wire must be bent into a spiral of 12-14 mm diameter with a pitch of about 5 mm. Keep to the drawing of fig. 5.1.
The filament posts are made of molybdenum. For spot-welding a piece of tantalum ribbon/thickness about 0.1 mm should be positioned between the filament wire and the post.



Remounting a new or repaired filament :

6. Cautiously replace the new or repaired filament in its mounting on the copper flange. In doing so, touching the filament must be avoided because of the danger of weakening the spot weld connections. Screw the filament holder by means of the M2 screw from the outside of the flange, however, do not tighten it yet.

7. Check the filament mounting according to fig. 5.1.
8. Return the copper flange onto the cooling body, avoiding damaging the filament. Fix the copper flange, the cable holding plates and the shutter bearing plate by means of the M3 screws.
9. Then tighten the M2 holding screw of the filament holder. Fix the connection wires by means of the collets. Hold the collets while fastening the M2 screws so as not to break the fragile ceramics of the filament holder. Check for possible short-circuits from filament to ground using an ohmmeter.

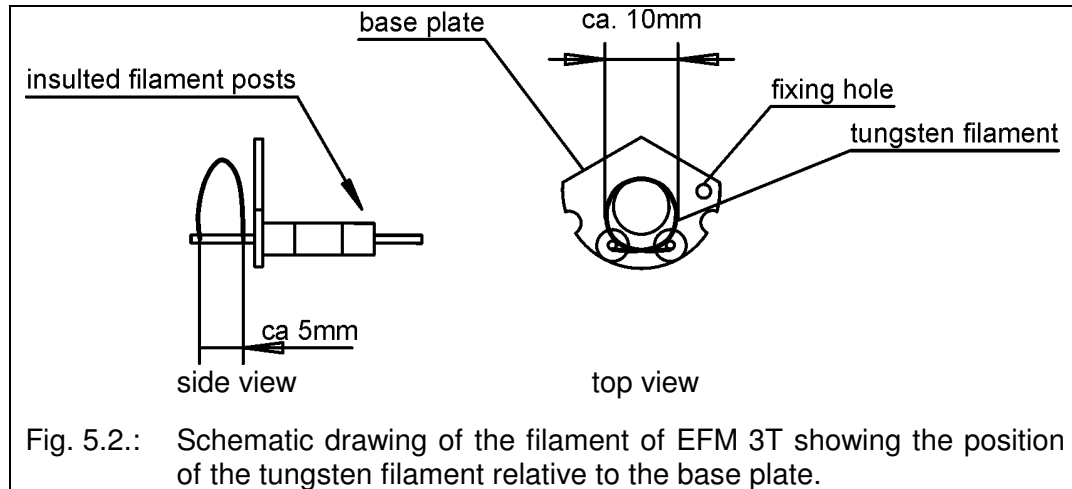
A new filament should be "tuned" in vacuum by gradually increasing the current from 1 A upwards (up to its operating current) over a period of about 10 min.

Changing the filament of EFM 3T:

1. Loosen the six collets which connect the current leads to the ceramic feedthroughs of the filament holders at the front of the cooling body (allen wrench 0.8 mm).
2. Remove the shutter bearing at the top of the flux-monitor (M2 screws).
3. Unplug the flux monitor cables at the feedthrough.
4. Loosen the M3 bolts of the front copper flange on the cooling body. Note that the front copper flange can be tight after extended use. It may be necessary to use a small screw driver or a razor blade to loosen it.
5. Move the shutter and the filament current leads with their holding plates some mm away from the front flange. Take care not to brake the ceramics with the filament current leads.
6. Remove the two screws on the lower side of the copper cooling shroud. Lift the copper flange fluxmonitor assembly with the flux monitor cabling. The filament plates are now assessable.
7. Remove the fixing screw of the filament plate, which needs inspection. Take care not to brake a still intact filament.
8. This applies only for the case of do-it-yourself repair of the filament:
Spot weld a spiral filament (thoriated tungsten of 0.125 mm thickness, see above, length = ca. 45 mm) to the holding bars of the filament holder. The tungsten wire must be bent into a spiral of 8-10 mm diameter with a pitch of about 4 mm. Keep to the drawing of fig. 5.2. The insulated filament posts are made of molybdenum. For spot-welding a piece of tantalum ribbon (thickness about 0.1 mm) should be positioned between the filament wire and the post (alternatively use the old cleaned Ta-ribbon) .
9. Check that all filaments make no short to the main cooling body. To do this, set the filaments to their original positions on the cooling body and make a visual inspection (use appropriate illumination) that the filament wires do not touch the cooling shroud from inside. Check the resistance between filament and ground.
10. Cautiously replace the new or repaired filaments in their mounts on the copper flange. In doing so, touching the filament must be avoided because of the danger of weakening the spot weld connections. Refit the fixing screw of the filament plate. Carefully replace the copper flange onto the cooling body.

- Fix the copper flange the cable holding plates and the shutter bearing plate by means of the M3 screws. Fix the current leads by means of the collets. Hold the collets while fastening the M2 screws so as not to break the fragile ceramics of the filament holder.

A new filament should be "tuned" in vacuum by gradually increasing the current from 1 A upwards (up to its operating current) over a period of about 10 min.

