

TriCon electron bombardment evaporation source

User's manual

(version 3.1/May 9, 2000)

Dr. R. Verucchi

INFM, UdR of Modena

Read the Manual carefully!

This manual replaces any previous version!

The TriCon evaporation source is not an “ordinary” device, even if based on well known physical principles: its use without a careful reading of this manual will damage seriously both the source and the power supplies.

Take the time to read the manual, in order to understand what is different and “how-it-works”.

Operations with the TriCon source are not “more difficult” than in usual sources, they are simply “different”. So, please read carefully the manual and spend some minutes to verify how the new triode electrodes configuration has been realized making an inspection of the evaporation zone: this will help you to understand why some precautions are necessary and how high performances can be achieved.

The triode electrodes configuration applied in an evaporation source based on electron bombardment is protected by the **italian patent n. GE98 A 000096, deposited on November 1998, 18th** by INFIM and R. Verucchi.

Table of contents.

1. Theory of operation.....	2
1.1. The new TriCon concept.....	2
1.2. Performances.....	5
Source description.....	7
3. Installation.	12
3.1. Source-substrate distance and beam divergence.....	13
3.2. Electrical connections.....	15
4. Common procedures	16
4.1. Starting conditions.....	16
4.2. Water cooling.....	16
4.3. Turning on the hot filament.....	16
4.4. “Z ₀ - Zero level conditions”.....	18
4.5. Inspection of the evaporation zone: removing the stainless steel cap.....	18
4.6. Evaporating material cooling down.....	19
5. Evaporating material mounting.....	20
5.1. Rod.....	20
5.2. Crucible.....	21
5.3. Anode positioning and centering.....	21
6. Degassing procedure.....	23
6.1. Filament degassing.....	23
6.2. Grid degassing.....	24
6.3. Anode degassing.....	24
6.3.1.Evaporation from rod	24
6.3.2.Evaporation from crucible.....	24
6.4. Internal walls degassing.....	25
6.4.1.Evaporation from rod.....	25
6.4.2.Evaporation from crucible.....	25
7. Evaporation procedure.....	26
7.1. Shutter.....	26
7.2. Evaporation from rod.....	26

7.3. Evaporation from crucible.	27
8. Some practical suggestions.	29
9. Maintenance.....	30
9.1. Internal parts cleaning.....	30
9.2. Filament replacing.....	30
10. Troubleshooting.....	33
10.1. Rod tip melting.	33
10.2. Strange anode current.....	33
10.3. Low evaporation rates from rod.....	33

Symbol legend.

HV = High Voltage

V_F = hot filament voltage

I_F = hot filament current

V_G = grid voltage

I_G = grid current

V_A = anode (evaporating material) voltage

I_A = anode (evaporating material) current

UHV = Ultra High Vacuum

R = evaporating point-substrate distance

1. Theory of operation.

1.1. The new TriCon concept.

The evaporation in UHV of material (mainly metals) by means of electron bombardment is a well established technique. The main scheme of such a source is that of a vacuum diode (see Fig. 1): electrons are emitted from a hot filament (cathode) by thermionic effect and are accelerated towards a higher potential electrode (anode). The energy transfer by electron bombardment on the anode can lead to very high surface temperature, up to 3000K: if the material has very high vapor pressure (at least 10^{-4} , 10^{-3} Torr) before the melting point, it is possible to maintain the evaporating material in the solid state and obtain good evaporation rates from a relatively simple device.

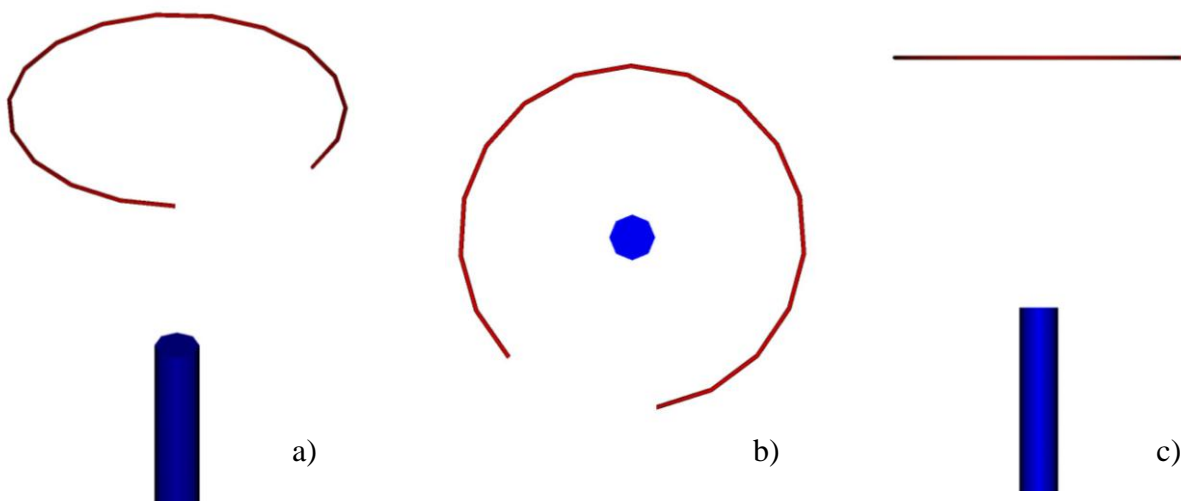


Fig. 1: Scheme for a conventional electron bombardment source, substantially a vacuum diode: hot filament (the cathode, red) and the evaporating material (in form of rod, the anode, blue) are shown. a) 3D view; b) top view; c) lateral view.

The main disadvantage is the strong limitation of the anode current due to space charge effect: very high anode voltages ($>2000\text{V}$), short distances between filament and anode, high thermionic emission (filament temperature $>2400\text{K}$) can only partially overcome the problem. Moreover, due to the continuous material consumption, the anode-filament distance progressively increases, thus producing a reduction of the anode current: this requires a continuous increase of the anode voltage

or/and of the hot filament temperature, until a repositioning of the anode is absolutely necessary (e.g., by means of a linear motion feedthrough). It is difficult to obtain a reproducible working condition; however, an “active” closed loop control on the evaporation rate (acting on the filament temperature or anode voltage) is possible by a direct measure of the evaporated material flux.

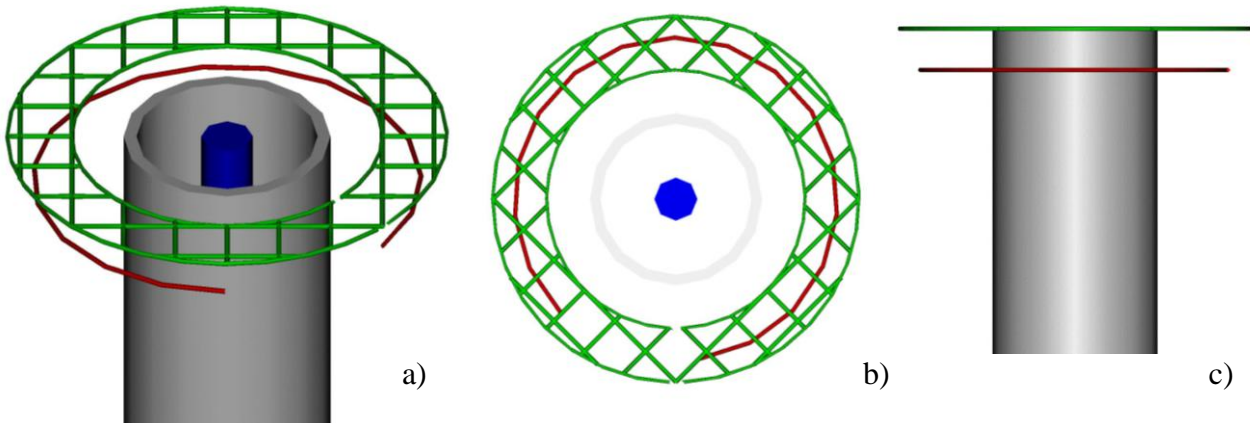


Fig. 2: Scheme for the TriCon electrodes configuration: the presence of the grid (green) between cathode (red) and anode (blue) shows the “triode” structure of the source. The gray cylinder around the blue anode is set at ground potential. a) 3D view; b) top view; c) lateral view.

The TriCon source has a different electrodes structure: it is the configuration of a “triode” (Italian Patent pending n. GE98 A 000096, November 18th 1998). As shown in Fig. 2, the anode is surrounded by a metal tube set at ground potential and the electron emitting filament is positioned around it. A grid, set at a positive potential, is located between the filament and the anode, on the top tube edge.

The main advantages are:

- absence of space charge current limitations due to the high electrical field created by the grid in the filament region;
- high anode current even at low filament temperature (2200-2300K) and low anode voltage (600-1200V);
- high electrical field uniformity inside the source, good electrons distribution on the anode surface;
- **“fine tuning” of the anode current:** once the filament power (i.e., temperature) and the anode voltage are set, the anode current/power is regulated by variations of the positive grid voltage.

The precision is higher than in conventional electron bombardment sources, with the possibility to work very close to the material melting point, with higher evaporation rates;

- **the anode current reduction due to its length reduction is counterbalanced by a grid voltage increase:** it is possible to work with the same evaporating condition up to a reduction of 1.5-2mm of the anode length;
- **anode power stability means evaporating rate stability:** it is possible to maintain this last constant just by variation of the grid voltage, without changing anything else;
- the anode length can be easily monitored when the source is “in-situ”, with a precision of $\pm 0.2\text{mm}$;
- **superior reliability and reproducibility of the performances,** higher and “easier-to-obtain” than in the conventional “diode” sources;
- **evaporation from crucible: it acts as a real Knudsen cell,** material evaporation is obtained by crucible walls heating due to electron bombardment of its external surfaces. Evaporation from semiconductors and insulating material too can be obtained.

