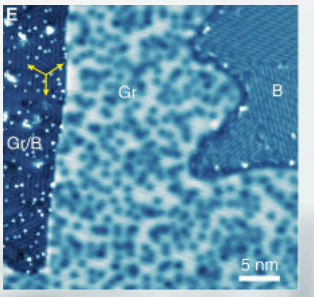
Electron beam evaporator with Flux Monitor

EFM Series

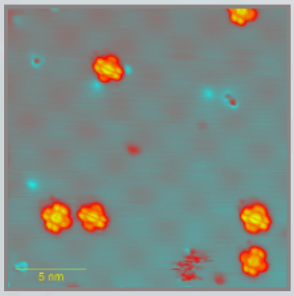
for Ultra-Pure Submonolayer and Multilayer Thin Film Growth

Graphene & Borophene-Graphene



H J Liu et al 2015 2D Mater. 2 034004
Physics Department, University of Hong Kong
doi.org/10.1088/2053-1583/2/3/034004

Organic Molecules

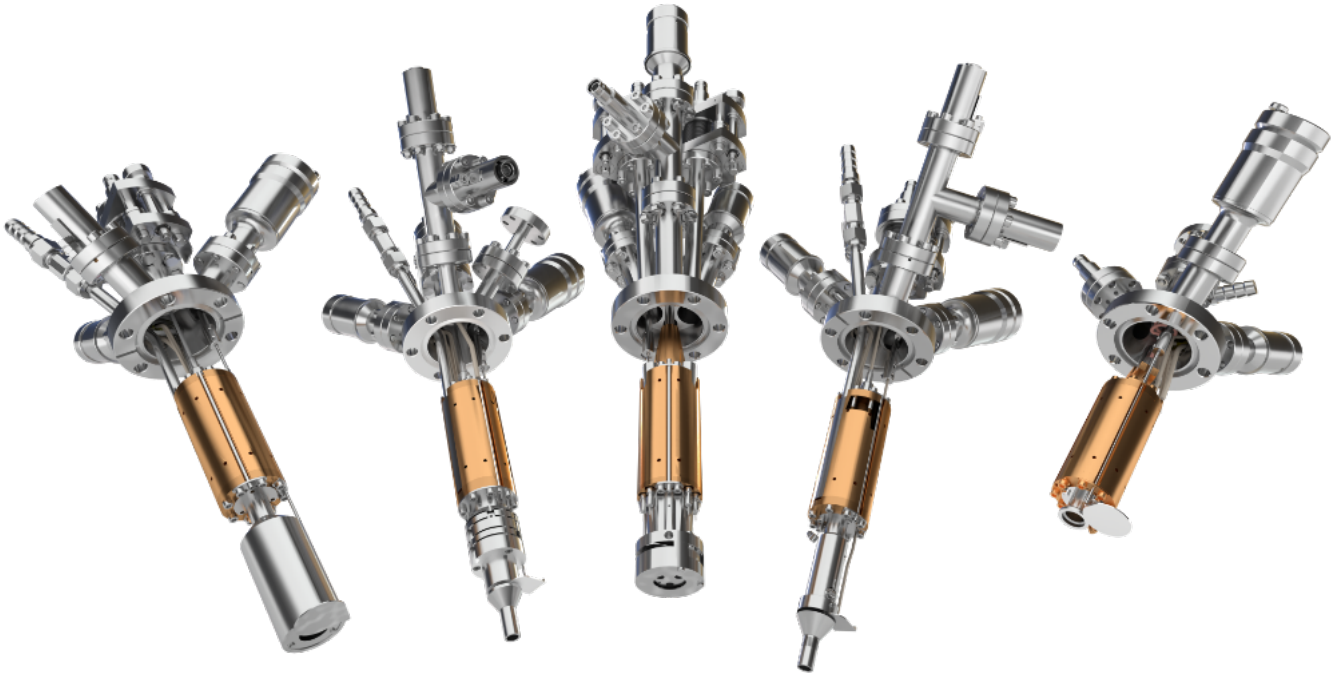


STM image of CuPc molecules (Copperphthalocyanine) on NaCl/Cu (100) @ 5 K.

Data acquired by the group of Prof. I. Swart, Debye Institute for Nanomaterials Science, Utrecht University, the Netherlands.

- Evaporation from Wires, Rods or Crucibles
- Temperature Range from 100°C up to 3300°C
- Integrated Flux Monitor
- Water Cooling for Minimum Pressure Rise
- Rear Loading for Alignment Preservation

FOCUS



THE

EFM PRODUCT RANGE

The FOCUS EFM e-beam evaporator originally has been designed for evaporation of magnetic materials such as Fe, Co, Cr, Mn and Ni as ultra-pure sub-monolayer and multilayer thin films. Materials like Pt, Ag, Au, Al, Ti, Ta, W and semiconductors, e.g. silicon, can be evaporated with the highest purity.

A careful choice of material composing the EFM, its proprietary cooling concept combined with an integrated and T/C controlled degassing up to 250°C enables evaporation in ultra-high vacuum (to below 10^{-10} mbar) and prevents cross-talk between sequentially evaporated substances.

This together with the robust and compact design and high quality manufacturing makes the EFM-series a valuable long term investment.

Hence the EFM-series evaporators EFM 2, EFM 3, EFM 3s, EFM 4, EFM 3T and EFM 3Ts are ideally suited for thin film evaporation and as doping cells in classical MBE-growth as well.