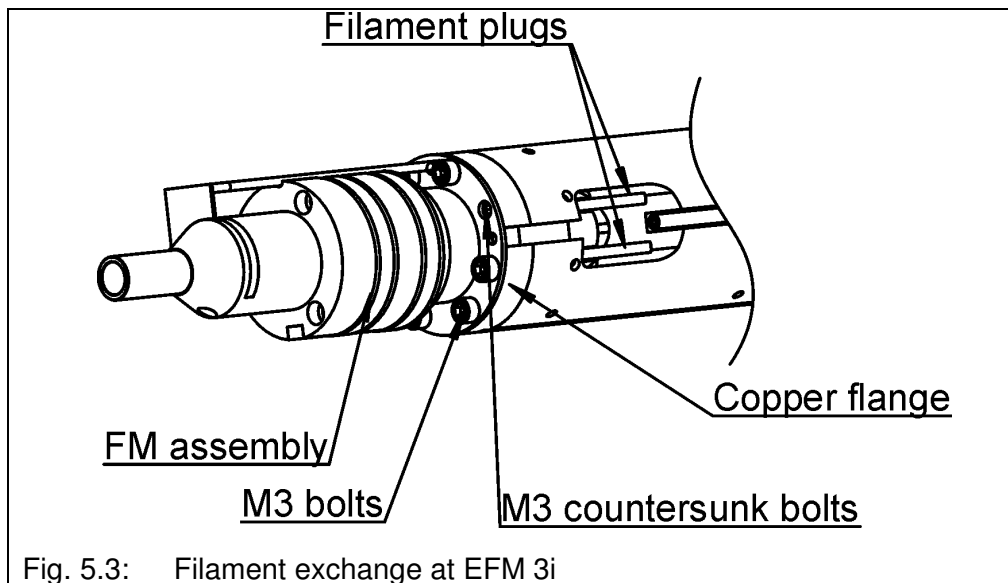


Before May 2004, the filaments have one insulated filament post. The second filament lead is directly connected to ground. Evaporators delivered later have both filament posts insulated (see chapter 2.3).

Changing the filament of EFM 3i:

Demounting the filament holder:

- Unplug the two filament leads (see fig 5.3)
- Loosen and remove the 6 M3 bolts at the flux monitor assembly. Cautiously remove the flux monitor assembly while bending the flexible cable connection towards the side. Take care not to break the ceramic leads of the flux monitor and lens connections.
- Loosen and remove the 2 M3 countersunk bolts that fix the copper flange. Remove the copper flange to get access to the filament holder and base.
- Pull out the filament holder off the copper shroud.
- Loosen the filament plate holder from the copper filament holder by loosening the M2 holding screw.
- For filament repair see above.
- Fix the removed parts in reverse order.



5.3. Replacing the Evaporant Holder Ceramics

EFM 2/3/3s/4/3i

After prolonged use of the evaporator the insulating ceramic (see Fig. 3.1) between the HV evaporant holder and the isolated tube which surrounds it may become coated. This may result HV leak currents or finally in a high voltage breakdown. Replacement ceramics should be fitted then.

EFM 3T

After prolonged use of the evaporator the insulating ceramic tube between the molybdenum barrel connector and the cooling shroud can be coated (slide bearing ceramics in Fig. 3.3). To remove this ceramics carefully loosen the headless M2 screw in the cooling shroud and remove the ceramics. The ceramics can be cleaned with acid or by heating it. Alternatively a replacement ceramics can be obtained by the manufacturer.

6. Fault diagnostics

No filament current:

- Cables disconnected
- Filament broken. Filament check is made in the startup-procedure of the EVC-power supplies

No emission current (i.e. no HV-current)

- No filament current or filament current too small, typical values where emission starts are 1.8A for EFM 2/3/3s/3i/4 and 2A for EFM3T. Note that new filaments will emit at higher filament currents.
- HV not connected, electrons are not accelerated onto evaporant.
- Evaporant retracted too far inside EFM.

No high voltage

- There may be a short circuit of evaporant rod to ground or to filament. Check with ohmmeter at high voltage socket. If there is a short check concentricity of evaporant and its correct length according to sec. 3.1 and 3.2 (maybe it has been moved too far forward). Before switching on again disconnect the cable from EFM and rise the high voltage to 1 kV to see whether the electronics alone is all right; refer to the corresponding manuals.



Lethal high voltage is present at the power supplies EVC 100, EVC 100s, EVC 300, EVC 300s and EVC 300i !

No Flux Monitor current

- Short-circuit of ion collector, check that the resistance from BNC socket ("FM" in Fig. 2.2) to ground is larger than 2M Ω . Eventually clean inside of flux collector on outlet of evaporator.
- Temperature too low for evaporation

Wrong temperature display

- Check the thermovoltage directly between the pins of the electrical feedthrough at the evaporator (See Fig.2.4 and 2.5). Appendix B gives a conversion table to temperatures. Refer to the manual of the power supply used.
- Check that the pins for the thermovoltage have contact to ground.

Flux negative with old power supply

(EVC 100 SN 86 00 01 001 xxxx, EVC 300 SN 86 00 02 001 xxxx)

- This always occurs during heating up before true evaporation starts and an ion current can be measured.
- HV not connected, evaporant does not heat up.
- Filament emits electrons directly into the fluxmonitor tube. Check by looking into the fluxmonitor tube. Correct position of filament or replace filament (cf. 5.2).

Flux negative with new power supply

(EVC 300i SN 86 80 00 035 xxxx, EVC 300 SN 86 80 00 036 xxxx, EVC 300s SN 86 80 00 048 xxxx, EVC 100 SN 86 80 00 037 xxxx)

- Shortcut between filament and ground. The power supply uses a filament offset voltage of 5 V, which prevents the negative flux. A shortcut leads to a vanishing offset. The power supply is not in danger.

Appendix

A. Background for Material Selection and Evaporation Parameters

The EFM is a UHV evaporator and once degassed it will operate in the 10^{-10} mbar pressure range with all but the most refractory evaporants. The pressure in the atom beam of evaporant is of course much higher than this.

The rate of evaporation is a function of vapour pressure, tables of which as a function of temperature can be found in a number of vacuum handbooks etc. The vapour pressure typically required for deposition rates of monolayers per minute (the rates this evaporator is intended for) are between 10^{-2} and 10^{-4} mbar. At higher pressures the source is likely to arc-over. Another limit to the pressure is, of course, the melting point. Evaporant wires melt and can form a small blob on the end of the wire if the surface tension is sufficient. At temperatures below 800 °C the evaporation from bars may not work very well.

Some materials may react with the thoriated tungsten filament.

Deposition Rates and Choice of Evaporation Source: Crucible or Rod?

The equation governing evaporation fluxes from effusive low pressure sources is:

$$\text{Deposition rate [atoms/(cm}^2 \text{ s)]} \approx 8 \cdot 10^{21} \frac{p \cdot r^2}{L^2 \sqrt{M \cdot T}} \cdot$$

As this formula does not contain a sticking coefficient it is an arrival rate.

