A VERSATILE ELECTRON BOMBARDMENT
EVAPORATION SOURCE

by

*R. L. Beno
T. Van Vorous*

RELEASED FOR ANNOUNCEMENT
IN NUCLEAR SCIENCE ABSTRACTS

THE DOW CHEMICAL COMPANY
ROCKY FLATS DIVISION
P. O. BOX 888
GOLDEN, COLORADO 80401
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13 January 1967

RFP-864

UC-2 GENERAL, MISCELLANEOUS,
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ABSTRACT

A versatile electron bombardment heated evaporation source was developed. This device may be used with many types of crucible materials selected for use with a particular metal. It is capable of use in an inverted direction with wire feeding of an evaporant metal.

SUMMARY

A versatile evaporation source, to be heated by electron bombardment, was designed. The base of low form crucibles is heated by electrons emitted and accelerated from a pancake niobium filament. These crucibles are supported by a ring and pin arrangement so that the source can be held in any position. A wire feeder mechanism is used with the device to supply evaporant metal to the crucible. In this way the source can be used to evaporate large volumes of metal at high rates.

INTRODUCTION

Electron bombardment heating is produced by accelerating electrons from a thermally heated filament (cathode) through a high potential to an anode. In an evaporation source the anode may be an auto-crucible or evaporant support crucible. Vinogradov⁽¹⁾ in his survey of evaporating techniques describes both types of sources and their limitations. The device described in this report utilized the evaporant support crucible technique.

The auto-crucible electron bombardment technique involves direct heating of a wire or rod as shown in Figure 1. A molten drop of the evaporant metal is

(1) Vinogradov, M. I., *Technique of Evaporating Metals in a Vacuum*, July-August, 1960, *Pribory i Tekhnika Eksperimenta* No. 4, pp 3-13.

Figure 1. AUTO-CRUCIBLE ELECTRON BOMBARDMENT EVAPORATION SOURCE

suspended by surface tension. Evaporation occurs from the molten pendant drop. The evaporation rate of this type of source is severely limited by space charge effects. Metal ionized by electron bombardment will produce arc discharge conditions between the filament and anode as rates of 100-200 mg/minute are reached.

The support crucible electron bombardment method involves heating of an electrically conducting crucible which contains the evaporant metal, as shown in Figure 2. The entire crucible must be maintained at a temperature at or above the temperature required for evaporation. Through careful design of a filament shield it is possible to eliminate space charge effects that would limit evaporation rates. Several investigators, such as Mattox⁽²⁾ and Smith,⁽³⁾ have investigated use of indirectly heated crucibles; however, no one has used a supported crucible arrangement to allow inverted evaporation with wire feeding to provide a constant evaporant metal supply.

EXPERIMENTAL

DESIGN

Figures 3 and 4 show two views of the evaporation source. Figure 5 is a concept drawing of the source

(2) Mattox, D. M., Personal communication.

(3) Smith, H., *Journal of Scientific Communication*, 26, 378 (1949).

Figure 2. SUPPORT CRUCIBLE - ELECTRON BOMBARDMENT EVAPORATION SOURCE

Figure 3. REAR VIEW OF ELECTRON BOMBARDMENT SOURCE

Figure 4. FRONT VIEW OF ELECTRON BOMBARDMENT SOURCE SHOWING FILAMENT AND TITANIUM DIBORIDE CRUCIBLE

to identify its components and show its power supply connections. The filament housing and support ring for this device are fabricated from Type 304 stainless steel. Other materials such as tantalum have been used for extreme temperature requirements. These two major parts are spaced by porcelain insulators with copper vapor-shields placed on their anode end. Focusing (spacing) dimensions are not critical in this device. One leg of the filament is attached to the filament housing, and the other leg is passed through a boron nitride insulator to a power lug

A support ring, crucible, and holding pins make up the anode portion of this device. Shallow dimples are drilled in the crucible side to allow three-point support. The holding pins are fabricated from tantalum rod to withstand high crucible temperatures. Boron nitride rods with a central tungsten wire lead have been used with crucibles that could not be drilled. The holding pins are positioned with set screws so that they may be moved to accommodate crucibles of various diameters. Filament placement and insulator length are also variable to allow use of different crucible thicknesses.

Figure 5. BOMBARDMENT SOURCE SHOWING METHOD OF POWER CONNECTION

Vapor shields are placed on the porcelain insulators to prevent shorts in the systems. The shield ID should allow a spacing of at least 3/32 inch so that the device can be used up to 6 kilovolts.

Provisions have been made to support this source from either the anode or cathode assembly. The support bar becomes a filament lead if cathode attachment is used. This lead also supplies high voltage to the unit; hence, its associated feedthrough should be insulated for voltages up to 6 kilovolts. Anode support requires no insulation because this part of the device is grounded.

Figure 6 illustrates twin sources mounted on a jig designed by Kurtz.⁽⁴⁾ This jiggling used a common high voltage connection and separate leads to the other filament leg. In this way the sources could be operated independent of each other. The anode rings use a common flexible copper-ground strap.