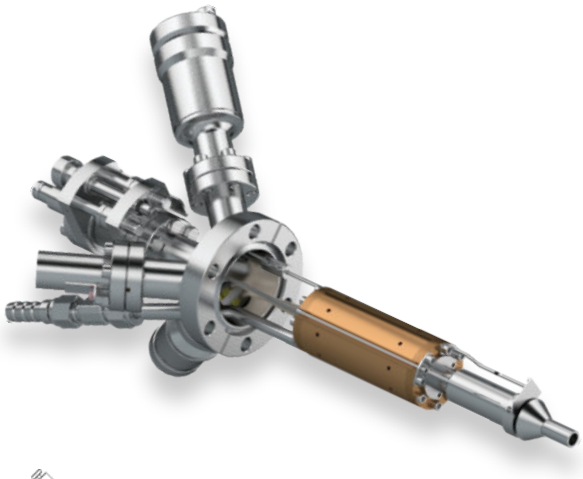
Today more than 1800 instruments are used in virtually every surface science research lab making sure that there is a set off parameters/recipes available for basically every evaporation request.
The EFM has been cited in several 1000 publications proving the scientific relevance of its dedicated design for surface science.

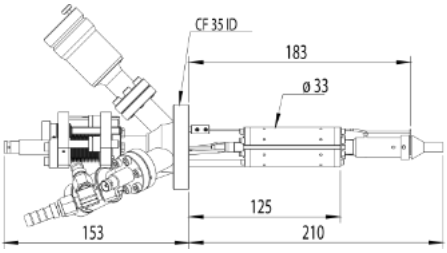
FOCUS provides service and supplies spare parts and support for all EFMs ever built since 1991.

The EFM 3 has been the first commercial evaporator using an integrated flux monitor. This enables reproducible real-time control of the deposition rate and, once calibrated, removes the need for a quartz thickness monitor. Start of evaporation is more accurately shown and controlled than by temperature measurement.

The high quality microprocessor controlled electronics provide stable and highly reproducible filament- and HV-regulation. All evaporation parameters are displayed on the control unit. By means of the PC software Epitass® the evaporation processes are completely remote controlled, if needed.

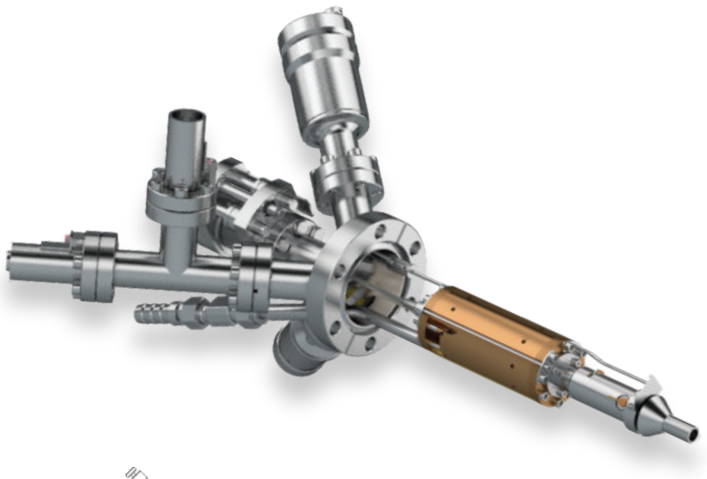
The rear-loading feature of the EFM evaporators enables crucible or rods to be exchanged without detaching the EFM from the vacuum chamber maintaining the source alignment. Low stray magnetic fields enable the evaporator to be used during e.g. RHEED or LEED analysis.

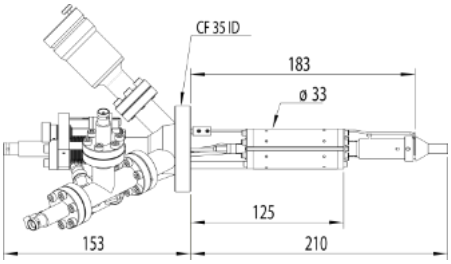




EFM 3 : The Classic

- Ultra-pure evaporation
- Temperature range: 100°C – 3300°C
- Fully bakeable up to 250°C
- Evaporation area Ø 4 – 20 mm
- Z-shift for material feed
- Rear-loading of evaporant
- Water cooling for minimum pressure rise
- Evaporation from rods and crucibles
- Integrated shutter
- Flux monitor
- Mounting flange DN 40 CF





EFM 3s : Suppressing Ions

- Evaporation area Ø 4 – 20 mm
- Flux monitor
- Integrated shutter
- Ion suppressor (neutral evaporant beam)
- Mounting flange DN 40 CF
- All other features same as EFM 3

As the very first family member the EFM 3 is designed for thin film growth and molecular beam epitaxy. Sub-monolayer and multilayer systems can be produced with evaporation rates varying from 1/10 monolayer per minute to several monolayers per second.

The precisely defined evaporant beam profile allows for highly uniform deposition on the sample (see Fig.4). The deposition area is determined by the choice of three different easily exchangeable exit apertures and the distance from the source to the sample. Integral part of the EFM 3 is a z-shift for material feed when using rods or simply to optimize the distance between the electron beam filament and material to be evaporated either from a rod or crucible.

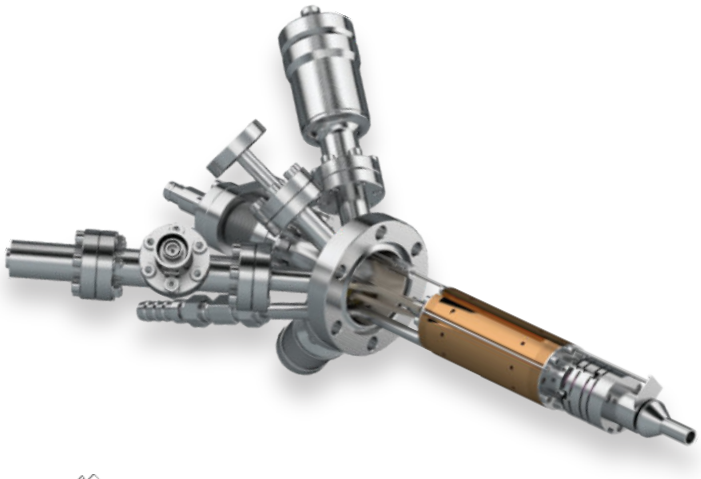
Integrated flux-monitor and shutter allow for a precise reproducibility of previous evaporation rates before the sample is exposed to the evaporant (even with shutter closed). In combination with the EVC 300/300s the flux monitor signal can be used to fully control the evaporation process by the integrated flux regulation.