- r: radius of evaporant bar or rod (typically 1 mm)
- L: distance from tip of evaporant to substrate (typically 110-150mm);
That corresponds to a distance of 10 - 50mm from the end of EFM to the sample.
- M: molecular weight of evaporant (g)
- T: temperature of evaporant (K)
- p: vapour pressure (mbar) - which is a function of temperature for a given material.
Consult appropriate tables.

The leading term in this formula is the partial pressure of the material, because it can cover several orders of magnitude within the possible temperature range. Rod evaporation should be possible if a high enough pressure can be reached at the melting point or at lower temperatures. If the evaporant has to be molten to reach the deposition rates needed a crucible should be used.

The choice of evaporation rate depends upon the experimental conditions, such as background pressure, and experimental requirements of purity and duration of the experiment. In general evaporation from bars is preferred, as heating of a crucible will deliberate gas from it. Also the heat loss due to the increased surface is higher. For the selection of crucible materials the table from the Balzers AG is very helpful (see the chapter on literature)

The basic equation from which the above has been derived, can be found in:

L. Holland, W. Steckelmacher, J. Yarwood: Vacuum Manual, London 1974

Fig. A1 illustrates this formula for parameters typical for EFM as discussed in the examples below. These are theoretical results giving a good starting point for planning an experiment.

Example 1: Can aluminium give good evaporation rates in the EFM ?

First we estimate the flux at the melting point (highest temperature that a bar mounting can work at). For aluminium the parameters are: $T_m = 933$ K, $P = 5 \cdot 10^{-6}$ mbar, $M = 27$ g, $r = 1$ mm, $L = 125$ mm. To calculate the flux in terms of monolayers per second, we assume 10^{15} atoms per square centimetre and per layer and get a deposition rate of $2 \cdot 10^{-5}$ monolayers per second. This is a very slow almost unusable evaporation rate.

Because other values of r and L give also low evaporation rates, molten aluminium has to be used (i.e. aluminium above its melting point). At a temperature of e.g. $T = 1450$ K and a partial pressure of $p = 5 \cdot 10^{-2}$ mbar (and $r = 1$ mm, $L = 125$ mm) a flux of 0.1 monolayers per second is estimated. This is a useful evaporation rate and shows that for certain experiments this evaporator can be used for aluminium. As molten aluminium is very reactive, boron nitride crucibles are recommended. Heating the aluminium in a crucible must be done very slowly and carefully because it tends to give gas burst (due to its oxide coat). The aluminium must not cover the Mo-liner of the crucible because then it alloys and the purity of evaporation is degraded.

Example 2: Platinum

1) The evaporation rate (or better say arrival rate at the sample) R at the melting point of Pt (1770°C) is quite small. The calculation gives 0.1 monolayers per minute. The experiment shows that the actual rate is a little bit higher: up to 0.5 ML/minute.

If in the actual experiment this small evaporation rate is sufficient, we strongly recommend evaporation from a bar as this is the most clean evaporation method.

In case a higher evaporation rate is needed the following two methods are applicable:

2) A piece of Pt-wire (some centimetres in length and some tenth of a millimetre diameter) is wound around the end of a straight carbon¹ rod of about 1 to 2 mm diameter, and 4 cm length. On heating the rod by electron bombardment, the Pt will melt and form a little droplet at the end of the bar. As it is only a small amount of material it will not drop off. Because of the surface tension it can be heated above the melting temperature of Pt. To be sure the molten Pt accumulates at the tip of the bar the EFM should point downward.

3) If it is necessary to have high evaporation rates and the EFM should point upwards, a carbon crucible should be selected.

¹ Any additional material that is heated during the evaporation, will degas itself and degrade the cleanliness of evaporation. This is especially the case with carbon. So it is best to avoid this situation. If it cannot be avoided take the minimum amount of carbon that is possible, so a rod is preferred compared to a graphite crucible.