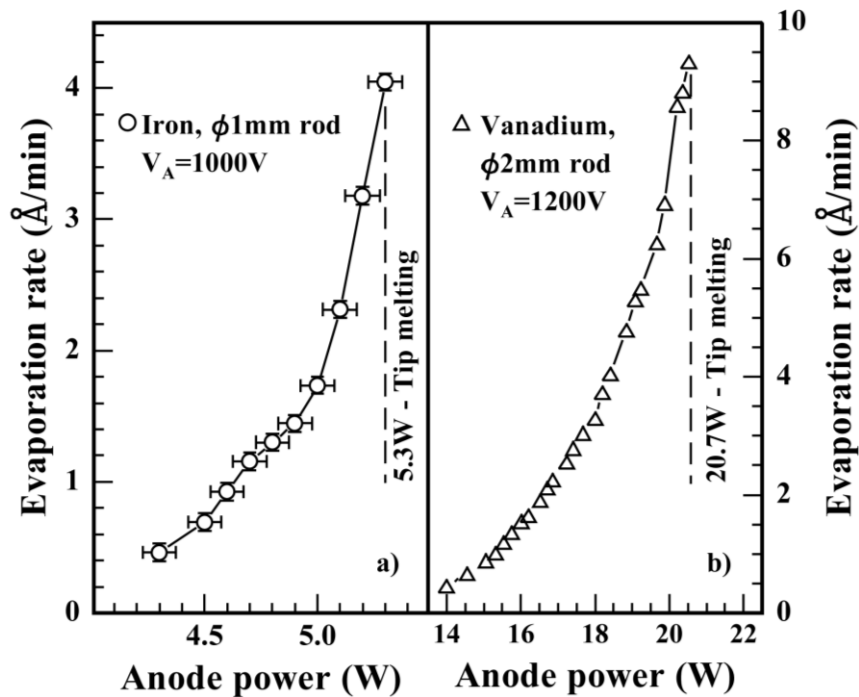


Fig. 3: Evaporation rates (source-substrate distance of 10cm) vs. anode power for:
a) 1 mm \varnothing Fe rod, 1000V anode voltage; a) 2 mm \varnothing V rod, 1200V anode voltage.

An additional power supply, $\pm 250\text{V}/50\text{mA}$, is necessary with respect to the diode-like sources. The power dissipated by the grid is usually low ($<4\text{W}$), even at the higher voltages: so, degassing and heat irradiation from the grid can be disregarded.

The TriCon concept has been inserted into a complete source designing, shown and described hereafter. The whole project has been developed and supported in 1997-1998 by INFM, Istituto Nazionale di Fisica della Materia, at the Physics Department of the University of Modena, Italy.

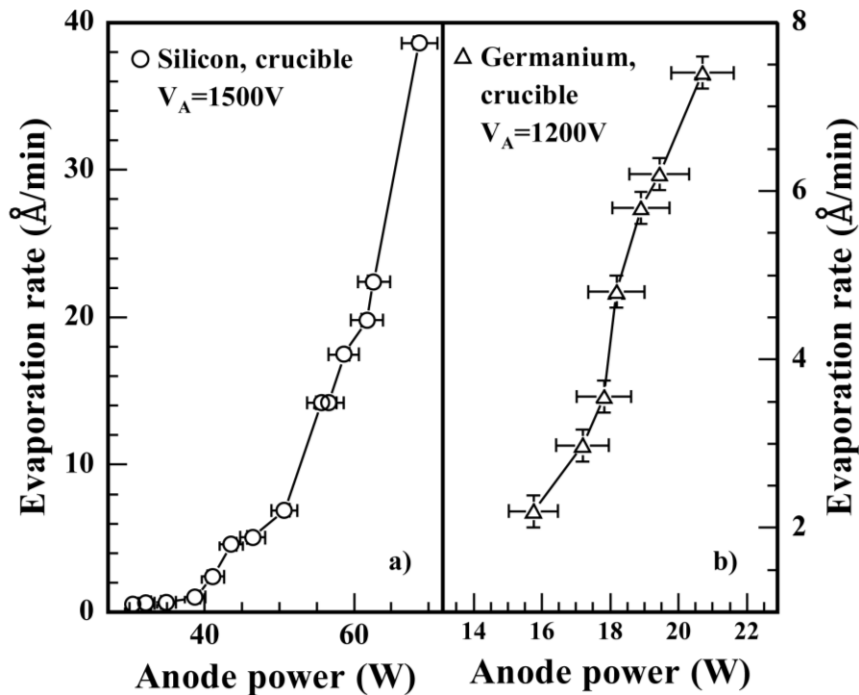


Fig. 4: Evaporation rates (source-substrate distance of 10cm) vs. anode power for crucible filled with: a) Si; b) Ge.

1.2. Performances.

Evaporation have been obtained from several materials, as shown in Tab. 1. Evaporation rates depends on the material thermal properties and on the source-substrate distance: data shown in Fig. 3 are for a 1mm \varnothing Iron rod (a) and a 2mm \varnothing Vanadium rod (b). Rods of different diameter will show very different performances. For what concern the role of the distance R, the well known $1/R^2$ flux dependence on the distance have to be taken into account.

It is very difficult to connect the anode power to the material temperature: in the Tab. 1, some indicative data are shown for both evaporation from rod and crucible. However, the shape, the

dimensions of the rod or rock-like crystal used are fundamental in determining the anode power for the required temperature, i.e. vapor pressure.

Material	Notes	Evaporation from rod (\varnothing 2mm)		Evaporation from crucible	
		Anode power	Temperature	Crucible power	Temperature
Ag				15W	1100-1200°C
B	large, irregular rock-like crystal	12W	1700-1900°C		
Co	Rod \varnothing 2mm	12W	1500°C		
Cu				13W	1000°C
Fe	Rod \varnothing 1mm	5.5W	1500-1600°C		
	Rod \varnothing 2mm	12W	1500-1600°C		
Ge	Large, irregular rock-like crystal	7W	1000°C	20W	1300-1400°C
Mo	Rod \varnothing 1.5mm	40W	2700°C		
PcCu	Powder, copper phthalocyanine			4W	~300°C
Ni	Rod \varnothing 2mm	12W	1500°C		
Si	Large, irregular rock-like crystal; wafer	9W	1400°C	60W	1700°C
Ta	Rod \varnothing 2mm; 0.2mm thin foil, 3mm wide	30W	2400-2600°C		
V	Rod \varnothing 2mm	21W	1900°C		
W	Rod \varnothing 2mm	45W	2700-3000°C		

Tab. 1: Temperature vs. anode power for several elements, for evaporation from rod or crucible.

2. Source description

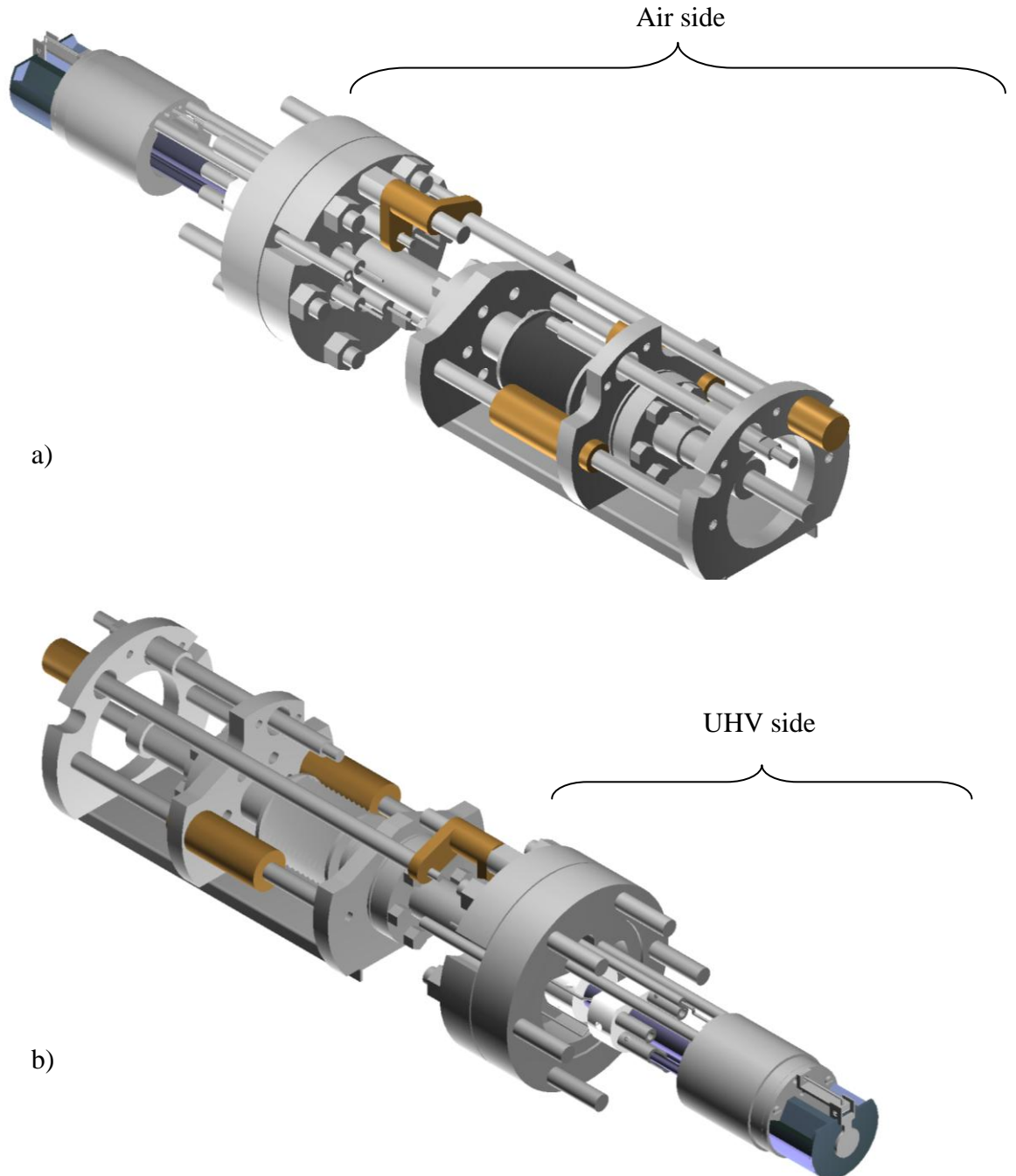


Fig. 5: The TriCon source: air side (a) and (b) vacuum side are put in evidence.

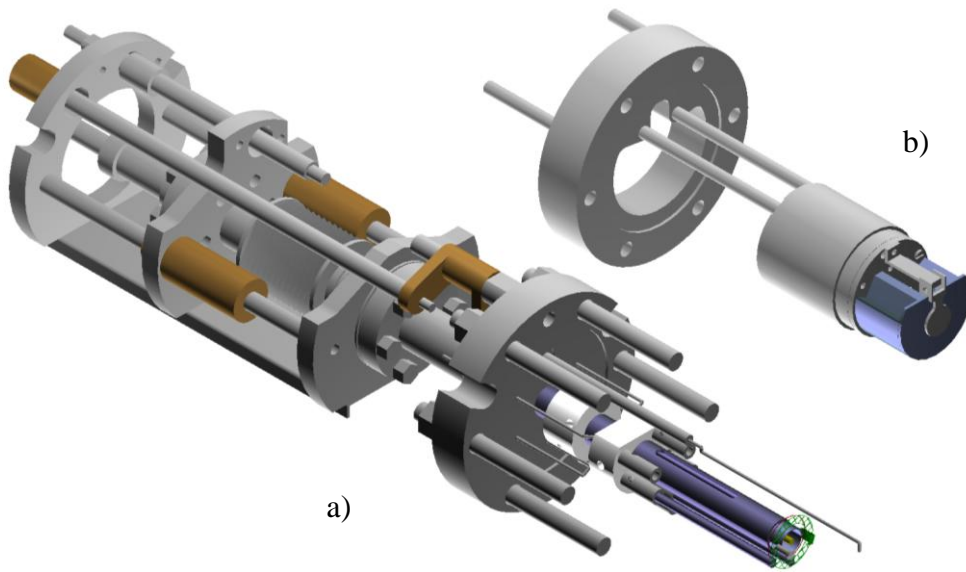


Fig. 6: The TriCon source, with the two parts not assembled: the CF35 main flange (a) and the CF35 double sided flange with the cooling shroud (b).