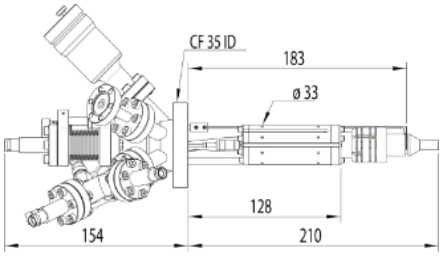
The EFM 3 can be combined with all EVC power supplies: EVC 100L, EVC 100s, EVC 300, EVC 300s and EVC 300i. All of the above holds for the entire product range in general.

The EFM 3s adds an additional electrode to the EFM 3.
A part of the evaporant beam in all products of the EFM-series is ionized by the electron bombardment during heating. Most of these ions are captured by the flux monitor electrode.

As the evaporant is on high voltage with respect to the grounded sample the remaining ions may create defects in the substrate surface and deposit energy. To generate a 100 % neutral beam an additional voltage is applied at the suppressor electrode of the EFM 3s. The additional voltage is supplied by the power supply EVC 300s or EVC 100s.

Upgrade packages for existing EFM 3 are available on request. All other features of the EFM 3 are fully preserved.





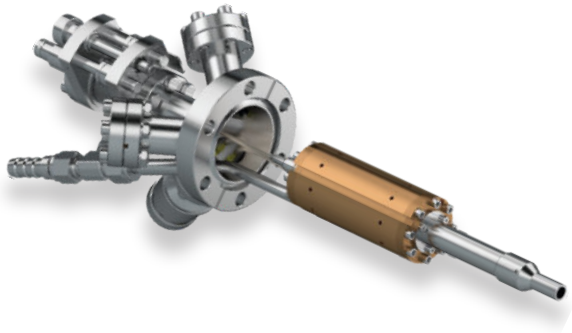
EFM 3i : Ion-Beam-Assisted Deposition

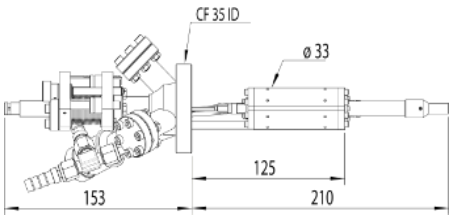
- Evaporation area Ø 4 – 20 mm
- Flux monitor
- Integrated shutter
- Gas inlet for additional rare gas ions
- Ion focusing lens for Ion-Beam-Assisted Deposition (IBAD)
- Ion suppression
- Mounting flange DN 40 CF
- All other features same as EFM 3

The EFM 3i is specifically designed to facilitate layer-by-layer growth in cases where it does not occur naturally. It allows for the controlled evaporation of the target material and the simultaneous generation of ions to create additional surface defects (Ion-Beam- Assisted Deposition (IBAD)). The ions can be produced either by an intrinsic process from the evaporated target material, or from inert gases with the help of an integrated gas inlet. The ions are focused onto the substrate by an electro-static lens. This focusing lens can adjust the ratio of ions to neutrals within the deposition area at the target and hence the additionally induced defect density.

Alternatively sensitive substrate materials can be protected against ion bombardment by a repelling lens voltage (see EFM3s). The dedicated EVC 300i power supply supports not only the evaporation process but also supplies the additional lens voltage and includes a sample current meter.

Due to the special design of the ionization region the max. crucible size is limited compared to the EFM 3.





EFM 2 : The Low Budget Solution

- Evaporation area Ø 4 – 20 mm
- Cost-effective
- No flux monitor
- No shutter
- upgradeable
- Mounting flange DN 40 CF
- All other features same as EFM 3

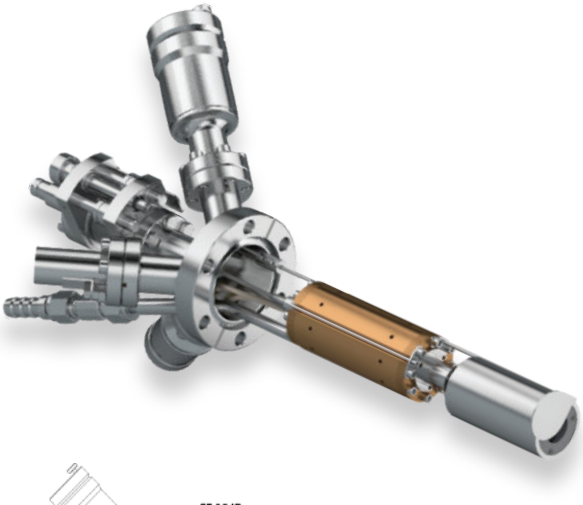
The EFM 2 is a low budget basic version of the EFM 3. It features the same proven capabilities in terms of cooling concept, purity, evaporation area, temperature range, reliability, quality, etc. as the EFM 3, but without the shutter and flux monitor.

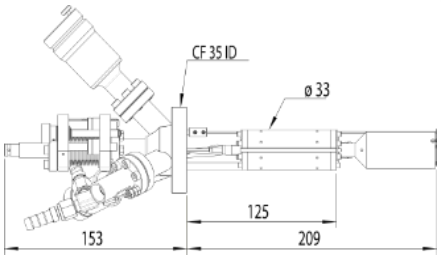
The dedicated EVC 100 L electronics is a robust analogue supply with 100 W output power and emission regulation for stable growth conditions.