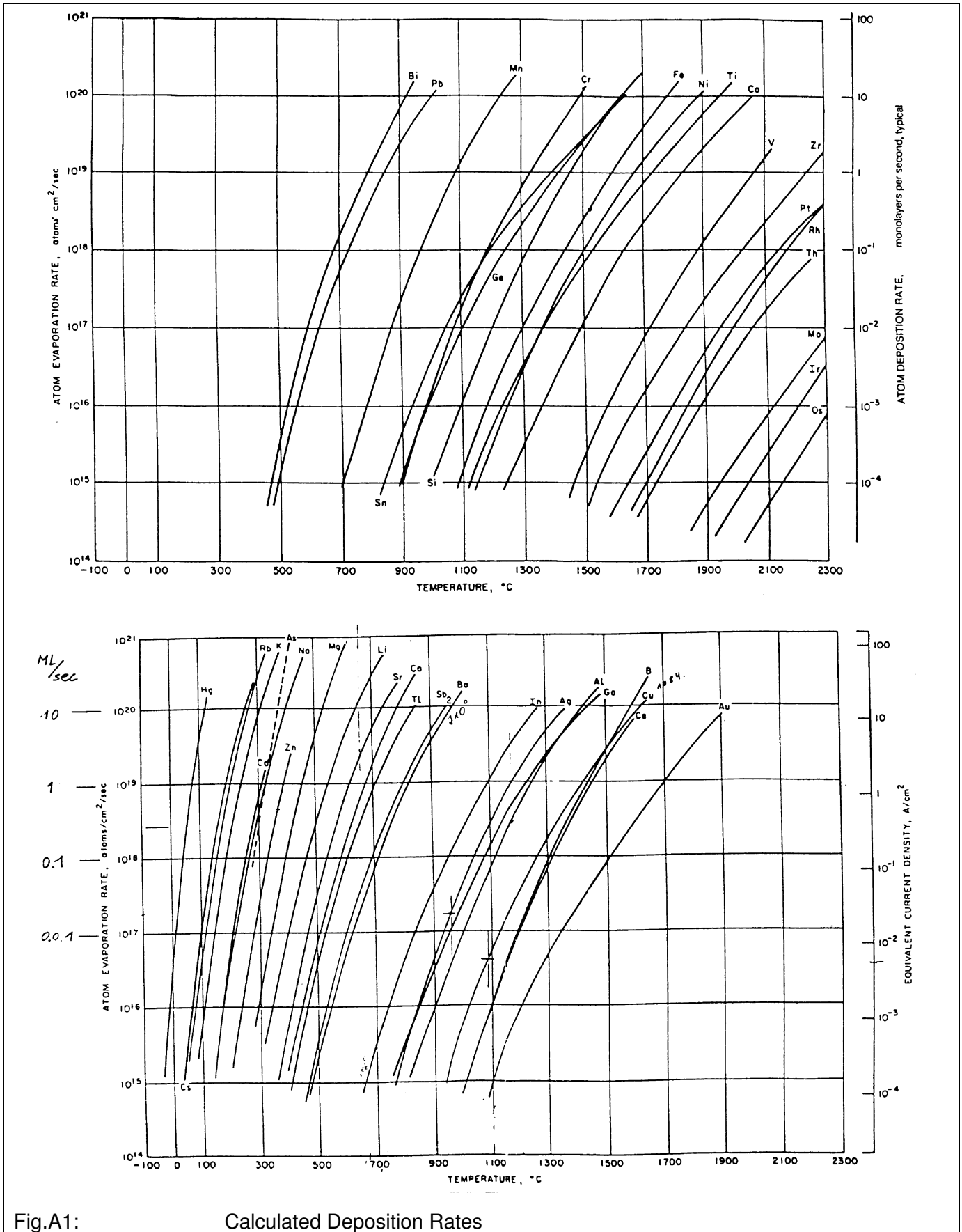


Fig.A1: Calculated Deposition Rates

Material overview

Brief overview about material deposition techniques

Material	Symbol	Melting temp./°C	Density g/cm ³	Vapour press./torr			E-beam suitability*	Crucible	Rod evaporation possible**	Comments
				10E ⁻⁸	10E ⁻⁶	10E ⁻⁴				
Aluminum	Al	660	2.70	677	821	1010	Ex	PBN	N	Wets crucibles. Fill crucible to max. 10% to avoid creeping
Aluminum Oxide	Al ₂ O ₃	2072	3.97	-	-	1550	Ex	-	Y	Sapphire excellent in E-beam; forms smooth, hard films.
Barium	Ba	725	3.51	545	627	735	F	Metals	< 10E ⁻⁶ torr	Wets without alloying, reacts with ceramics.
Beryllium	Be	1278	1.85	710	878	1000	Ex	BeO, C	< 10E ⁻² torr	Wets W/Mo/Ta. Powder and oxides toxic. Evaporates easily.
Bismuth	Bi	271	9.80	330	410	520	Ex	Al ₂ O ₃	N	Toxic vapor. Resistivity high.
Boron	B	2300	2.34	1278	1548	1797	Ex	-	Y	Explodes with rapid cooling. Forms carbides easily.
Boron Carbide	B ₄ C	2350	2.52	2500	2580	2650	Ex	C	N	
Cadmium	Cd	321	8.64	64	120	180	P	Al ₂ O ₃	N	Bad for vacuum systems. Low sticking coefficient.
Carbon (Graphite)	C	~3652	1.8 ⁻² .1	1657	1867	2137	Ex	-	Y	E-beam preferred. Poor film adhesion.
Cerium	Ce	798	~6.70	970	1150	1380	G	Al ₂ O ₃ , BeO, W	N	
Chromium	Cr	1857	7.20	837	977	1157	G	W	Y	Films very adherent. High rates possible.
Chromium Oxide	Cr ₂ O ₃	2266	5.21	-	-	~2000	G	-	Y	Disproportionates to lower oxides; reoxidizes at 600°C in air.
Cobalt	Co	1495	8.9	850	990	1200	Ex	BeO	< 10E ⁻² torr	Alloys with refractory metals.
Copper	Cu	1083	8.92	727	857	1017	Ex	Al ₂ O ₃ , Mo, Ta	< 10E ⁻⁴ torr	Adhesion poor. Use interlayer (Cr). Evaporates using any source material.
Dysprosium	Dy	1412	8.55	625	750	900	G	W	Y	-
Erbium	Er	1529	9.07	650	775	930	G	-	Y	-
Europium	Eu	822	5.24	280	360	480	F	Al ₂ O ₃	< 10E ⁻² torr	Low tantalum solubility.
Gadolinium	Gd	1313	7.90	760	900	1175	Ex	Mo, W	< 10E ⁻² torr	High tantalum solubility.
Gallium	Ga	30	5.90	619	742	907	G	PBN	N	May condense at crucible exit.
Germanium	Ge	937	5.35	812	957	1167	Ex	W, C,	< 10E ⁻⁶	Excellent films from E-beam guns.