Crucibles for this device were fabricated from a very wide variety of materials. The following list includes most of the materials that have been used:

1. Refractory metals such as tantalum, tungsten, molybdenum, and niobium.
2. Graphite and perforated graphite.
3. Refractory compounds including carbides, nitrides, borides, and silicides of tantalum, tungsten, molybdenum, niobium, and zirconium. These materials were produced by hot pressing under inert gas by the Ceramics Research and Development group.⁽⁵⁾
4. Ceramics including Al_2O_3 , BeO , MgO , and ZrO_2 .

Crucibles for this device were chosen on the basis of nonreactivity with a particular molten metal, good thermal shock properties and/or wettability by an evaporant metal. All of the crucibles were about 1 inch diameter by 1/4 inch thick and had a rounded depression about 3/4 inch diameter by 1/8 inch deep. This type of cavity design is used for directional evaporation, such as sharp perpendicular walls for narrow patterns.

(5) Kessel, D. J., et al., *Research and Development Progress Report for October, November, and December 1965, Metallurgy Section, Ceramics*, The Dow Chemical Company, Rocky Flats Division, RFP-700-E (Classified) (on subsequent quarterly reports).

(4) Kurtz, R. G., Personal communication.

Figure 6. ELECTRON BOMBARDMENT SOURCES MOUNTED ON SUPPORT JIGGING

OPERATION

A crucible was heated by first applying current to the filament until it reached a suitable emission temperature. High voltage for acceleration is then connected between the cathode and anode. The electron emission from a heated cathode varies with the absolute temperature in accordance with the following relation:

$$I = AT^2 e^{-b_0/T}$$

where I = emission per unit area at T degrees K° . The A and b_0 are constants.

Table I lists values of I at a series of temperatures for three filament materials.⁽⁶⁾

TABLE I
COMPARISON OF ELECTRON EMISSION
(I = amperes per square centimeter)

$T^\circ K$	$T^\circ C$	Tungsten	Niobium	Thoriated Tungsten
1200	927			3.95×10^{-5}
1400	1127			2.03×10^{-3}
1600	1327	9.27×10^{-7}	2.19×10^{-5}	4.06×10^{-2}
1800	1527	4.47×10^{-5}	6.95×10^{-4}	0.428
2000	1727	1.00×10^{-3}	1.16×10^{-2}	2.864
2200	1927	1.33×10^{-2}	0.115	

The device described in this report uses a niobium filament fabricated from 0.030-inch diameter wire. This filament has an emission of 7×10^{-4} amperes at $1500^\circ C$. Thoriated tungsten filaments would be very desirable for this source, but they were not readily available. The niobium filament has a surface area of approximately 2.8 cm^2 and is operated at about 20 amperes. Filament current is varied to obtain suitable emission for a particular temperature and crucible material. An accelerating voltage of 3000 volts, and a beam current of 200 milliamperes will heat a molybdenum crucible to $1500^\circ C$ in about 4 minutes.

(6) Dushman, S., *Scientific Foundations of Vacuum Technique*, 2nd Ed., New York, John Wiley and Sons.

Evaporant wire is fed into the heated crucible intermittently so that splattering will not occur. Usually the crucible temperature is adjusted about $100^\circ C$ above that necessary to produce a 10-micron metal vapor pressure. Metal vapor pressures versus temperature are listed in Dushman⁽⁶⁾ as well as in numerous other references.

It is important to oversize the nozzle on the wire feed mechanism. This will avoid jamming caused by evaporated metal building up on the nozzle end. In some instances it is also desirable to use a heated nozzle so that the evaporant wire does not act as a heat sink when it touches the crucible.

Some typical results for this device are listed in Table II. The "Time to Temperature" value is the time required to achieve evaporation temperature. A number of other metals have been evaporated with this source but are omitted for clarity.

TABLE II
EVAPORATION RATES AND CRUCIBLE DATA

Evaporant	Wire Size (in.)	Crucible Material	Time to Temp. (minutes)	Evaporation Rate
Al	0.060	TiB ₂ - sintered	3	0.36 g/min
Al	0.060	TiB ₂ - hot pressed	2.5	0.40 g/min
Al	0.060	Tungsten	1	2.0 g/min
Au	0.060	Tungsten	1	1.5 g/min
Au	0.060	TiB ₂ - sintered	3	1.0 g/min
Cu	0.060	Tantalum	3	0.5 g/min
Ag	0.045	Molybdenum	4	0.8 g/min
Ni	0.060	85 TiC/10 Mo/5 Co hot pressed	1.5	0.15 g/min

This source can be used for some metals in conjunction with the accelerated ion deposition technique developed by Mattox.⁽⁷⁾ It has been used successfully with aluminum and gold; however, it does not work with nickel because of space charge effects.

(6) Dushman, S., *loc. cit.*, p 4.

(7) Mattox, D. M., *Film Deposition Using Accelerated Ions*, Electrochemical Technology, Vol. 2, No. 9-10, September-October 1964.