Together both add up to a cost-effective evaporator package to cover almost the same material range as the EFM 3.

Shutter and flux monitor can easily be factory retrofitted. The EFM 2 accepts the same crucible sizes as the EFM 3. Also included is the z-shift for material feed when using rods or simply to optimize the distance between the electron source and the material to be evaporated either from a rod or crucible.

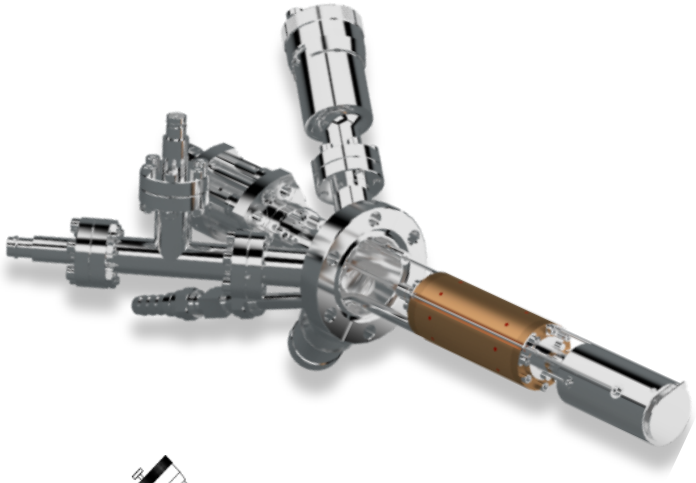
See also: J.Kirschner, H. Engelhard, and D. Hartung, Rev. Sci. Instrum., Vol. 73, No. 11, p. 3853-3860, 2002

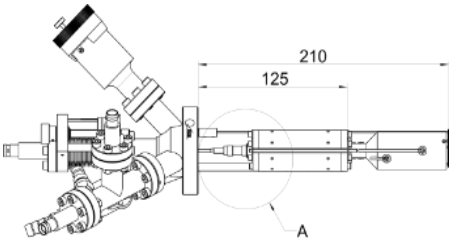




EFM 4 : For larger samples

- Evaporation area Ø 10 – 50 mm
- Flux monitor
- Integrated shutter
- Crucible capacitiy up to 700 mm^3
- Mounting flange DN 40 CF
- All other features same as EFM 3.





EFM 4s : Suppressing Ions

- Evaporation area Ø 10 – 50 mm
- Flux monitor
- Integrated shutter
- Ion Suppressor (neutral evaporant beam)
- Mounting flange DN 40 CF
- All other features same as EFM 4.

The EFM 4 provides the same features as the EFM 3 but is intended for the deposition on substrates with a larger diameter up to about 50 mm.

The three different exit apertures allow to adapt the evaporation area exactly to the size of the sample. Evaporation rates varying from 1/10 monolayer per minute to over 1000 monolayers per second can be achieved by selection of the appropriate crucible and e-beam power.

The EFM 4 is suitable for crucible capacities up to 700 mm^3.

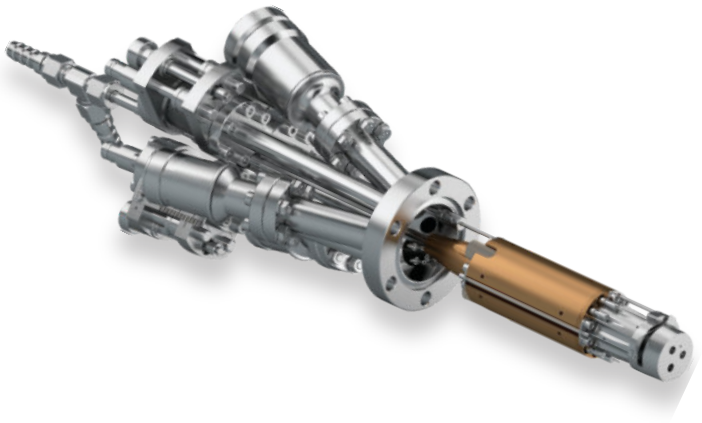
The effective water-cooling ensures low background pressure (typically in the 10^{-10} mbar range) even during prolonged operation at high evaporant temperatures.

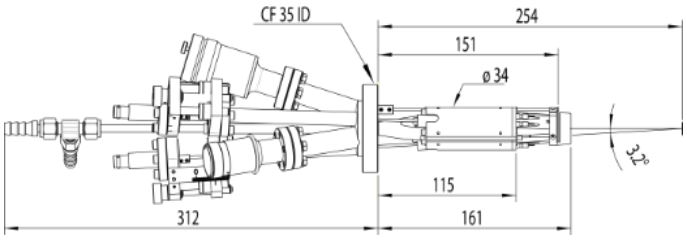
Due to the larger deposition areas being targeted the operation of the EFM 4 with the EVC 300 power supply is the most suitable configuration.

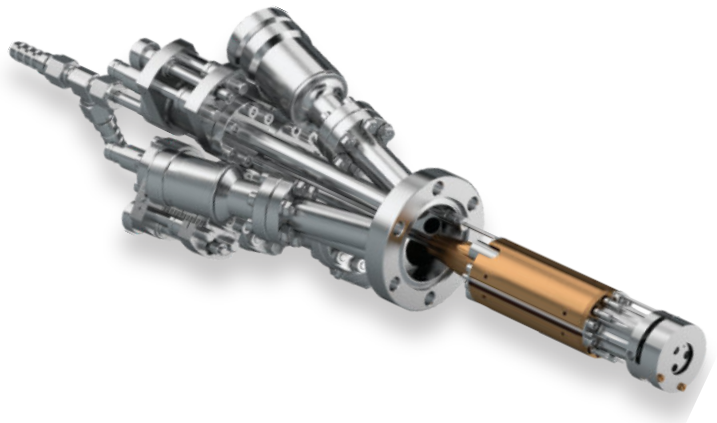
The EFM 4s adds an additional electrode to the EFM 4.
A part of the evaporant beam in all products of the EFM-series is ionized by the electron bombardment during heating. Most of these ions are captured by the flux monitor electrode.