								Ta	torr	
Gold	Au	1064	19.32	807	947	1132	Ex	PBN, Al ₂ O ₃	< 10E ⁻⁶ torr	reacts with W in e-beam guns. Films soft, not very adherent.
Hafnium	Hf	2227	13.31	2160	2250	----	G	C	< 10E ⁻⁵ torr	
Holmium	Ho	1474	8.80	650	770	950	G		Y	-
Inconel	Ni/Cr/ Fe	1425	8.5	-	-	-	G		Y	Low rate required for smooth films
Indium	In	157	7.30	487	597	742	Ex	Mo, PBN	N	Wets tungsten . Use molybdenum or ceramic crucible. May condense at crucible exit.
Iridium	Ir	2410	22.42	1850	2080	2380	F	-	Y	-
Iron	Fe	1535	7.86	858	998	1180	Ex	Al ₂ O ₃ , BeO	< 10E ⁻⁴ torr	Al ₂ O ₃ below melting point, BeO above melting point. Films hard, smooth. Preheat gently to outgas.
Lanthanum	La	921	6.15	990	1212	1388	Ex	W, Ta	N	Films will burn in air if scraped.
Lead	Pb	328	11.34	342	427	497	Ex	W, Mo	N	Toxic.
Lead Telluride	PbTe	917	8.16	780	910	1050	-	Mo, Pt, Ta	< 10E ⁻⁶ torr	Vapors toxic. Deposits are tellurium rich.
Lithium	Li	179	0.53	227	307	407	G	Al ₂ O ₃ , SS, PBN	Y	Reacting quickly in air, fill under protective atmosphere
Lutetium	Lu	1663	9.84	-	-	1300	Ex	-	Y	-
Manganese	Mn	1244	7.20	507	572	647	G	-	Y	-
Molybdenum	Mo	2610	10.2	1592	1822	2117	Ex	-	Y	Films smooth, hard. Careful degas required.
Molybdenum Oxide	MoO ₃	795	4.69	-	-	~900	-	Mo, Pt	N	Slight oxygen loss.
Neodymium	Nd	1021	7.01	731	871	1062	Ex	Ta	< 10E ⁻⁵ torr	Low tantalum solubility.
Nichrome IV	Ni/Cr	1395	8.50	847	987	1217	Ex	-	Y	Alloys with refractory metals.
Nickel	Ni	1455	8.90	927	1072	1262	Ex	W	< 10E ⁻² torr	Alloys with refractory metals. Forms smooth adherent films.
Niobium	Nb	2468	8.57	1728	1977	2287	Ex	-	Y	Attacks tungsten source.
Osmium	Os	2700	22.48	2170	2430	2760	F	-	Y	-
Palladium	Pd	1554	12.02	842	992	1192	Ex	-	< 10E ⁻² torr	Alloys with refractory metals. Rapid evaporation suggested.
Permalloy	Ni/Fe	1395	8.7	947	1047	1307	G	W	< 10E ⁻⁴ torr	Film low in nickel.
Platinum	Pt	1772	21.45	1292	1492	1747	Ex	C	< 10E ⁻⁴ torr	Alloys with metals. Films soft, poor adhesion.
Praseodymium	Pr	931	6.77	800	950	1150	G	Ta	< 10E ⁻⁶ torr	-
Rhenium	Re	3180	20.53	1928	2207	2571	P	C	N	Fine wire will self-evaporate.

Rhodium	Rh	1966	12.4	1277	1472	1707	G	-	Y	E-beam gun preferred.
Ruthenium	Ru	2310	12.3	1780	1990	2260	P	W	N	-
Samarium	Sm	1074	7.52	373	460	573	G	-	Y	-
Scandium	Sc	1541	2.99	714	837	1002	Ex	-	Y	Alloys with tantalum.
Selenium	Se	217	4,2	89	125	170	G	Mo, Ta, Al ₂ O ₃	Y	
Silicon	Si	1410	2.32	992	1147	1337	F	Ta,G, PBN	< 10E ⁻² torr	-
Silver	Ag	962	10.5	847	958	1105	Ex	W, Mo, Ta	< 10E ⁻⁴ torr	Rod evaporation with W-wire wound around the silver rod.
Strontium	Sr	769	2.6	239	309	403	P	W, Ta, Mo	N	Wets but does not alloy with refractory metals. May react in air.
Tantalum	Ta	2996	16.6	1960	2240	2590	Ex	-	Y	Forms good films.
Terbium	Tb	1356	8.23	800	950	1150	Ex	W	Y	-
Thorium	Th	1875	11.7	1430	1660	1925	Ex	W, Ta,Mo	Y	Toxic, radioactive.
Thulium	Tm	1545	9.32	461	554	680	G	-	Y	-
Tin	Sn	232	7.28	682	807	997	Ex	Ta, PBN	N	Wets molybdenum. Use tantalum or PBN in E-beam guns.
Titanium	Ti	1660	4.5	1067	1235	1453	Ex	-	Y	Alloys with refractory metals; outgas gently.
Tungsten	W	3410	19.35	2117	2407	2757	G	-	Y	Forms volatile oxides. Films hard and adherent.
Uranium	U	1132	19.05	1132	1327	1582	G	Mo, W	N	Films oxidize.
Vanadium	V	1890	5.96	1162	1332	1547	Ex	W	Y	Wets molybdenum. E-beam-evaporated films preferred.
Ytterbium	Yb	819	6.96	520	590	690	G	-	Y	-
Yttrium	Y	1522	4.47	830	973	1157	Ex	-	Y	High tantalum solubility.
Zirconium	Zr	1852	6.49	1477	1702	1987	Ex	C	< 10E ⁻⁶ torr	Alloys with tungsten. Films oxidize readily.

* Ex = Excellent, G = Good, F = Fair, P = Poor

** Y = rod always possible, N = not recommended due to melting point or e-beam suitability. Otherwise recommended pressure limit is given.

NOTE:	<p>This information has been collected to give customers a brief overview about material deposition techniques. The provider of this information is not responsible for the performance of any evaporation process which is developed based on the informations provided in this table.</p> <p>Some materials in this table are hazardous. Determine toxicity, flammability radioactivity or any other risks for health, environment or system components before using.</p>
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For some materials experimental hints for evaporation with the EFM evaporators are additionally given below. The provider of this information is not responsible for the performance of the described evaporation process in this table.

Pb, Cu, Ag, Au:	W or Mo crucible, Cu gives max values exceeding 10 ML/ sec., Cu wets Mo. Ag: high voltage 800V, emission 15-20 mA, flux 1-10 μ A Au: I_{Fij} = 2A, I_E =20 mA, U_{HV} = 750 V flux approx. 900nA. Attention: reacts with W under high e-beam current. Films soft, not very adherent. alternatively : Au wire 1 mm diameter, supported by spiral of tungsten, wire 0.25mm dia. at 800V, 2mA
Fe, Co:	bar 2 mm \varnothing , 800 V, 5-10 mA, 20 nA of ion current giving about 1 ML/minute alternatively: BeO-crucible (ideal) or ZrO ₂ -crucible
W:	wire 1-1.5 mm dia.: 900 V, 50-70 mA, flux up to 3 μ A, above about 2 μ A molten material may drop from the tip
Al:	pBN crucible, 800 V, 25 mA
C60 or other low melting point (T<500°C) materials	stainless steel crucible with nozzle, high voltage typ. 200 V
Si:	rod \varnothing 2 mm or a nearly square package of wafer strips 500 V, 40-50 mA yields 20 nm in 3 h This is just one example, a much higher flux is attainable. A supply source for rods of Si (either square or circular cross section) is: Holm, Gigerenz 1, D - 84367 Tann (Niederbayern), Tel: +49-8572/91213
Nd, Ce, Gd	Mo-crucible, fill under nitrogen atmosphere in glove box.

