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## Electron-gun evaporators of refractory metals compatible with molecular beam epitaxy ✓

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## SHOP NOTES

These are "how to do it" papers. They should be written and illustrated so that the reader may easily follow whatever instruction or advice is being given.

# Electron-gun evaporators of refractory metals compatible with molecular beam epitaxy

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The construction and performance of electrostatic electron gun evaporators, placed in a molecular beam epitaxy system are described. Minimizing electron emittance led to an evaporation of refractory metals at a vacuum level of  $2 \times 10^{-10}$ – $2 \times 10^{-9}$  Torr.

The molecular beam epitaxy (MBE) technique, a tool used for the production of single crystal thin films, especially semiconductor compounds<sup>1</sup> and metal–semiconductor junctions,<sup>2</sup> has gained widespread use. The main advantages of the MBE technique are (1) the ability to grow very pure thin films (due to the UHV environment), (2) accurately controlled film compositions, and (3) a wide range of alternating layers with well-defined interfaces. However, the materials used for generating the molecular beams are limited to relatively high vapor pressure materials, because evaporation cells, which are filament heated, do not usually exceed temperatures of 1300 °C.

Evaporation of lower vapor pressure materials, such as silicon or refractory metals requires the use of electron gun evaporators (EGE's). Some problems, however, are arising during EGE's operation in a MBE system. The most important of them is the emitting of high- and low-energy electrons, which in turn may cause the following effects:

(1) Electron stimulated desorption (ESD) of gas molecules adsorbed on the system walls and the cold shroud. This will cause the rise of system pressure and lead to film contamination.

(2) Damage to the grown film by the impinging electrons.

(3) An interference with *in situ* monitoring instruments, such as ion gauges and the high energy electron diffraction apparatus.

Another problem is the outgassing of the gun assembly and nearby system walls due to the high temperatures of the source materials.

Of the two major types of EGE's, namely, magnetically and electrostatically focused, the former is more prone to the phenomena described above and hence, less compatible with MBE requirements. We have designed, built, and incorporated into an MBE system electrostatically focused EGE's, similar in principle to the Unvala gun<sup>3</sup> and to the commercially available EGE's,<sup>4</sup> but incorporated some major design changes which eliminated most of the above-mentioned problems.

A simple schematic of the gun's head which has a cylindrical symmetry is shown in Fig. 1(a). A circular line filament, made of thoriated-tungsten, is situated between a shield and

a cage, both biased at a high negative voltage of 5–10 kV (–HV). The filament is heated by a 12 V/30 A power supply, and is floating also on –HV. Electrons emitted from the filament, span trajectories which follow the electric field lines established by the potential difference between the –HV components and the grounded hearth. A focusing mechanism (not shown) can change the relative position between the hearth and the rest of the gun's head, thus enabling an exact focusing of the electron beam at the top of the evaporated metal. In a degas mode, both cage and shield are grounded and the filament is held at –3 kV [Fig. 1(b)]. Electrons then are accelerated toward the cage and shield, heat and outgas them thoroughly.

The outgassing due to heating mentioned above was reduced considerably by building all the head's parts from mo-

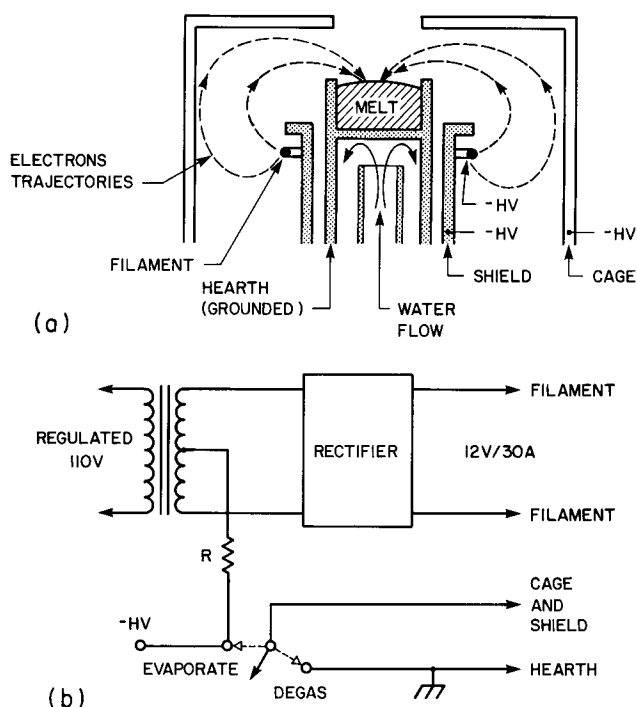


FIG. 1. (a) Side view of the gun's head, with approximate electrons trajectories. (b) A schematic diagram of the electrical connections to the gun's head.

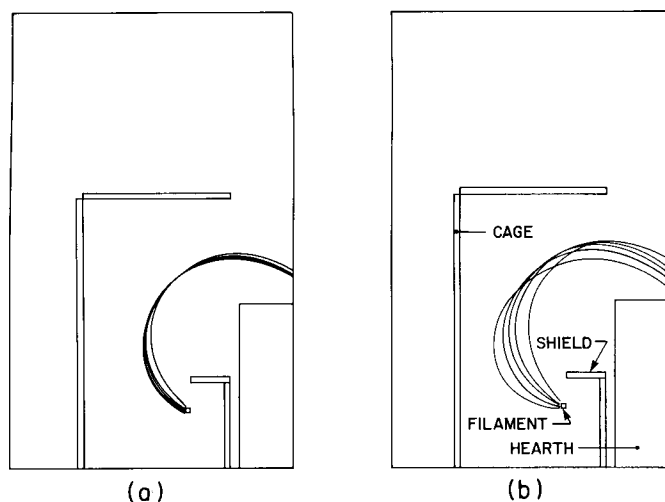


FIG. 2. Calculated trajectories of the electrons. (a) When cage, shield and filament are in the same potential ( $-10$  kV), and hearth grounded. (b) When the filament is at  $-9.9$  kV, cage and shield are at  $-10$  kV and hearth grounded.

lybdenum, tantalum, and tungsten. The shield and hearth were made out of Mo. The cage was constructed from a Ta mesh on a Mo frame with a W top cover with a hole in its center. The Ta mesh prevented heat development inside the gun, while reducing the temperature of the cage itself.