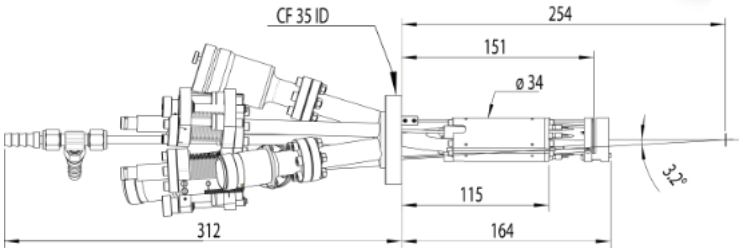
As the evaporant is on high voltage with respect to the grounded sample the remaining ions may create defects in the substrate surface and deposit energy. To generate a 100 % neutral beam an additional voltage is applied at the suppressor electrode of the EFM 4s. The additional voltage is supplied by the power supply EVC 300s or EVC 100s.

Upgrade packages for existing EFM 4 are available on request. All other features of the EFM 4 are fully preserved.









EFM 3T : Three independent sources at once

- Evaporation area Ø 8.5, 11 and 15 mm
- Working distance 93 mm (± 10 mm)
- 3 independent cells,filaments & flux monitors
- No crosstalk during compound growth
- Integrated multi-position shutter
- Mounting flange DN 40 CF
- All other features same as EFM 3

EFM 3Ts : Same but ions are suppressed

- Evaporation area Ø 8.5, 11 and 15 mm
- Working distance 93 mm (± 10 mm)
- 3 independent cells/filaments/ flux monitors
- No crosstalk during compound growth
- Flux monitor
- Integrated multi-position shutter
- Ion suppressor (neutral evaporant beam)
- Mounting flange DN 40 CF
- All other features same as EFM 3

Based on the design concept of the EFM 3 evaporators, the triple evaporator EFM 3T features three independent cells for the evaporation of a wide range of materials from wires, rods or crucibles.

The three individual cells have crossing beams at about 93 mm distance from the exit aperture (254 mm from the mounting flange) to ensure a maximum overlap of the evaporation area.
Each cell is equipped with a separate filament and HV supply to prevent crosstalk. Three independent flux monitors enable the controlled stoichiometric growth of compounds.
The integral multi-position shutter enables precise dosing and simultaneous or consecutive evaporation to produce e.g. super lattices or multilayers.
The latter is easily accomplished by means of the optional shutter motorization and the PC software tool Multi Epitass®.

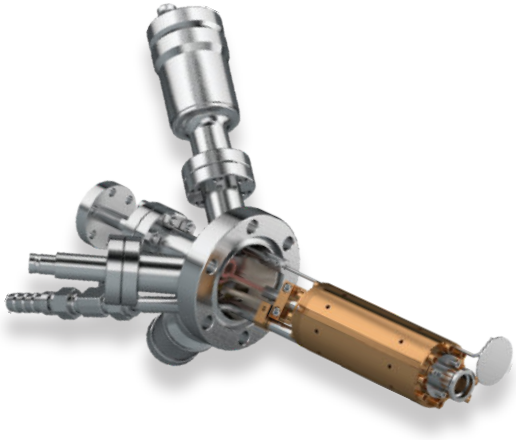
The deposition area is governed by the choice of one of three apertures. The three evaporation targets are independently mounted on three 25 mm z-shifts for alignment. This feature allows evaporation from rods with a multi-pocket instrument. The target materials can be easily refilled from the rear (3x DN 16 CF).

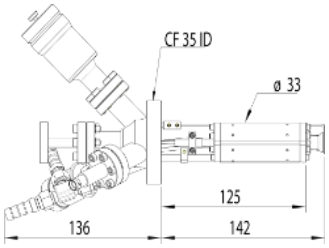
One power supply can operate all three cells sequentially. In case of co-evaporation each material requires a separate power supply.

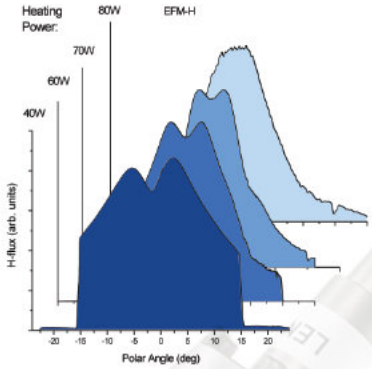
The EFM 3Ts adds an additional electrode to the EFM 3T. A part of the evaporant beam in all products of the EFM-series is ionized by the electron bombardment during heating. Most of these ions are captured by the flux monitor electrode. As the evaporant is on high voltage with respect to the grounded sample the remaining ions may create defects in the substrate surface and deposit energy.

To generate a 100 % neutral beam an additional voltage is applied at the exit of the EFM 3Ts which acts as an ion suppressor.

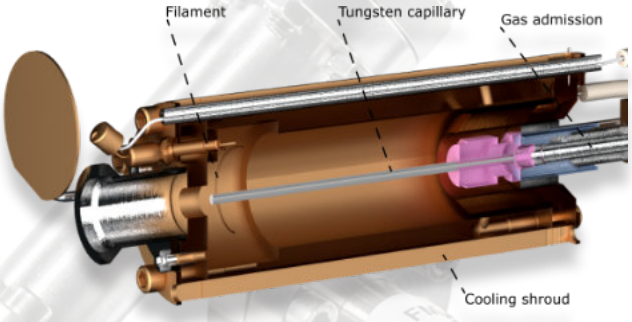
The additional voltage is supplied by the power supply EVC 300s or EVC 100s. In case of co-evaporation each material requires a separate power supply. Only one of these needs to have the s-configuration (EVC 300s, EVC 100s).