Note that rare earth materials (as some other materials) yield extremely high flux-monitor currents due to their anomalous high impact collision ionisation cross sections.

B. List of Thermovoltage

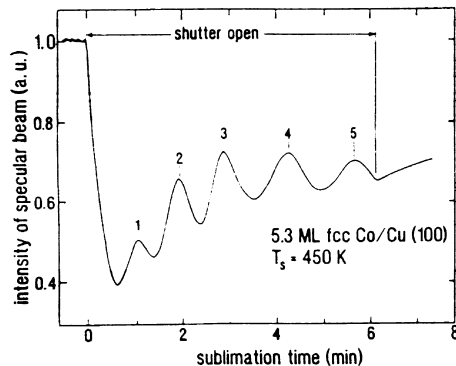
Nickelchromium (NiCr) / coppernickel (CuNi), type E (steps by 10)

Reference temperature 0 °C

°C	μV	°C	μV	°C	μV
-270	-9835	160	10501	590	44278
-260	-9797	170	11222	600	45085
-250	-9719	180	11949	610	45891
-240	-9604	190	12681	620	46697
-230	-9455	200	13419	630	47502
-220	-9274	210	14161	640	48306
-210	-9063	220	14909	650	49109
-200	-8824	230	15661	660	49911
-190	-8561	240	16417	670	50713
-180	-8273	250	17178	680	51513
-170	-7963	260	17942	690	52312
-160	-7631	270	18710	700	53110
-150	-7279	280	19481	710	53907
-140	-6907	290	20256	720	54703
-130	-6516	300	21033	730	55498
-120	-6107	310	21814	740	56291
-110	-5680	320	22597	750	57083
-100	-5237	330	23383	760	57873
-90	-4777	340	24171	770	58663
-80	-4301	350	24961	780	59451
-70	-3811	360	25754	790	60237
-60	-3306	370	26549	800	61022
-50	-2787	380	27345	810	61806
-40	-2254	390	28143	820	62588
-30	-1709	400	28943	830	63368
-20	-1151	410	29744	840	64147
-10	-581	420	30546	850	64924
0	0	430	31350	860	65700
10	591	440	32155	870	66473
20	1192	450	32960	880	67245
30	1801	460	33767	890	68015
40	2419	470	34574	900	68783
50	3047	480	35382	910	69549
60	3683	490	36190	920	70313
70	4329	500	36999	930	71075
80	4983	510	37808	940	71835
90	5646	520	38617	950	72593
100	6317	530	39426	960	73350
110	6996	540	40236	970	74104
120	7683	550	41045	980	74857
130	8377	560	41853	990	75608
140	9078	570	42662	1000	76358
150	9787	580	43470		

C. Calibration of Flux Monitor

a) MEED assembly:



MEED (Medium Energy Electron Diffraction) curve measured for the epitaxial system cobalt on copper (100). Specular (0,0) beam at grazing incidence as a function of evaporation time. The equidistant oscillations indicate layer-by-layer growth. The evaporation rate was kept constant using the flux monitor.

Ref.: C.M. Schneider, J.J. de Miguel, P. Bressler, J. Garbe, S. Ferrer, R. Miranda and J. Kirschner, Journal de Physique C8, 1657 (1988)

b) Quartz thickness monitor

c) RHEED

d) SPA-RHEED (= Spot Profile-Analysis Rheed)

e) Conductivity measurement

see M. Henzler, W Göpel: Oberflächenphysik des Festkörpers, Stuttgart 1991, p.535

f) Hall Effect

see W.D. King et al, J. Vac. Sci. Technol. **B9** (1991) 2771

g) Ellipsometry

Meyer, F.: Ellipsometric Study of Adsorption Complexes on Silicon Surf.Sci. 27 (1971) 107

Habraken, F. et.al.: Ellipsometry of Clean Surfaces, Submonolayers and Monolayer Films, Surf.Sci. 96 (1980) 482

h) Dynamic Optical Reflectivity In Situ (DORIS)

III - Vs Review, Val 5, p. 40:

Real-time monitoring of the growth of AlGaAs layers by dynamic optical reflectivity

T. Farrell, J.V. Armstrong, P. Kightley: Dynamic Optical Reflectivity, Appl.Phys.Lett. **59** (10) 1991, 1203 - 1205

J.V. Armstrong et.al., Appl.Phys.Lett 61 (23) (1992) 2770

for details on instrumentation contact Omicron

D. Literature

General literature

- [1] S. Schiller, U. Heisig: Bedampfungstechnik, Berlin 1975
- [2] M.v. Ardenne, Tabelle zur Angewandten Physik Bd II B, Berlin 1975³
- [3] S. Dushman: Scientific Foundations of Vacuum Technique; New York, London 1962
- [4] Balzers AG: Aufdampf- und Zerstäubungs- Materialien, Verdampfungsquellen, Lichtenstein, 1990
- [5] Balzers AG: Coating Materials and Sources Selector Guide.
- [6] Jeffrey Y. Tsao: Materials Fundamentals of Molecular Beam Epitaxy, Academic Press 1992

IBAD evaporation with EFM 3i

- [7] J. Kirschner, H. Engelhard and D. Hartung, Rev. Sci. Instrum., Vol. 73, No. 11, Nov. 2002.
(Description of EFM 3i)

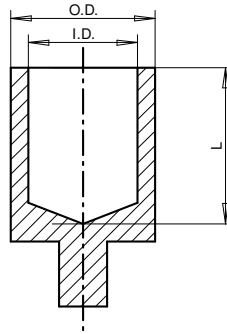
and references therein.