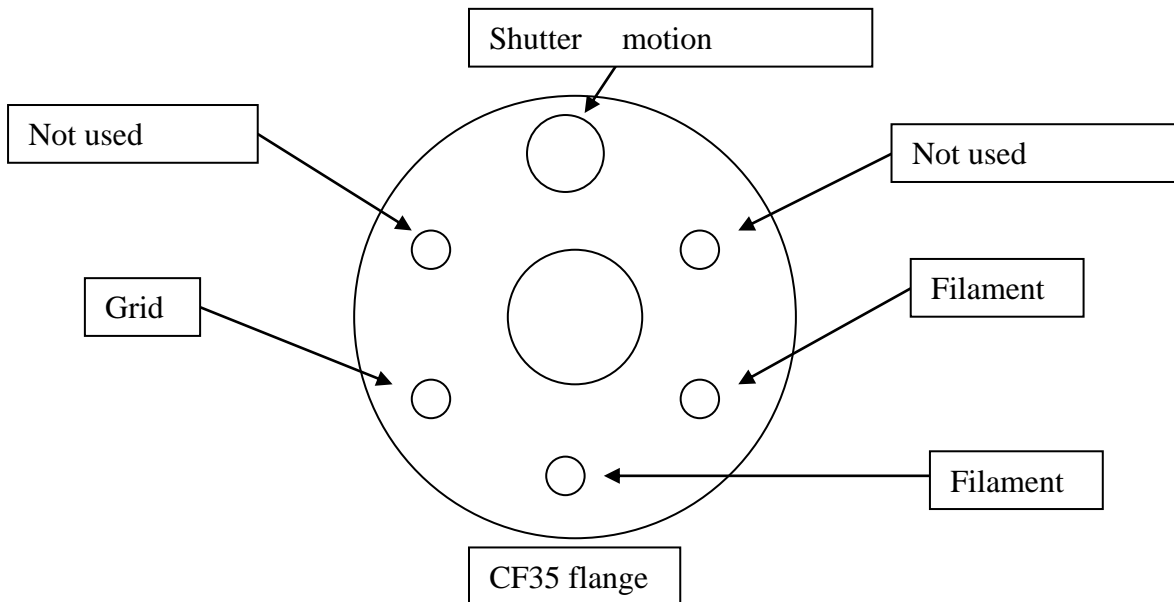


Fig. 7 Sketch of the rear view (source air side view) to show the pins for electrical connections on the main CF35 flange. The HV anode feedthrough is mounted on a CF16 flange at the bottom of the linear motion feedthrough (not shown here).

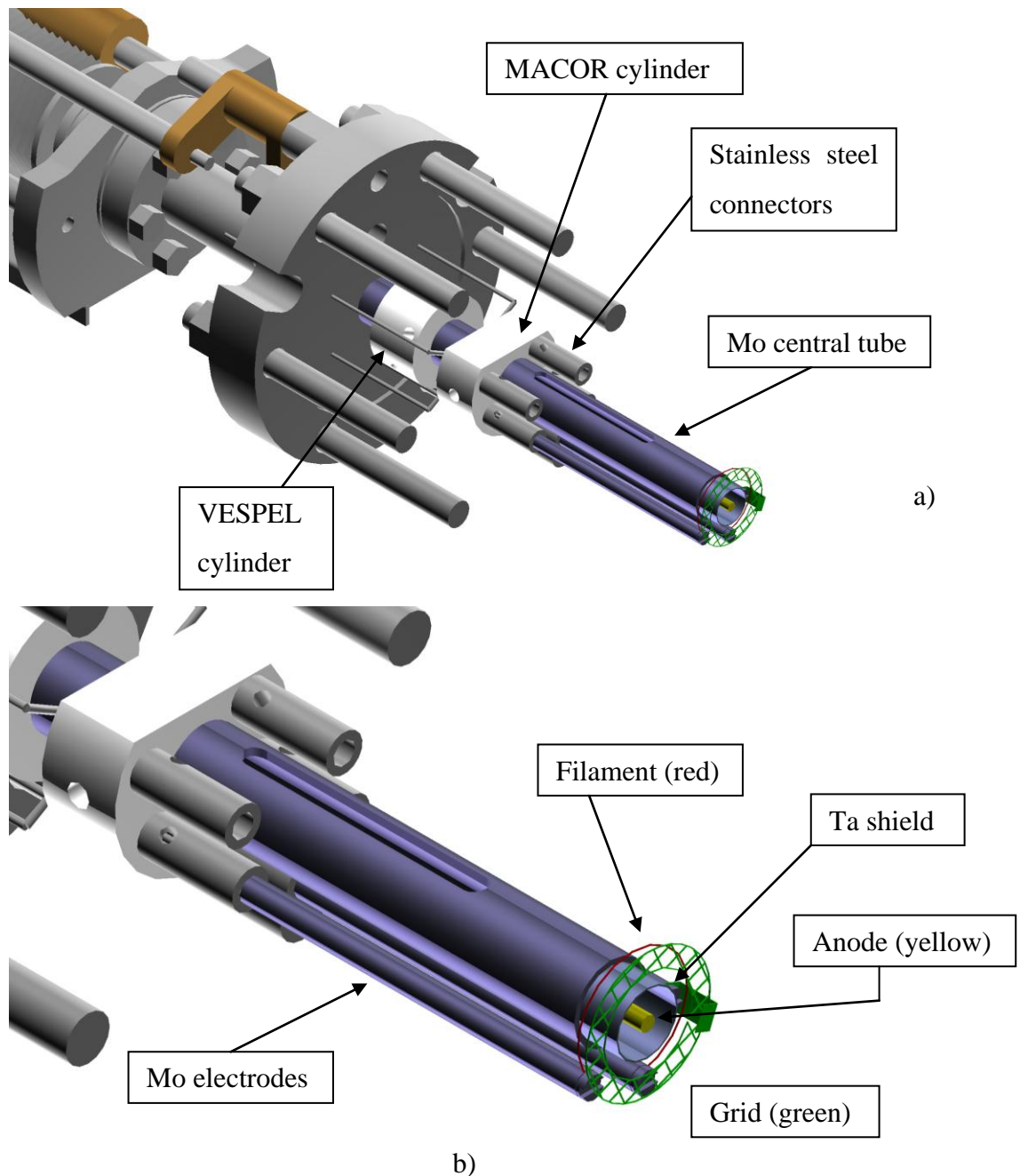


Fig. 8: UVH side of the main CF35 flange assembly (a), with an expanded view of the electrodes zone (b).

The source is composed of two parts (Fig. 6), i.e. two CF35 flange assemblies that must be mounted together by means of special studs:

- 1) the motion and electrical feedthrough CF35 flange (the main flange, see Fig. 5a). On the air side it is composed of (see also Fig. 6a):
 - a) the 5cm UHV linear movement (located on the main source axis, with an edge welded bellow and a millimetric scale located on a stainless steel plate): the movement is obtained

by means of a M6 screw on which is mounted a M8 hexagonal nut. Remember: **1 complete turn=1mm movement, counterclockwise rotation=anode insertion, clockwise rotation=anode extraction** .;

- b) the High Voltage (HV) feedthrough on a CF16 flange (located at the source bottom on the CF16 flange of the linear motion mechanism);
- c) five electrical feedthroughs (3000V/5A) welded on the CF35 flange (see Fig. 7): Filament +, Filament -, Grid, and two not used. The electrical connections (one MHV connector and a 6 pins-connector) for the power supply are positioned on the stainless steel plate.
- d) the linear motion for the shutter: a small edge welded bellows located on the CF35 flange, connected by a mechanical arm with the on/off lever on the bottom source air side.

In the UHV side are present (see Fig. 5b, Fig. 8):

- e) a Molybdenum (Mo) tube O.D. 10mm, I.D. 8mm, with a Tantalum (Ta) shield inserted on its top; the anode (a stainless steel cylinder 4mm \varnothing , 12cm long with the Mo material holder screwed on it) is inside the Mo tube and screwed on the main HV feedthrough.
 - f) On the basis of the Mo tube (near the CF35 flange) are located a brown VESPEL ceramic cylinder for anode centering and correct positioning (see Par5.3), and a white MACOR ceramic cylinder, to hold and grant electrical insulation among the 5 pins coming from the CF35 flange.
 - c) On the MACOR cylinder are located the two filament and the grid electrodes. Each of them is composed of a stainless steel connector, inserted into the MACOR cylinder and on the rod of an electrical feedthrough (contact is granted by a M2 grain), and of a Mo 3mm \varnothing with a M2 hole on the top. By means of M2 screws, the filament and the grid are fixed on the Mo cylinders.
 - d) **The grid is a Ta woven mesh, not welded: handle it carefully!**
- 2) The cooling shroud (see Fig. 6b and Fig. 9): it is a 316LN stainless steel tube connected to a double-sided tapped CF35 flange by means of two tubes, entirely located on the UHV source side. The CF35 flange on the vacuum side will be connected to the CF35 main chamber flange. The tube is closed, at the top side, by a hollowed stainless steel cap (hole, 6mm \varnothing); this is also the support for a Ta shield and a diaphragm, for the beam monitor electrodes and for the shutter. The tube has three longitudinal holes to allow the presence of the shutter mechanical arm (1mm \varnothing stainless steel bent rod) and of the two electrical contacts for the beam monitor. 6 special studs are screwed on the CF35 flange. The Ta diaphragm can be set in the correct position using

the two M2 screws which hold the diaphragm itself on the Ta shield: tighten them after any centering operation.