The EFM-3H is carefully designed to deliver a sharply defined beam profile. By adjusting the heating power, different spot profiles can be selected.



Schematic cross section of EFM-H

EFM-H : Atomic Hydrogen Source

- Effective water cooling
- FWHM: ± 15° to ± 6° (approx.)
- Flange to sample distance: 203 mm or larger
- Insertion depth: 141.5 mm
- Compatible with EVC Series Controllers
- Options: shutter, pumping bypass and ion suppressor (neutral beam)
- Mounting flange NW 40 CF

The EFM-H is a source to provide atomic hydrogen based on the design of the EFM 3. A flange NW 16 CF on the rear side is used for molecular hydrogen inlet including a pumping by-pass to clean the piping prior to H_2 disposal.

The EFM-H is an ideal instrument for the cleaning and etching of semiconductor surfaces (such as Si, GaAs, Ge or InP), for surface passivation, for improvement of thin film growth and other similar applications using atomic hydrogen.

The EFM-H features a cracking efficiency close to 100%, a smooth, flat and sharply defined spot profile, a low background pressure and a surprisingly low power consumption demonstrating the outstanding performance of the EFM-H.

The temperatures, heating power, and other parameters required for the thermal dissociation of H_2 molecules at a tungsten surface are very similar to those applied during operation of the well-known EFM evaporator. The crucible is replaced by a thin tungsten capillary, and Hydrogen is flown through that capillary. The tungsten capillary is then heated by electron beam bombardment.

The typical kinetic energy of the Hydrogen atoms produced is about 250 meV. Since the heated area is efficiently shielded by the EFM's distinctive copper cooling shroud, the level of outgassing is negligible.

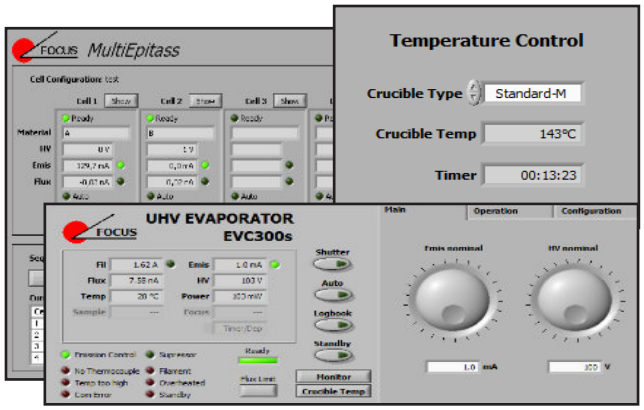
The geometry of the EFM-H enables excellent alignment of the hydrogen beam onto the sample, allowing for example an atomic hydrogen flux of $2x10^{15}cm^{-2}s^{-1}$ at a chamber background pressure of $2x10^{-6}$ mbar (mainly recombined hydrogen molecules).

Depending on the heating power (i.e. the temperature and length of the heated Tungsten area), the spot profile can be varied. Either a focused beam for small samples, or a wider beam for larger samples may be selected.

The EFM-H is compatible with all EVC power supplies and cable sets.

ELECTRONICS & SOFTWARE

The microprocessor controlled EVC power supplies make the operation of all EFM-type UHV evaporators very convenient and safe. With up to 300 W power output they are sufficient for the evaporation of any desired material.



All parameters of the EVC power supplies including those for the optional motorized shutter can be set manually on the front panel or via the Lab VIEW™*-based Epitass® software.

The EVC power supply provides a regulated filament emission current down to 1 mA in order to precisely regulate crucible temperatures down to 100°C e.g. for molecules (see front page). Between 100°C and 800°C the temperature stability is 0.1°C or better.

As a unique feature Epitass® software has a temperature display providing the actual crucible temperature.

The EVC 300-2 is powered with 2 kV for high flux evaporation from the rod of materials such as C, W, Ti and Ta.

The EVC 300 series power supplies come with full flux regulator in addition to the emission current regulator which can be programmed for constant flux or integral flux values.

The software logbook of Epitass® ensures a full documentation of the experiment and allows to re-load complete sets of parameters.

For Multilayer growth or co-evaporation from different cells several EVC power supplies need to be controlled. This functionality is provided by the MultiEpitass® software which can control up to four different cells mastering the individual Epitass® software of each EVC power supply. The MultiEpitass® is an option or part of an EFM 3T-EVC 300-2 package.

All evaporators including the EFM-H can be equipped with a motorized shutter which is either mounted at the factory or can easily be refitted by the customer.

The shutter electronics are integrated in all power supplies EVC 300-2 / 300s-2 / 300i. The shutter software control is an integral part of Epitass® and MultiEpitass®. The shutter motor is an ideal support for multi-layer growth and co-evaporation.

Other features:

- Highly reproducible closed loop flux regulation with adjustable parameters
- Regulated emission current
- Programmable automatic thin film deposition (as a function of exposure or time)
- Easy adjustment of operating parameters via user friendly menu structure
- Deposition parameters can be stored and recalled for repeated procedures
- Automatic growth based on control of the shutter position or the heating power
- Automated temperature monitoring of evaporator cooling

* LabView™ is a trade mark of National Instruments

CRUCIBLES

A wide selection of crucible materials and sizes ensures the optimum choice for each material and quantity of evaporant. All standard sizes listed in the table fit the EFM 2, EFM 3/3s and the EFM 4. The EFM 3T/3Ts and the EFM 3i only accept crucibles with outer diameter of ≤ 8 mm.

For evaporation from crucibles, the source should be mounted almost vertical or preferably pointing slightly upwards.

The crucible can be exchanged or refilled by just opening one DN 16 CF flange at the rear of the evaporator.

Special crucibles with respect to size and material are available on request.

	I.D. (mm)	O.D. (mm)	L (mm)	capacity (mm^3)	Tmax (°C) @10^{-4} Torr vapor pressure
Tungsten, small	4.0	6.0	6.5	75	2750
Tungsten, medium	6.0	8.0	9.0	250	2750
Tungsten, large	8.0	10.0	12.0	600	2750
Mo small	4.0	6.0	6.5	75	2120
Mo medium	6.0	8.0	9.0	250	2120
Mo large(esp. for EFM 4)	8.0	10.0	12.0	600	2120
Ta small	4.0	6.0	6.5	75	2590
Ta medium	6.0	8.0	9.0	250	2590
Ta large	8.0	10.0	12.0	600	2590
$Al2_0O_3$ small	3.0	7.5	5.5	60	1320
Al_2O_3 medium	5.0	9.0	8.0	150	1320
Al_2O_3 large	6.0	10.0	10.0	280	1320
Al_2O_3 x-large	10.0	11.0	13.0	650	1320
Silica s,m,l on re-quest					1025
Silica x-large	8.0	11.0	15.0	630	1025
Pyrolytic BN medium for 3T/3i	5.5	8.0	8.0	190	1600
Pyrolytic BN medium	5.5	8.5	8.0	190	1600
Pyrolytic BN large	8.0	11.0	12.0	600	1600
Graphite small	4.0	6.0	6.5	75	>1800
Graphite medium	4.5	6.0	8.0	125	>1800
Graphite large	6.0	8.0	9.0	300	>1800
Graphite x-large	9.0	11.0	12.0	700	>1800
Stainless Steel (St/St)with removeable nozzle (i.d. 1mm)	5.0	7.0	8.0	150	800
Beryllium Oxide (BeO) small	4.0	8.0	7.0	85	1900
Beryllium Oxide (BeO), medium	6.0	10.0	7.0	190	1900
Zirconium Oxide (ZrO_2)	4.5	8.0	7.6	110	n.a.

Mo-Barrel connectors are available for wires and rods up to 4 mm in diameter.

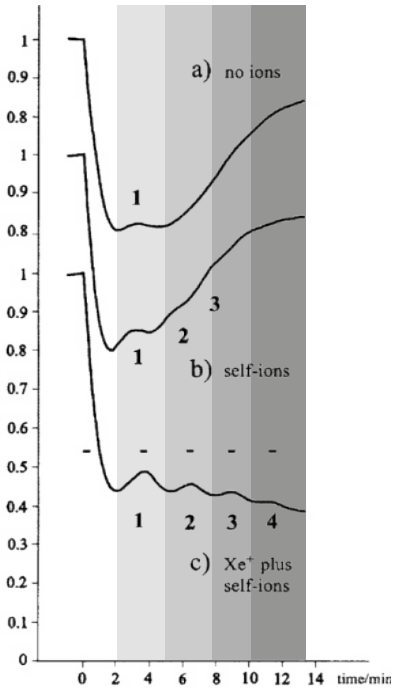


Fig. 1

EFM 3i : Improvement for layer by layer growth

Results for deposition of Co onto Cu(111) are given in panels (a) - (c). Shown is the intensity of the (00) electron beam using medium energy electron diffraction as a function of time after opening the shutter. The sample temperature is 80 °C in all cases.

a) Operation of the source in the MBE mode. There is only one minor maximum visible. This result is typical for three-dimensional growth.

b) Operation in the ion beam assisted deposition mode using self-ions. A number of oscillations are visible on a rising background, indicating an intermediate stage between three-dimensional growth and layer-by-layer growth.

c) Operation in the pulsed IBAD mode with Xe gas added. The ion beam is switched on for 25 s at the beginning and near each maximum, indicated by the short horizontal bars. A good layer-by-layer growth is achieved.

J.Kirschner, H. Engelhard, and D. Hartung, Rev. Sci. Instrum., Vol. 73, No. 11, November 2002

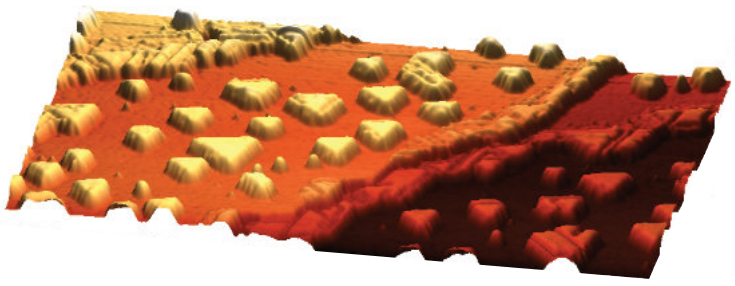


Fig. 2

Sub-monolayer of CaF_2 on Si(111)
Imaged with STM

P. Rahe, P. Moriarty (University of Nottingham)

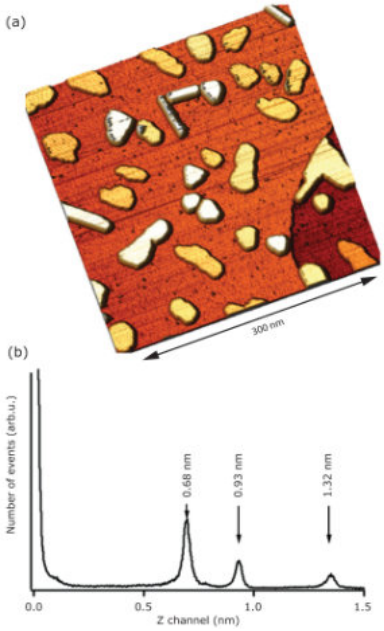


Fig. 3

(a) 3D view of the $Al_{13}Co_4(100)$ surface dosed with 2.6 ML of Bi (300 × 300 nm²).
(b) Height histogram of the Bi film showing the three specific island heights.

Ref.: S. Bobaru, É. Gaudry, M.-C. de Weerd, J. Ledieu, V. Fourneé, PHYSICAL REVIEW B 86, 214201 (2012)

EFM-Series Spot Profiles

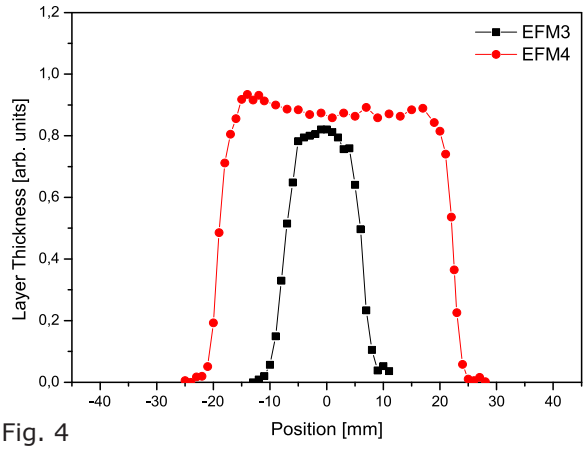


Fig. 4

EFM 3: Flat top diameter 10 mm,
(Nozzle - sample distance 90 mm,
Mo-crucible 8 mm diameter)

EFM 4: Flat top diameter 36 mm,
(Nozzle - sample distance 90 mm,
Mo-crucible 8 mm diameter)

Reproducable & Stable Flux Control

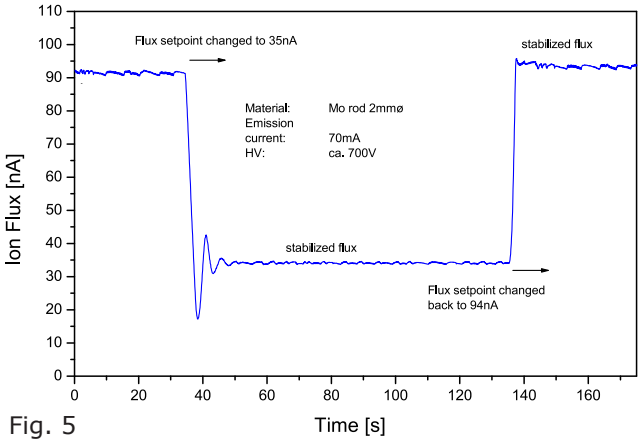


Fig. 5

Stable conditions:
The EVC 300 flux monitoring system provides precise control of the evaporant flux.
The regulation parameters can be adjusted to ensure excellent response even with rapid changes of the flux setting as demonstrated here.

Deposition area vs. distance to sample

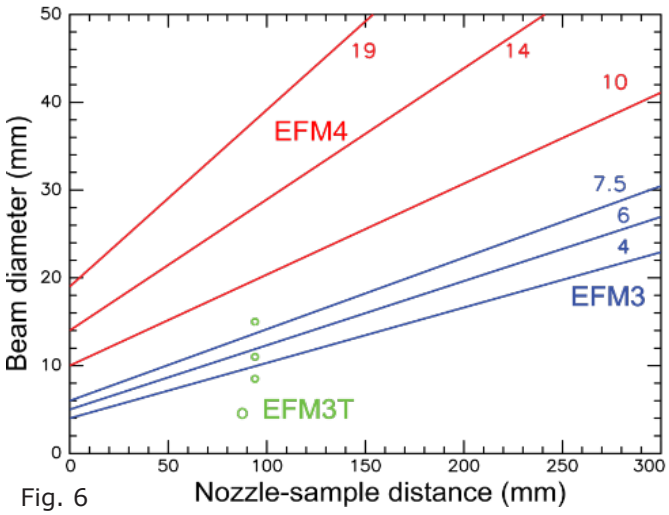


Fig. 6

Deposition area as a function of distance for three different aperture sizes for EFM 3, EFM 4 and EFM 3T.

Evaporant temperature in dependence of crucible size & heating power

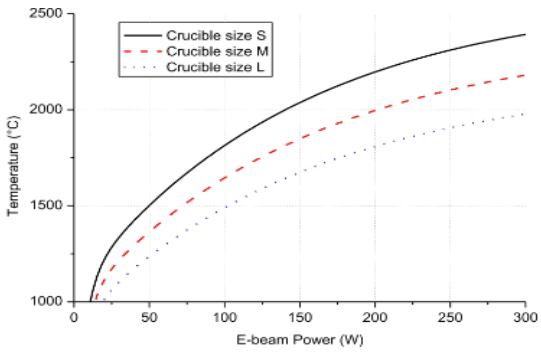


Fig. 4.1: Typical crucible temperatures (averaged) as measured for various high voltages. W-crucibles of given type were used.

Fig. 7

Crucible temperatures for different crucible sizes with three typical e-beam energies. For this measurement Mo-crucibles were used.

FOCUS GmbH develops and manufactures scientific instrumentation for Materials Research and advanced Electron Beam and Laser Welding systems. We are situated in Hünstetten, Germany and are close to Frankfurt airport. Since our formation in 1990, we have worked closely with the research community to collaborate on new innovative products. A dedicated team of electronic engineers, software engineers, designers and physicists ensures that a FOCUS product will exceed the expectations of high technology operators now and into the future.

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FOCUS GmbH, Neukirchner Str. 2,

D-65510 Huenstetten-Kesselbach,

Germany

Tel.: +49 (0) 6126 4014 0

E-Mail: sales@focus-gmbh.com