Among all electrons in the gun, the low energy electrons are secondary electrons which are emitted from the metal source with a potential close to ground. They can never escape from the cage which is biased at  $-HV$ .

The high energy electrons are of two types: those which are directly emitted from the filament and have the potential  $V_f$  in the range  $-HV < V < -HV + V_f$  (where  $V_f$  is the filament voltage), and backscattered electrons. Most of the backscattered electrons are at potential  $V$ , and few are at some positive potential relative to  $V$ , due to energy loss upon scattering.<sup>5</sup>

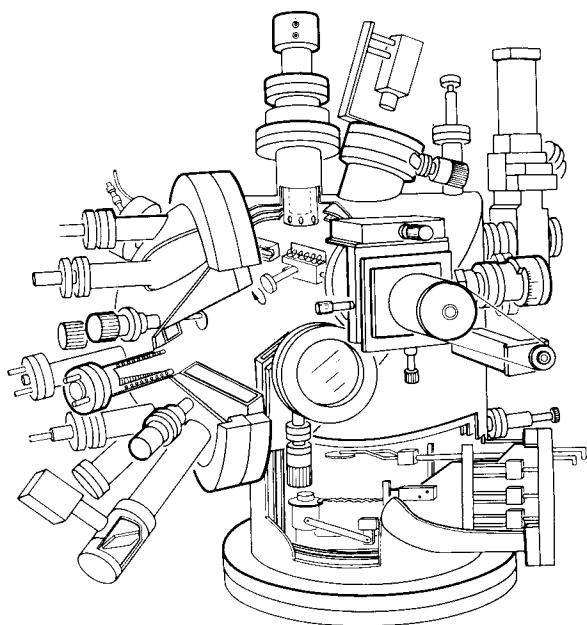


FIG. 3. MBE system with the EGE's incorporated under the LN<sub>2</sub> shroud.

If  $V_f > 0$ , most of the direct electrons will stay in the interior of the cage, since they can never reach the top hole and cage's screen. Since the potential in the center of the top hole can be positive relatively to  $-HV$  by a few hundred volts (since the distance between the top of the hearth and cage is similar to the hole's diameter), backscattered electrons will escape through it, as was also found experimentally. To alleviate this problem, two steps had been taken: (a) The filament potential had been raised positively by  $V_+$  relatively to  $-HV$ , by inserting a series resistor between  $-HV$  and the filament [Fig. 1(b)]. The resistor had been optimized for maximum emission current at a particular  $V_f$  ( $100 \Omega$ , leading to  $+30$  V at  $300$  mA). Because  $V_f > 0$ , emitted electrons had a potential in the range  $-HV + V_+ < V < -HV + V_+ + V_f$ . An additional benefit from biasing the filament is demonstrated in Fig. 2. When  $V = -9900$  V, focusing is much better comparatively to  $V = -HV = -10$  kV. (b) The top hole had been covered with a tungsten mesh (with  $\sim 2$  mm spacing), which held its entire area at a potential close to  $-HV$ , (note that  $-HV < V$ ). These two steps almost entirely prevented electrons escaping from the cage.

Two EGE's were mounted into a RIBER-1000-1 MBE system below the liquid nitrogen shroud (Fig. 3). Bare surfaces nearby were water cooled on the outside. Total electron emittance from the guns was monitored by the RHEED screen and the ion gauges. The effects of rectifying  $V_f$ , ( $V_f > 0$ ), changing the series resistance to the filament and adding the mesh on the top hole were checked and optimized to minimize electron emittance, which was negligible thereafter.

W and Mo were evaporated onto GaAs, while the environment was monitored by a residual gas analyzer. After an initial period of outgassing, the constituents detected during evaporation (at rates  $1-3$  Å/min) were mainly hydrogen and to a lesser extent As (As resides on the walls of the chamber from previous evaporations). The background pressure was monitored immediately after the evaporation was terminated, and was  $\leq 2 \times 10^{-9}$  and  $\leq 3 \times 10^{-10}$  Torr for W and Mo, respectively. Auger studies of evaporated W and Mo did not detect any impurities in the films. GaAs layers grown afterward in the system were as pure as before the EGE's installation.

In summary, we report here on UHV compatible electrostatic electron gun evaporators, which enable the evaporation of refractory metals at pressures  $\leq 2 \times 10^{-9}$  Torr. Special means are described which minimized outgassing and electron emittance from these guns.

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<sup>1</sup>A. Y. Cho and J. R. Arthur, *Prog. Solid State Chem.* **10**, 157 (1975); K. Ploog, *Crystals, Growth, Properties, and Applications*, edited by H. C. Freyhardt (Springer, New York, 1980), Vol. 3, p. 73.

<sup>2</sup>R. Ludeke, *J. Vac. Sci. Technol. B* **2**, 400 (1984).

<sup>3</sup>B. A. Unvala, *Le Vide* **104**, 109 (1963).

<sup>4</sup>Manufactured by Vacuum Generators (VG).

<sup>5</sup>C. Kittel, *Introduction to Solid State Physics* (Wiley, New York, 1976), p. 295.