E. Options

E 1. Crucibles

Dimensions of crucibles

* can be used with EFM 3T and EFM 3i



	I.D./mm	O.D./mm	L / mm	capacity / mm ³	T _{max} (°C) @10 ⁻⁴ Torr vapor pressure	order code
Tungsten*, small	4,0	6.0	6.5	75	2750	C W S
Tungsten*, medium	6.0	8.0	9.0	250	2750	C W M
Tungsten, large	8.0	10.0	12.0	600	2750	C W L
Mo small*	4.0	6.0	6.5	75	2120	C Mo S
Mo medium*	6.0	8.0	9.0	250	2120	C Mo M
Mo large(esp. for EFM 4)	8.0	10.0	12.0	600	2120	C Mo L
Ta small*	4.0	6.0	6.5	75	2590	C Ta S
Ta medium*	6.0	8.0	9.0	250	2590	C Ta M
Ta large	8.0	10.0	12.0	600	2590	C Ta L
Al ₂ O ₃ small*	3.0	7.5	5.5	60	1320	C HA S
Al ₂ O ₃ medium	5.0	9.0	8.0	150	1320	C HA M
Al ₂ O ₃ large	6	10	10	280	1320	C HA L
Al ₂ O ₃ x-large	10	11	13	650	1320	C HA XL
Silica s,m,l on request					1025	C Q xx
Silica x-large	8	11	15	630	1025	C Q XL
Pyrolytic BN medium for 3T/I *	5.5	8.0	8.0	190	1600	C BN S
Pyrolytic BN medium	5.5	8.5	8.0	190	1600	C BN M
Pyrolytic BN large	8	11	12	600	1600	C BN L

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Graphite small*	4.0	6.0	6.5	75	>1800	C Gr S
Graphite medium*	4.5	6.0	8.0	125	>1800	C Gr M
Graphite large*	6.0	8.0	9.0	300	>1800	C Gr L
Graphite x-large	9.0	11.0	12.0	700	>1800	C Gr XL
Stainless Steel (St/St)* with removeable nozzle (i.d. 1mm)	5.0	7.0	8.0	150	800	C SS
Beryllium Oxide (BeO) small*	4.0	8.0	7	85	1900	C BO S
Beryllium Oxide (BeO), medium	6.0	10.0	7	190	1900	C BO M
Zirconium Oxide (ZrO ₂)*	4.5	8.0	7.6	110	n.a.	C ZrO

Notes:

PBN-crucibles: For temperatures above 1300° the heating of the PBN results in a raising N₂ background pressure

BeO-crucibles: **WARNING! Safety precautions must be followed**

Beryllium oxide ceramics are harmless as supplied but should be handled with care. A potential health hazard may exist if secondary operations which can generate dust or fumes are carried out. It is recommended not to machine the beryllium oxide crucibles.

Ceramic crucibles: The ceramic crucibles consists of a ceramic insert with a Mo-liner. This must be considered for evaporation of materials with a tendency to react or alloy with molybdenum. Then the crucible should not be heated too fast and must not be filled to a large extend.

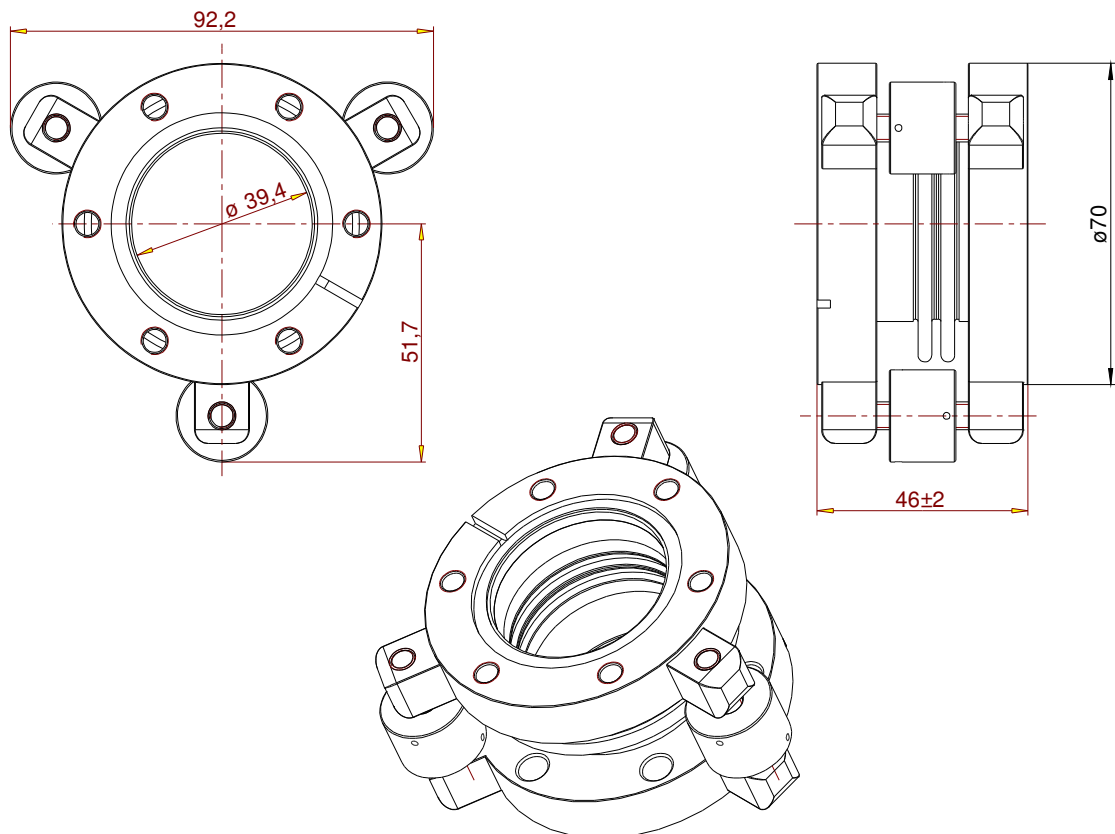
E 2. Shutter Motor

The shutter motor is either mounted at the factory or can easily be refitted by the customer. It serves to operate the shutter for all EFM evaporators including EFM 3T.

The shutter electronics is integrated in the evaporator power supply EVC 300, EVC 300s or EVC300i, which can be driven from RS232 interface of a computer. See related EVC manual for details.