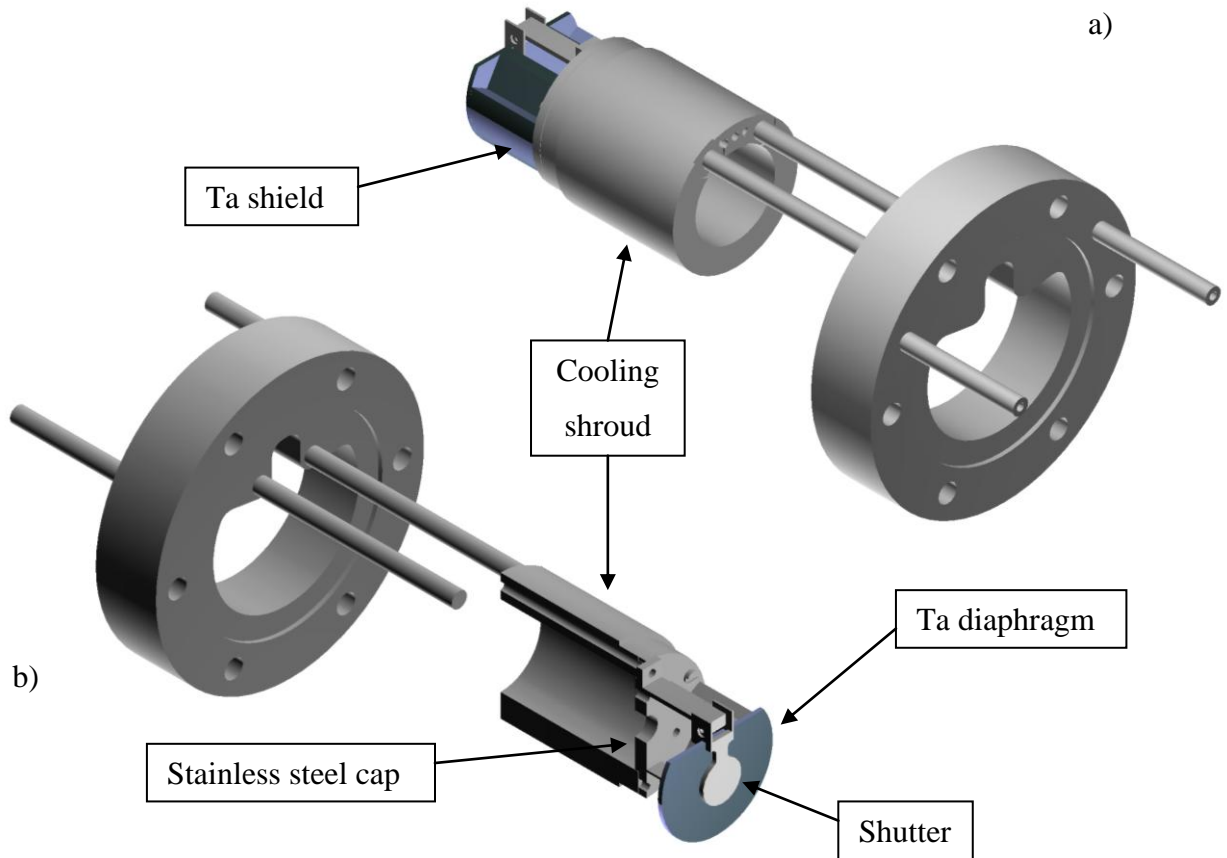


Fig. 9: Double sided CF35 flange with the cooling shroud (a); cutaway view (b) to show the stainless steel cap, the Ta diaphragm and shield, the shutter.

When the two main parts are disassembled, they must be joined before inserting the source on the UHV chamber (see Fig. 5). The cooling shroud (without the stainless steel cap) must be inserted axially on the main flange (do not forget the gasket!), with the supplied special 6 M6 studs (with silver coverage) already screwed on the cooling shroud flange: carefully, insert the shutter mechanical arm into the central hole of the cooling shroud and do not touch internal electrodes with the stainless steel cylinder. Then fix the two flanges by means of six M6 nuts and tighten them gently: to avoid possible damaging to the electrical feedthroughs, perform a stronger tightening when the source has already been inserted into the UHV system. See Par. 4.5 for complete source mounting (stainless steel cap, Ta shield and diaphragm, shutter).

3. Installation.

The source mounting on a UHV system requires the presence of a CF35 flange, with not threaded holes and standard internal diameter requirements: use M6 nuts, do not use M6 plates. Depending on the evaporation material type and form, care must be taken in positioning the source, e.g. vertical or off-vertical, downward or upward facing.

Evaporation from rod

If a rod is installed in the source, the device can be mounted in every position, vertical (upward or downward) or horizontal. In case of vertical mounting, with the source pointing downward, more care must be taken to avoid evaporating material tip melting, with possible drop falling outside from the source or inside it.

Evaporation from crucible

In case of the presence of a crucible, a horizontal mounting will produce a material fall out from the source, so a minimum inclination of about 10°-20° (upward) should be used.

The source is fixed by means of six M6 nuts tightened on the supplied special studs. The six M6 nuts on the main flange grants joining of the two source parts: so, **to remove the TriCon source from the vacuum chamber, do not unscrew the nuts on the source flange air side, remove only those on the vacuum chamber flange side.**

Before inserting the source in the UHV system, and after any kind of “in air” operation on the source, perform a careful inspection of the electrodes zone (see Fig. 10 and Par. 4.5). Moreover,

- **verify the grid-filament distance to be about 2mm, the distance between grid and the top of the two filament electrodes (Mo 3mm Ø cylinder) to be about 1mm;**
- **verify the grid and filament perpendicularity to the main source axis;**
- **verify that the grid and the filament distance with respect to the Ta cylinder and shroud internal walls is higher then 1mm;**
- **verify the resistance between filament, grid, anode to be higher than 20MΩ in any case;**
- **verify the resistance between filament and ground, grid and ground, anode and ground to be higher than 20MΩ in any case;**
- **verify filament resistance: it should be about 0.4-1Ω;**
- **verify the correct position of the Ta diaphragm.**

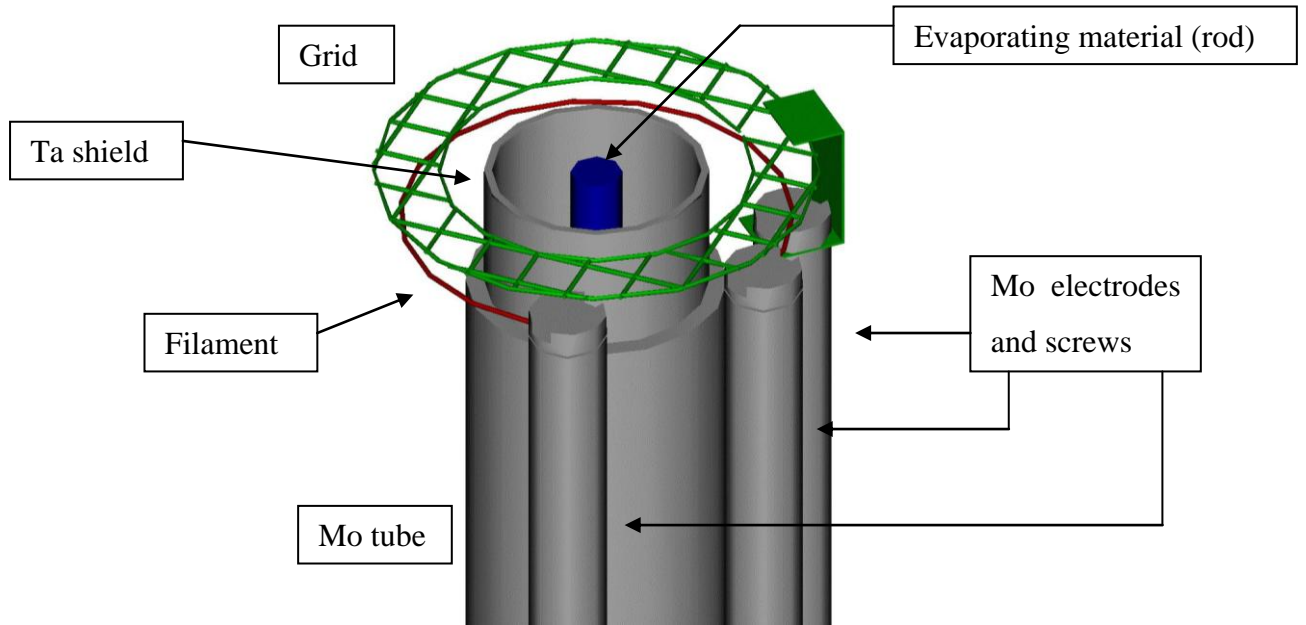


Fig. 10: Particular of the electrodes zone.

3.1. Source-substrate distance and beam divergence.

To evaluate the correct distance R [mm] between the source, i.e. the real evaporating point, and the growing substrate (or the thickness monitor) some parameters have to be known. If the distance L_1 between the substrate and the top of the source (i.e. the shutter or the Ta diaphragm) is known, $R = L_1 + 30$ mm; if the distance between the UHV chamber CF35 flange and the substrate is L_2 , $R = L_2 - 90$ mm. A typical working distance is $R = 100$ mm.

For what concern the beam divergence, it depends on the hole diameter of the Ta diaphragm: see Tab. 2. In case of evaporation from crucible, the TriCon source inclination can lead to different results from those reported in Tab. 2.

Ta diaphragm hole \varnothing (mm)	Beam divergence
1	$\pm 0.9^\circ$
2	$\pm 1.9^\circ$
3	$\pm 2.8^\circ$
4	$\pm 3.8^\circ$
5	$\pm 4.7^\circ$
6	$\pm 5.7^\circ$

Tab. 2: Beam divergence vs. the diameter of the Ta diaphragm hole.

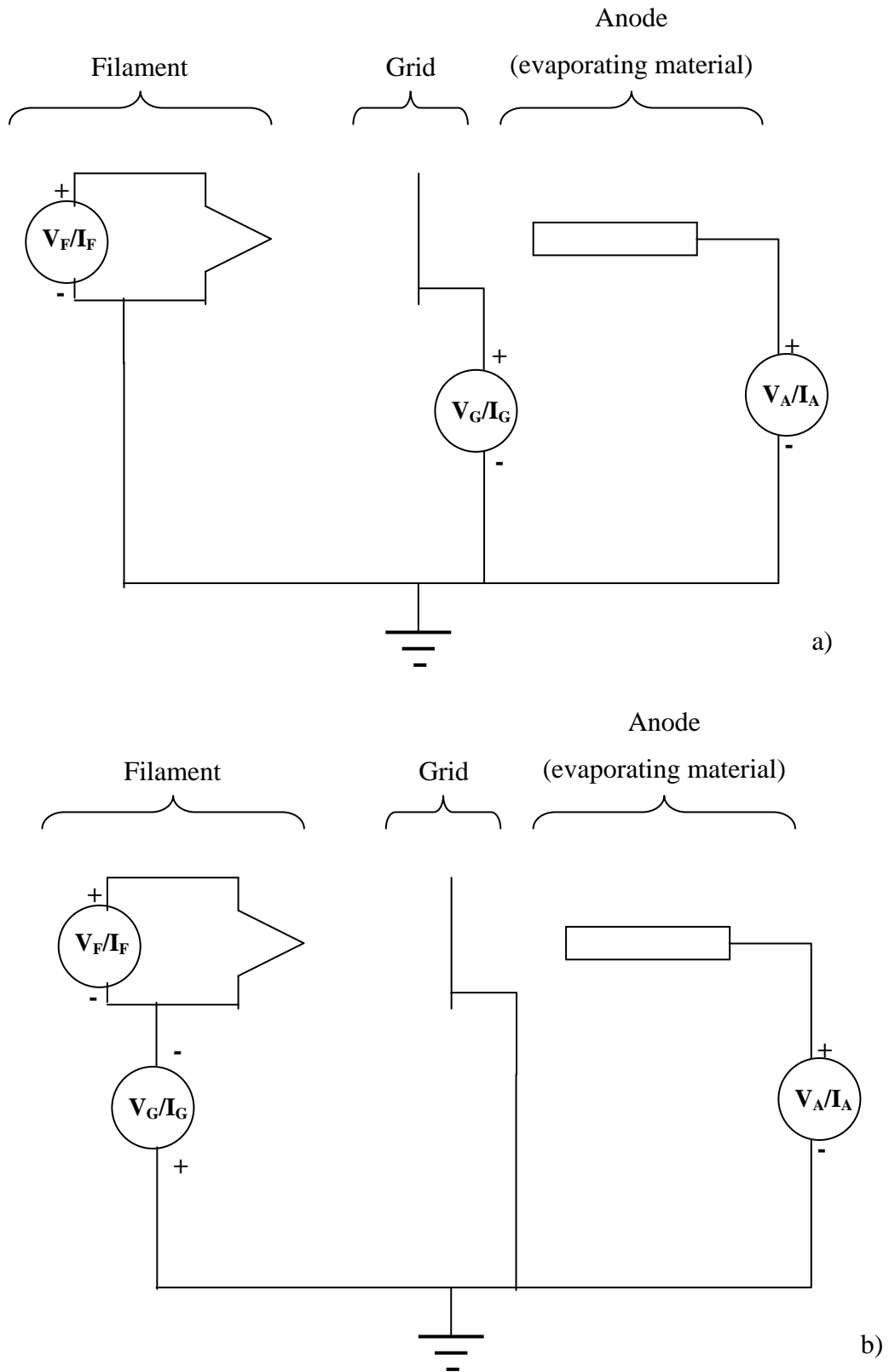


Fig. 11: Sketch for the electrical connections for the normal (a) and internal wall degassing (b) working condition: V_F/I_F , filament low voltage power supply; V_G/I_G , grid $\pm 250\text{V}$ power supply; V_A/I_A , anode (evaporating material) HV power supply. The “ground” symbol refers to the UHV chamber ground.

3.2. Electrical connections.

In Fig. 11a are described the electrical connections between the power supply and the source electrodes: **all the power supply negative connections are referred to the chamber ground level.** The use of the TriCon power supply is strongly recommended to ensure safe and reliable operations. In case of use of different power supplies, always respect the shown electrical connections.

Electrical connections shown in Fig. 11a are those for normal operation and for the filament, grid and anode degassing (grid power supply switch in NORMAL), while those shown in Fig. 11b are for the case of internal walls degassing (grid power supply switch in DEGASSING), as described also in Par.6.4.

4. Common procedures

The hint of this paragraph is to describe some operations that are “common” for several working procedures, as well as to give suggestions that are useful in most of practical situation.

4.1. Starting conditions.

Before every operations, check connections between the source and on the power supply. **Turn all potentiometers fully counterclockwise.**

The UHV system pressure must be in the 10^{-6} mbar range or lower: never turn on the filament or give voltage to the electrodes when the source is in air!

4.2. Water cooling.

Check the presence of a water flow. If evaporation has to be performed, be sure the water flow is cooling the shroud from at least 15 minutes. If degassing has to be performed, water has to be blown out from the cooling circuit, as well as connecting tubes have to be removed.

A water flux of 0.5 l/min is sufficient for a good cooling action: the maximum water pressure is related to the type of water connections chosen.

4.3. Turning on the hot filament.

The supplied filament is a 0.25mm \varnothing Tantalum wire, one coil self sustaining (see Fig. 10). Tantalum has been chosen because of its thermionic emission properties and its ductility: it can be easily shaped and its substantially unaffected by stress release during heating. However, in case of strong current increases, wire bending can occur and shorts can occur: so, **the following turning on procedure must be followed, every time the filament is turned on.** This will ensure long filament life and absence of deformation.

See Fig. 11a for electrical connections. Set the filament voltage and current potentiometer fully counterclockwise, set the grid voltage $V_G=+10V$ and the anode voltage $V_A=100V$. **Filament current I_F must be increased in step of 0.25A (maximum current increasing step is 0.33A) every 30sec, or every 45sec if filament is new,** as shown in Tab. 3. Faster step and higher current increase will result in filament deformation.

Ta filament life	Current step	Time step	Total time
New	0.25A	$\geq 45\text{sec}$	$\geq 12\text{min}$
Used	0.33A in the 0÷2A range	$\geq 30\text{sec}$	$\geq 3\text{min}$
	0.25A in the 2÷5A range	$\geq 30\text{sec}$	$\geq 4\text{min}$

Tab. 3: Filament turning on procedure parameters.

The typical working condition for the supplied filament are: current in the 3.8-4.4A range, typically 4.1A, maximum 4.7A, applied voltage depends on the filament life. As you can see from Fig. 12, after several hundreds of working hours, there will be an increase in the filament resistance, i.e. an increase in V_F to obtain the desired I_F (see Fig. 12). Remember: rarely you will need I_F values higher than 4.3A for your evaporations.

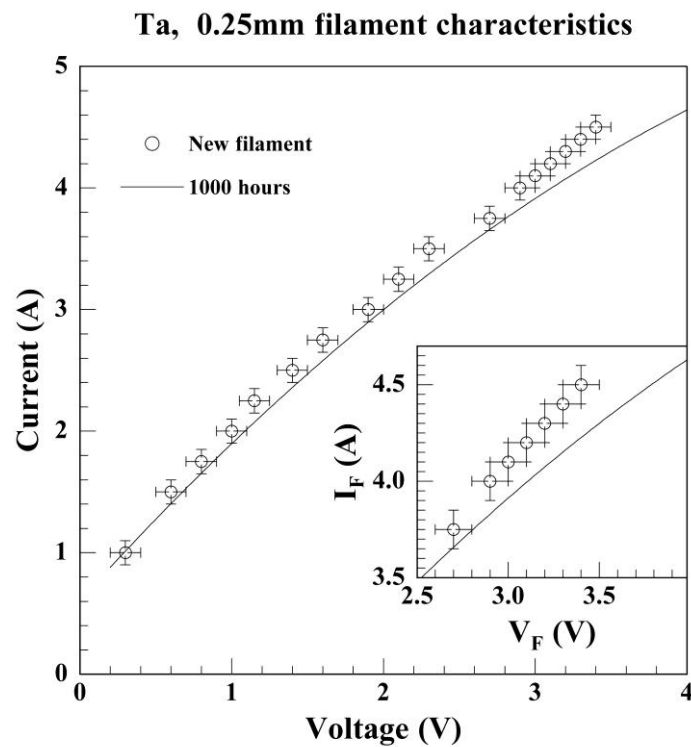


Fig. 12: Current-voltage characteristic for the Ta $\varnothing 0.25\text{mm}$ filament, new and after 1000 working hours. The region where thermionic emission occur is shown in the inset.

4.4. “Z₀ - Zero level conditions”.

One of the main feature of the TriCon source is the possibility to obtain evaporation always in the same working condition (see Par.1.1). Moreover, in case of evaporation from rod, it is possible a direct and precise evaluation of the anode consumption during evaporation.

When an evaporating material rod is mounted, its tip must be positioned by means of the linear motion feedthrough at the grid level, i.e. with the evaporating material tip just outside (0.5mm) of the Ta foil cylindrical shield on the top of the Mo central cylinder (see Fig. 2 and Fig. 10). **When the source is in UHV conditions, anode position reproducibility can be performed by measuring the operation parameters (I_G, I_A) in well defined working conditions and/or before any evaporating operations.** For example:

- **I_F=4.10A**, or the filament current used in your evaporation experiment
- **V_G=40V, 50V**, or lower in case of low anode power required (<5W)
- **V_A=1000V, 1200V** or lower in case of low evaporating temperature required (<1000K)

This values should permit to work far from strong evaporation or melting temperatures of the used material: however, **lower I_F and/or V_G and/or V_A values must be used, if necessary**. Best results in terms of reproducibility are obtained if two or more zero level conditions are determined for different V_G and V_A values. Moreover, as voltage values used in current evaporation are usually different from those of the Z₀ conditions, **a useful practice during operation is to note the V_G, I_G and V_A, I_A values at the beginning of each evaporation session**.

After several evaporation session, **the original anode position can be found by setting the Z₀ voltage or user defined conditions and moving the anode linear feedthrough until the correct current values are obtained**. A retraction of the anode will produce an I_G increase and an I_A decrease. An insertion of the anode will produce an I_G decrease and an I_A increase, up to a saturation of I_A: this means the anode tip distance from the Z₀ position is ≥6mm.

The original position can be reproduced within ±0.1mm and the effective material consumption can be easily “in-situ” monitored. **Define the Z₀ level after a complete source degas**.

4.5. Inspection of the evaporation zone: removing the stainless steel cap.

When the source is “in air”, there is the possibility to control the grid-filament-anode zone without removing the cooling cell double sided CF35 flange from the main source flange. To perform this, the Ta shield/diaphragm and the stainless steel cap have to be removed from the stainless steel shroud.

- 1) Put the shutter in the open position and unscrew the two M2 grains in order to loosen it from the mechanical arm (stainless steel rod, 1mm Ø);

- 2) remove the two M2 nuts (they are tightened on two M2 grains with two M2 washers) holding the Ta shield: now gently remove the Ta shield, together with the Ta diaphragm (it is not necessary to remove it from the Ta shield);
- 3) remove the third M2 nut (and its washer) that holds the stainless steel cap;
- 4) displace the 1mm \varnothing mechanical arm from the shutter;
- 5) dislodge the cap from the cooling shroud (the three M2 grains will remain tightened on the shroud itself).

Now the evaporation zone can be seen, with the grid over the filament, the internal Ta cylinder with the evaporating material and material holder inside of it.

After the required operations, reinstate the source cap on the cooling shroud and tighten the M2 nut (the one opposite to the shutter mechanism). Insert the mechanical arm in the proper shutter hole and position the Ta shield on the cap, tightening the two M2 nuts. Tighten the two M2 grains on the shutter: when the shutter is closed, it must be parallel to the Ta diaphragm and far 1mm from this one. **Be careful: if the shutter touches the Ta diaphragm, the linear motion for its movement will be damaged!** Adjust the position of the Ta diaphragm on the Ta shield, in order to center the hole on the main source axis.

4.6. Evaporating material cooling down.

In case of evaporation from rod, no problems will occur for every working condition if a quick or sudden cooling occurs:.

In case of evaporation from crucible, instead, great care must be taken for the cooling down procedure to prevent crucible cracks due to a sudden temperature reduction. If the evaporating material is in a liquid state in a ceramic or metallic crucible, and interactions among the crucible material and the melted substance occur, anode power must be decreased very slowly. In case of crucible breaking, the internal part of the TriCon source could be irremediably damaged! Take also into account that some material, after having been melted, must remain in liquid state.

5. Evaporating material mounting.

The source is equipped with two Mo material holder, 3cm long (see Fig. 13), respectively designed for 1mm and 2mm diameter rod. This last is also used for crucible supporting.

To mount the evaporating material, first remove the stainless steel cap (see Par. 4.5) in order to see the evaporation zone. Unscrew the three M3 grains for anode positioning (1 turn will be enough) to leave the anode totally free. Then use the following procedures for rod or crucible mounting.

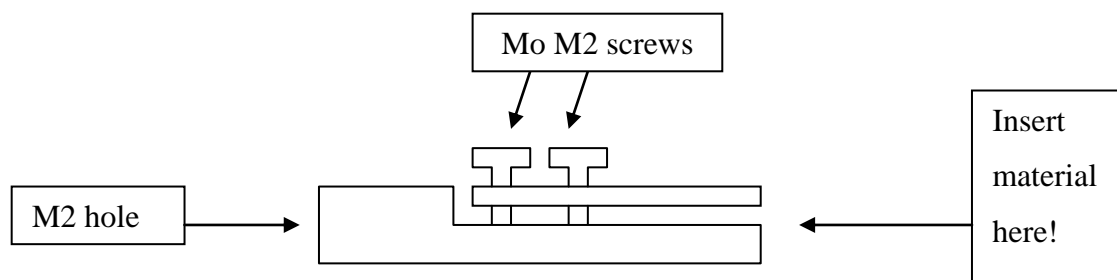


Fig. 13: Sketch for the Mo material holder (lateral view): the material (or the crucible) must be inserted from the right. The left part is M2 threaded in order to screw the device on the stainless steel cylinder.

5.1. Rod.

With the two material holders supplied, it is possible to insert cylindrical rod with diameter in the 0.8-3mm range. Move the anode to the uppermost position: the material holder is now at the level of the Ta cylinder on the top of the Mo tube. Unscrew the material holder by means of flat end pins, then extract it carefully without touching the grid and/or the filament. Never mind if the material holder together with the 4mm \varnothing stainless steel rod are extracted (see Par.5 for more details): this is due to the coupling among them, that is stronger than that of the rod and the HV feedthrough.

Unfasten the two Mo M2 screws located on the material holder (see Fig. 13): they are very short, do not unscrew them completely. If the two screws are blocked, wet the tip of the screwdriver with some alcohol drops and gently unfasten the two screws. The rod can be inserted in the material holder for a maximum/minimum length of 8mm/3mm: then, gently tighten the two Mo M2 screws, alternatively, to reach a good mechanical contact among the rod and the material holder.

After material mounting, just screw the material holder on the central rod (or the whole central stainless steel rod on the HV feedthrough).

It is possible to insert material in form of foils, wafers, or even with no regular shape, as rocks: in this case, absolutely avoid contacts between the material itself and the Mo tube, whose internal diameter is 8mm. Avoid material pieces with external dimension that are larger than $3 \times 3 \text{ mm}^2$ or with strong longitudinal deformations: it could be even impossible to center the anode for the whole material length.

Minimum and maximum lengths for rod or rocks are 5mm and 55mm! Remember that the maximum anode linear excursion is 50mm! See the scale on the metal plate, on the air side of the TriCon source.

5.2. Crucible.

The crucible is shaped in order to be positioned on the molybdenum $2\text{mm } \varnothing$ material holder.

If the used crucible has an outside diameter of 9mm or higher, the grid must be removed to avoid shorts among the grid itself and the HV anode. However, it is strongly recommended to always remove the grid when using the crucible: contact 2M Strumenti service for further information. To remove the grid, first unscrew with an allen key the M2 grain which holds the grid-molybdenum electrode-stainless steel barrel system on the electrical feedthrough; then gently take away the whole from the white MACOR cylinder and remove it from the top of the cooling shroud, avoiding contacts among grid and the filament. Put the grid assembly in a safe place.

Remove the tantalum cylinder inserted on the top of the molybdenum central tube: there is a tight mechanical fitting among the two tubes, so extraction have to be done firmly. This operation is always necessary, whatever the crucible dimensions are.

Move the linear motion feedthrough in order to put the anode in a position where extraction of the evaporating material holder is possible, as previously described in Par.5.1; then, insert the crucible in the material holder and tighten the two material holder screws to fix the crucible. **By using the linear motion feedthrough, put the crucible with its external lower edge +1mm over the filament plane, i.e. ~4mm from the Mo tube edge.**

The crucible can be filled with the evaporating material when it is positioned in the source or before. **Do not let fall pieces of material inside the Mo tube**, otherwise electrical shorts or mechanical damaging of the edge welded bellows of the linear motion feedthrough will occur. **Do not fill completely the crucible volume; maximum material quantity is about $\frac{3}{4}$ of the total volume, but may be lower for materials which tends to wet the crucible walls.**

5.3. Anode positioning and centering.

An important operation with the TriCon source is to put the anode in the correct position from the point of view of the main source axis and distance from the grid. The correct axial anode lateral

position have to be found to reach the best electrical field uniformity inside the source, as well as to establish the necessary distance between the HV anode and the grounded internal walls of the Mo tube. Do not bother about the anode movement or apparent absence of rigidity: the poor mechanical fitting among the 4mm \varnothing central anode rod and the HV electrical feedthrough (mounted on the CF16 flange) is necessary to allow anode centering and electrical contact without stress transfer to the main external ceramic. The source is provided with a **three-point centering system located in the VESPEL cylinder: three M3 grains (120° spaced) are transversally screwed into** and can touch the central anode. Observing the anode from the top of the source, screw and unscrew the three grains until the correct axial position of the evaporating material tip is found, i.e. when the anode tip is at the center of the Ta shield. Sometimes, only two of the three grains have to be moved to reach the correct central location.

Five M3 grains are present on the VESPEL cylinder, but two are used to hold the insulating cylinder on the Mo tube. The three grains necessary for anode centering can be easily distinguished from the other two observing Fig. 14. **Do not tighten firmly the three grains on the central anode**, otherwise the anode itself will be blocked and the linear motion could be damaged due to the strong mechanical strains: the anode must be free to move for the whole evaporating material length. **Verify that the anode position is correct within $\pm 0.5\text{mm}$ of lateral movement, for the whole length (use the linear motion feedthrough)**. If the evaporating material is in form of irregular rocks, avoid electrical shorts among the HV anode and the Mo tube.

In case of crucible mounting, the same procedure must be observed in order to correctly position the crucible itself.

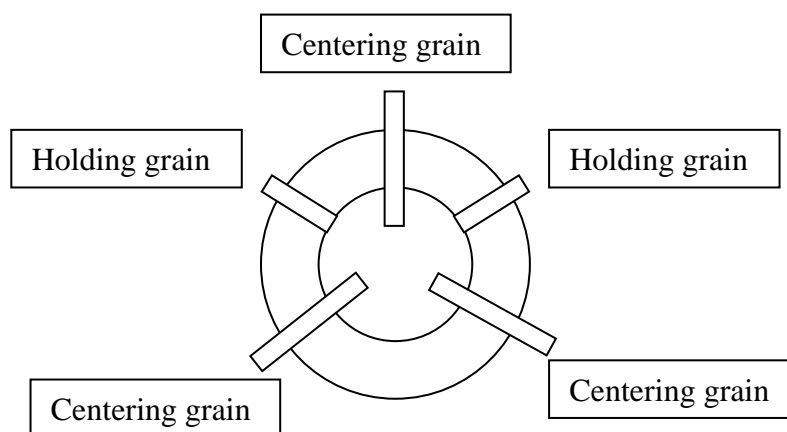


Fig. 14: Horizontal cutaway of the VESPEL cylinder, to put in evidence the different M3 grains screwed into.

6. Degassing procedure.

When the source is inserted in a UHV system, a bake out is necessary to reach the best performances from the point of view of low degas levels during normal operation. **The maximum bakeout temperature is 180°C; if the bake out is performed by tapes, avoid direct contact with the source parts. When the source is hot, do not move any mechanical part.**

Furthermore, a specific degassing procedure have to be done to clean the source internal walls and electrodes. The degassing operation should be performed before the main UHV system bakeout and after this, to assure best performances and low degas pressure after the bakeout.

Four consecutive steps must be performed in the following sequence:

- i)* filament degassing (by direct heating)
- ii)* grid degassing (by electron bombardment)
- iii)* anode, i.e. the evaporating material degassing (by electron bombardment)
- iv)* source internal walls degassing (by electron bombardment).

All operations will increase the UHV system pressure. **To avoid shorts and arc formation between electrodes inside the source, do not exceed 2×10^{-6} mbar chamber pressure and do not use anode voltages higher than 1000V when pressure is in the 10^{-6} mbar range.** Moreover, in case of strong pressure increase, degassing time have to be prolonged and intermediate stages at increasing power are necessary: a complete degassing procedure can request from 12 to 24 hours or more, depending also on the pumping system of the UHV chamber.

Before starting, read Par. 4.1 and see Fig. 11a and b for electrical connections. **Blow away the water from the cooling shroud to avoid formation of high pressure-high temperature water vapor inside of it.** Moreover, take into account that the source external parts can easily reach a temperature of about 70°C on the main flange: be careful while touching it!

Open the shutter before any degassing operation. After that, wait several hours before closing the shutter or starting using the source for evaporation.

6.1. Filament degassing.

See Par. 4.3 for filament turning on. During this operation, the pressures can rise up to 10^{-7} mbar. If the filament is new, reach a current $I_F=4.5A$. If the filament is not new, just reach the current that will be needed for normal evaporation, there is no need to exceed it.

Typical time required for the filament degassing is 30-60 min. After that, you will not observe a pressure reduction as both grid and walls have been heated by radiation in the meantime.

6.2. Grid degassing.

After the filament degassing, set $V_G = +20V$, $V_A = 100V$, $I_F = 4.1A$, then increase slowly the grid voltage up to $+240-250V$ whilst observing the grid current and the system pressure: do not exceed the suggested maximum pressure. **The maximum power on the grid is 6W, i.e. 240V and 25mA, the maximum current is about 30mA.** Increase the grid power with great care to avoid arc formation: pressure can reach values in the high 10^{-7} mbar range.

Do not care of I_G variation during degassing, do not vary I_F in order to correct grid current value: just wait for slow degassing as time required is about 60-120min.

6.3. Anode degassing.

After the filament and grid degassing, this is the most critical stage, as high voltage are involved while system pressure is usually in the high 10^{-7} mbar range. Material melting have to be avoided, possibly even if the crucible is used: use the correct anode maximum power (an indicative temperature vs. anode power table is given in Tab. 1, even if different material in different shapes will have different performances).

The presence of the crucible requires a different degassing procedures, as described hereafter.

6.3.1. Evaporation from rod

Set $I_F=4.1A$, $V_A=300V$, $V_G=+20V$. Increase V_A to reach in sequence 500V, 800V, 1000V, whilst observing the anode current and the chamber pressure. Slowly increase V_G to reach about 80-90% of the anode power that will be used during operation, or power required for material tip melting. If very high power are requested ($>15W$), higher anode voltages and I_F can be used, provided the system pressure is in the low 10^{-7} mbar range. If very low anode power are requested ($<5W$), use lower I_F . Time required for the operation is about 60-120min.

6.3.2. Evaporation from crucible.

Set $I_F=3.9A$, $V_A=100V$ ($V_G=+20V$, if grid is present). Increase V_A to reach in sequence 500V, 800V, 1000V, whilst observing the anode current and the chamber pressure. Reach about 80-90% of the anode power that will be used during operation, or power required for material melting. If very high power are requested ($>15W$), higher anode voltages and I_F can be used, provided the system pressure is in the low 10^{-7} mbar range: set $I_F=3.9A$, $V_A=500$, then increase I_F of 0.1A and increase V_A to reach in sequence 500V, 800V, 1000V. Repeat the procedure until the necessary I_F value is reached. If very low anode power are requested ($<5W$), use lower I_F .

If the crucible is empty, power as high as 50, 70 Watt maximum can be set: be careful, 60-70 Watt corresponds to more than 2000K. If the crucible is filled, reach the melting point in a good pressure environment, e.g. $<10^{-9}$ mbar and after the chamber bakeout. Take great care of the cooling down procedure to avoid crucible breaking (see Par.4.6).

Time required for this procedure are 2-3 hours for a new empty crucible, 30min-2hours for a filled crucible. **In case of a new crucible, always perform a degassing before filling it.**

6.4. Internal walls degassing.

After the previous three degassing procedures, this is the last step. The filament power supply will be set at a negative voltage and the grid will grounded: in such a way, electrons will hit the internal walls in the evaporation zone. **Remember, now V_G is the negative referring voltage at which the filament is set and the grid is grounded. If the original TriCon power supply is used, set $V_G=0V$ and turn the DEGASS switch from OFF to ON. When the degassing operation has been performed, set $V_G=0V$ and turn the DEGASS switch from ON to OFF (power supply switch in NORMAL). If the original TriCon power supply is not used, change the electrical connection: control Fig. 11b for the right scheme and always set $V_G=0V$ before changing the filament referring voltage.**

6.4.1. Evaporation from rod.

Set $I_F=3.8A$, set $V_G=0V$ and turn the DEGASS switch from ON to OFF, then set $V_A=100V$, $V_G=10V$. Slowly increase V_G up to about 250V whilst observing the chamber pressure and the I_G value: the maximum current must be 50mA (some internal power supply fuses could broke). The maximum I_G value at $I_F = 3.8A$ will be lower than 30-40mA: so, set $V_G=-10V$, increase the filament current by 0.1A, then again increase V_G up to 250V. Repeat the sequence until the maximum $I_G=50mA$ value is reached.

Time required for this operation is 60min or higher.

6.4.2. Evaporation from crucible.

Set $I_F=3.8A$, set $V_G=0V$ and turn the DEGASS switch from ON to OFF, then set $V_A=50V$, $V_G=10V$. Slowly increase V_G up to about 250V whilst observing the chamber pressure and the I_G value: the maximum current must be 50mA (some internal power supply fuses could broke). The maximum I_G value at $I_F = 3.8A$ will be lower than 30-40mA: so, set $V_G=-10V$, increase the filament current by 0.1A, then again increase V_G up to 250V. Repeat the sequence until the maximum $I_G=50mA$ value is reached.

Time required for this operation is 60min or higher.

7. Evaporation procedure.

While the procedures are substantially the same for evaporation from rod or crucible, great care must be taken for what concern the thermal properties of the material to be evaporated (i.e. melting point, vapor pressure vs. temperature, thermal conductivity): best performances are obtained through a knowledge of this information. Moreover, source damaging can be avoided taking into account chemical reactions between source and evaporating materials.

For starting conditions, see Par. 4.1 and 4.2, then turn on the filament (see Par. 4.3).

7.1. Shutter

Open the shutter only for evaporation rate measurement or to perform film deposition.

The shutter is moved by the lever on the top of the 50mm linear motion mechanism, in the air side: it is a two position movement which corresponds to opened and closed shutter. At the end of the mechanical arm in the air side of the source there is an adjustment system, composed of two M3 nuts: **do not regulate the mechanism**, contact the 2M Strumenti service.

7.2. Evaporation from rod.

The typical working current for the Ta 0.25mm \varnothing filament is 4.1A, even if higher values up to 4.7A can be necessary if very high temperature ($>2700\text{K}$) have to be reached. **Set $V_A=1000\text{V}$ (or lower, if you are using low melting point material)**, then increase slowly the grid voltage whilst observing the anode current increase. **The required anode power should be reached slowly, perform a waiting step at 50% and 80% of the requested power**, to leave the system reach a thermal equilibrium. In case of very high evaporation rates or temperature close to the material melting point, great care must be taken whilst reaching the desired anode power: **a strong pressure increase and a sudden anode current decrease are observed if material melting occur** (see Par.10.1). **Tip melting must be avoided to obtain the best source performances.**

The main advantage of the TriCon source is the possibility to control the anode power, i.e. the evaporation rate adjusting only the grid voltage: once you have set the filament current and the anode high voltage, the desired anode power is reached and maintained constant just varying V_G . As can be seen in Fig. 15, the evaporation rate is kept constant for long time by varying the grid voltage in order to preserve the 10w anode power: **if an I_A decrease is observed due to the anode consumption, increase the V_G to set the correct anode current.** Grid voltage variations may be different from those shown in Fig. 15, as it depends on the several working condition.

Always take note of the V_A , I_A and V_G , I_G values when starting an evaporation session. When V_G is 80%-100% greater than the original value, e.g. V_G changed from 80V to 140-160V, it is

due to an anode length reduction of about 1-2mm or higher: so, the anode must be repositioned by means of the 50mm motion feedthrough to set the tip in the right position (see Par. 4.4). Decrease V_G of about 30% and insert the anode until I_A reaches the desired value. Perform this procedure until the original V_G value ($\pm 2V$) has been reached. **Do not perform this procedure with anode power close to the tip melting point. Reposition the anode only if the grid voltage is 80%-100% higher than the original value.**

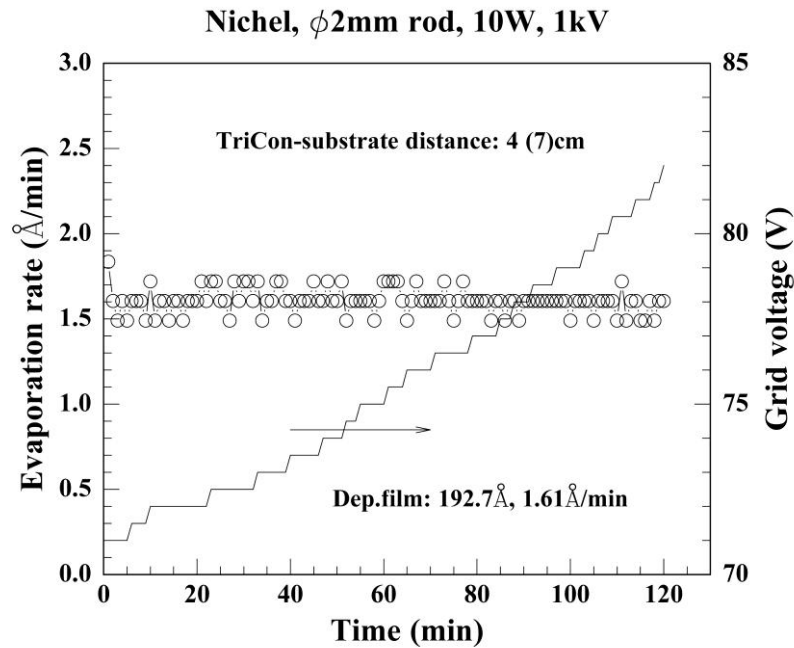


Fig. 15: Evaporation rate (left axis) from a Ni 2mm \varnothing rod vs. time: it is maintained constant, $1.6 \pm 0.1 \text{ \AA}/\text{min}$ by variation of the grid voltage (right axis) for a total time of two hours.

7.3. Evaporation from crucible.

This crucible is acting like a Knudsen cell, that is the material evaporation is obtained by heating the crucible and not by direct electron bombardment of the evaporating material. The desired temperature must be reached slowly to reach a good thermal equilibrium.

Set $I_F = 3.8A$, $V_A=200V$: the grid is usually not present, so leave the V_G value to zero (or to about 20V if grid is present). Increase slowly the anode voltage up to about 500-600V, where the anode current is about 70-80% of the maximum current at the highest anode voltage (3000V). So, you can evaluate the anode power that can be obtained if $V_A>600V$: if the anode current is insufficient, set again $V_A=200V$ and increase the filament current by 0.1A, then reach the $V_A=600V$

value to evaluate the power increase. Take care when material melting has to be reached, as it will cause a strong pressure increase.

After having reached the requested anode power, i.e. crucible temperature, wait at least for 5-10 minutes to reach a reliable working condition from the point of view of the crucible temperature. Depending on the evaporating material and its state and/or quantity, the evaporation rate can vary: to maintain it constant, just maintain the anode power constant itself, acting on I_F and/or V_A . **Usually, the power is constant without needing of further adjustment: if large power variations or high I_A instability occur, it means something is not working in the ideal condition, e.g. some melted material is falling down from the crucible.**

The same evaporation rate can be reached at the same anode power, provided the anode voltage is the same or within $\pm 20\%$: otherwise, strong differences are possible if, e.g. 10W are obtained at 500V and 20mA, or at 1000V and 10mA.

Remember: you are using a real effusion cell, so the beam monitor can not be used as there are no charged particles in the evaporated material flux!

8. Some practical suggestions.

The TriCon source is an advanced evaporating source: evaporating procedures are different from those of a conventional “diode source”, maybe easier but surely different. Some brief suggestions for a correct use will therefore be given, to summarize the procedures explained in this manual.

- To insert the evaporating material, the Ta shield and the stainless steel cap must be removed and repositioned after the filling operation, with a careful check of the electrodes zone (see Par.4.5).
- After evaporating material mounting, always set the rod in the correct position (see Par.5.3) and control the position of the Ta diaphragm.
- Crucible mounting usually requires grid removal: see Par. 5.2 and Fig. 10.
- Beware of irregular or large rods; do not fill the whole crucible volume (Par. 5).
- Perform the necessary controls before the TriCon source installation on the UHV chamber (see Par. 3).
- Minimum UHV system pressure required: 10^{-6} mbar. Never turn on the filament or give tension to the electrodes if the pressure is higher than 2×10^{-6} mbar.
- Follow the filament turning on procedure: see Par. 4.3.
- Perform a source degassing before and after the UHV system bakeout: see Par. 6. Be careful whilst reaching temperature close to the material melting point.
- Record the “zero-level conditions” after the degassing: see Par. 4.4.
- Low degassing level during evaporation can be reached only after the UHV system bakeout, with a proper degassing procedure and using water cooling during evaporation.
- Once the filament current and the anode voltage have been set, the anode current will be controlled by the grid voltage (Par. 7).
- Record the initial value of V_G , I_G and V_A , I_A before starting any evaporating session.
- Rarely you will need high filament current ($>4.2A$) and/or high anode voltage ($>1300V$): control the thermal properties of the evaporating material.
- After evaporation from crucible, cool down the anode slowly to avoid cracks and/or source damaging: see Par. 4.6 and 7.3.

9. Maintenance.

Use gloves while performing any kind of mechanical operation on the UHV parts of the TriCon source: avoid any direct contact whit skin.

9.1. Internal parts cleaning.

After several evaporating session, the internal parts of the source will be covered by coatings. Typically, the cooling shroud internal surface, the Ta cylinder internal/external surface, the stainless steel cap are the most “dirty” parts: the latter two can be cleaned after removing them from the source (see Par.4.5), while the cooling shroud can be cleaned also with the two source CF35 flange joined together, taking care to not touch the electrodes. The Ta diaphragm and the Ta shield too can show material deposition.

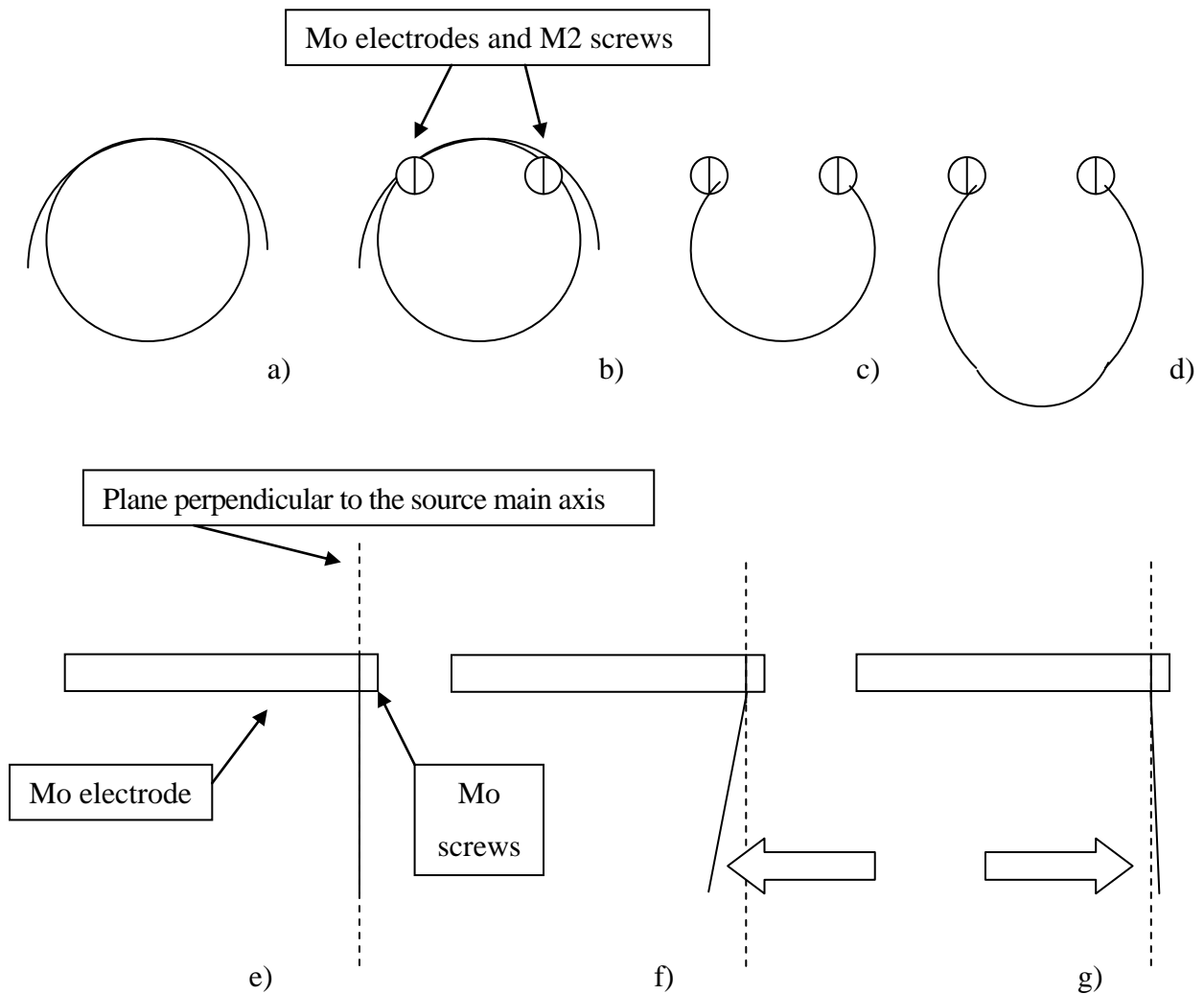
9.2. Filament replacing.

If the correct turning on procedure are followed, the Ta filament life will be several hundreds of hours (see Par.4.3). The Ta filament can be even gently touched, moved, bent without breaking it; in case of a W wire, this can not be done.

Use a Ta 0.25mm \varnothing wire unless the source is used in a strongly oxidizing atmosphere: in case of different material, take into account that **5A is the maximum filament current that can be applied to the source electrical feedthroughs. To replace the filament, use a wire 50mm long and bend it to form a 13mm \varnothing coil** (see ig. 16a).

Then, observe the following procedure:

- 1) remove the water connections from the source;
- 2) remove the stainless steel cap: see Par. 4.5;
- 3) carefully unscrew the 6 M6 nuts on the main CF35 source flange; then, dislodge the CF35 double sided flange with the cooling shroud. Avoid to damage or touch the internal electrodes. Now the electrodes zone is accessible;
- 4) remove the whole grid electrode assembly, taking note of its original position: first unscrew with an allen key the M2 grain which holds the grid-molybdenum electrode-stainless steel barrel system on the electrical feedthrough; then gently take away the whole from the white MACOR cylinder, avoiding contacts among grid and the filament;
- 5) observe the shape of the filament coil (it should be oval-shaped, see ig. 16c or d): it must be reproduced on the new filament!



ig. 16: Filament replacement: a-d, top view; e-f, lateral view. a) New filament (from a 50mm long wire, \varnothing 13mm coil); b) place the coil on the electrodes and fasten the two Mo screws; c) cut the wire in excess; d) shape the wire to reproduce the original form; e) the wire can be in whatever vertical position but must be bent downward (f) and then slightly upward (g).

- 6) now remove the old filament: unscrew (do not remove) the two M2 Mo screws. If they are blocked, wet the screwdriver tip with some alcohol drops and gently unfasten the two screws;
- 7) now install the new filament (ig. 16b): the wire must be positioned between the Mo M2 washer and the Mo threaded 3mm \varnothing cylinder. Fasten the two screws, then cut away the wire in excess among the two electrodes (ig. 16c);
- 8) if necessary, bend the filament in order to reproduce the old filament shape, pressing laterally the coil with your finger (ig. 16d);

- 9) now push the coil downward (see ig. 16e) and then slightly upward (see ig. 16f) with respect to a plane perpendicular to the source main axis, as shown in ig. 16f;
- 10)replace the grid electrode assembly, avoiding to touch the filament; position the grid in order to reproduce the original configuration;
- 11)replace the cooling shroud source part: remember to replace the gasket! Insert the flange avoiding to touch the electrodes;
- 12)replace the stainless steel cap (see Par.4.5), the Ta shield and diaphragm; control the position of the diaphragm hole (see Par.3).

10.Troubleshooting.

10.1. Rod tip melting.

If melting of the anode tip is reached, there will be the formation of a sphere at the top of the rod: this will produce a decrease of the anode length, thus reducing the anode current I_A in those working condition. The anode tip surface will be increased and the required power for the correct evaporation rate will consequently be higher. Moreover, this situation is really unstable as the sphere will reduce its dimensions continuously during evaporation, thus changing electrical field distribution inside the source and, consequently, the bombarding electron distribution on the tip itself. An equilibrium situation will be reached when the material forming the sphere has been totally evaporated: in this way the source will work at its best performance level.

10.2. Strange anode current.

If the anode has been inserted or extracted too much, with respect to the correct working position, the anode current will be higher or lower than the original one, respectively, and very high or very low working grid voltage will be needed. This is an anomalous situation that must be corrected to avoid rod bending, damaging of source internal parts, strong degassing.

Provided the procedure to find the zero-level condition has been performed, move the anode to find the correct position (refer to Par. 4.4).

10.3. Low evaporation rates from rod.

In case of very high temperature to be reached, i.e. $>1700^{\circ}\text{C}$, to obtain good evaporation rates, some thermal dispersion through the anode support can occur. Due to the continuous anode length reduction, the evaporation point will get closer to the Mo material holder and a decrease in the evaporation rate will be observed at constant anode power: thus, some increase in the anode power can be necessary to obtain the same evaporation rate. If still the problem remains, or low temperature are needed for the used evaporating material, the position of the anode is not correct: see Par.4.4.