Order Code: EFM SM

E 3. Port Aligner



$\pm 3^\circ$ type $^\circ L = 46\text{mm}$

Order Code: PA3

E 4. Elongated target shift (50mm)

The standard target shift is 25mm.

With rod evaporation 25mm longer rods, wires or bars can be used with the elongated target shift.

Order Code EFM LW

E 5. Barrel connectors for larger rods

Different barrel connectors are available to support the use of crucibles and rods with different diameter. The evaporators EFM2/3/3s/4, EFM 3i and EFM 3T use different models, please refer to the table:

Order Code	maximum rod diameter	material	use with	comment
EFM BC Mo1.5	1.5 mm	molybden with molybden screws	EFM2/3/3s/4 EFM 3i	standard barrel connector; it is supplied with the spare kit box
EFM BC Mo2.0	2.0 mm	molybden with molybden screws	EFM2/3/3s/4 EFM 3i	
EFM BC Mo4.0	4.0 mm	molybden with molybden screws	EFM2/3/3s/4 EFM 3i	
EFM BC Mo6.0	6.0 mm	molybden with molybden screws	EFM2/3/3s/4	
EFM BC SS1.5	1.5 mm	non magnetic stainless steel	EFM2/3/3s/4 EFM 3i	
EFM BC3T	1.5 mm	molybden with stainless steel screws	EFM 3T	

E 6. Quick-lock water hose connectors

To facilitate the connection and de-connection of the water hose for e.g. baking the system.

Order Code: EFM QWC

Service at OMICRON

Should your equipment **require service**

- Please **contact OMICRON** headquarters or your local OMICRON representative to discuss the problem. An up-to-date address list is available on our website under

<http://www.omicron.de/contact/representatives.html>

or via e-mail reply service under

contact.info@omicron.de

- Make sure all necessary information is supplied. Always **note the serial number(s)** of your instrument and related equipment (e.g. head, electronics, preamp...) of your instrument or have it at hand when calling.

If you have to **send any equipment back to OMICRON**

- Please contact **OMICRON headquarters** before shipping any equipment.
- Place the instrument in a polythene bag.
- **Reuse the original packaging and transport locks.**
- Take out a **transport insurance policy.**

For ALL vacuum equipment:

- Include a filled-in and signed copy of the "Declaration of Decontamination" form which can be found at the back of the equipment manual.



No repair of vacuum equipment without a legally binding signed decontamination declaration !

- Wear suitable cotton or polythene gloves when handling the equipment.
- **Re-insert all transport locks** (if applicable).
- Cover the instrument with aluminium foil and/or place it in a polythene bag. Make sure no dust or packaging materials can contaminate the instrument
- Make sure the **plastic transport cylinder** (if applicable) **is clean.**

Fix the instrument to its plastic cylinder (if applicable).

Service FAX Reply

To
OMICRON Nanotechnology GmbH

Test and Service Department
Limburger Str. 75
D - 65232 Taunusstein
Germany

Tel: +49 - 61 28 - 987-230
FAX: +49 - 61 28 - 987 33 230

From

.....
.....
.....
.....
.....
.....
Tel:
FAX:

Type of Instrument
Serial Number
Purchasing Date
(Last Service Date)

Problem:

Date:

Signature:

Decontamination Declaration

If performing repair or maintenance work on instruments which have come into contact with substances detrimental to health, please observe the relevant regulations.

If returning instruments to us for repair or maintenance work, please follow the instructions below:

- **Contaminated units** (radioactively, chemically etc.) must be decontaminated in accordance with the radiation protection regulations before they are returned.
- **Units returned** for repair or maintenance must bear a clearly visible note "free from harmful substances". This note must also be provided on the delivery note and accompanying letter.
- Please use the attached attestation declaration at the end of this manual.
- **"Harmful substances"** are defined in European Community Countries as "materials and preparations in accordance with the EEC Specification dated 18 September 1979, Article 2" and in the USA as "materials in accordance with the Code of Federal Regulations (CFR) 40 Part 173.240 Definition and Preparation".

No repair will be carried out without a legally binding signed declaration !

Declaration of Decontamination of Vacuum Equipment and Components

The repair and/or service of vacuum equipment/components can only be carried out if a correctly completed declaration has been submitted. **Non-completion will result in delay.** The manufacturer reserves the right to refuse acceptance of consignments submitted for repair or maintenance work where the declaration has been omitted.

This declaration may only be completed and signed by authorised and qualified staff.

1. Description of components

Type: _____ Serial No: _____

2. Reason for return _____

3. Equipment condition

Has the equipment ever come into contact with the following (e.g. gases, liquids, evaporation products, sputtering products...)

- | | | |
|--|------------------------------|-----------------------------|
| • toxic substances? | Yes <input type="checkbox"/> | No <input type="checkbox"/> |
| • corrosive substances ? | Yes <input type="checkbox"/> | No <input type="checkbox"/> |
| • microbiological substances (incl. sample material)? | Yes <input type="checkbox"/> | No <input type="checkbox"/> |
| • radioactive substances (incl. sample material)? | Yes <input type="checkbox"/> | No <input type="checkbox"/> |
| • ionising particles/radiation (α, β, γ , neutrons, ...)? | Yes <input type="checkbox"/> | No <input type="checkbox"/> |

For all harmful substances, gases and dangerous by-products which have come into contact with the vacuum equipment/components please list the following information on (a) separate sheet(s): trade name, product name, manufacturer, chemical name and symbol, danger class, precautions associated with substance, first aid measures in the event of an accident.

Is the equipment free from potentially harmful substances? Yes No

The manufacturer reserves the right to refuse any contaminated equipment / component without written evidence that such equipment/component has been decontaminated in the prescribed manner.

4. Decontamination Procedure

Please list **all harmful substances, gases and by-products** which have come into contact with the vacuum equipment/components together with the decontamination method used.

SUBSTANCE	DECONTAMINATION METHOD

(continue on a separate sheet if necessary)

5. Legally Binding Declaration

Organisation: _____

Address: _____

Tel.: _____ Fax: _____

Name: _____ Job title: _____

I hereby declare that the information supplied on this form is complete and accurate.

Date: _____ Signature: _____ Company stamp: