Chapter 18

Design of Heat Source for Electron Bombardment Setup

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18.1 Introduction

Design of high temperature furnace for evaporating small quantities of material requires appropriate method of heating. Typical methods of heating are resistive heating, radiation heating, electron bombardment and electron beam evaporation. In resistive heating high current is passed through the crucible containing substrate. The heat is produced through the action of Joule heating in the form of I^2R. Here I is the current passing through the substrate and R is the electrical resistance offered by it. This system requires high current (in the order of 100s of Ampere) due to low electrical resistance which can give rise to other issues. This type of system is advantageous where a small cross-sectional area gives sufficient electrical resistance and few 10s of amperes of current generates enough heat. In radiation heating, the crucible is heated by the action of radiation heat transfer from filament. The filament is heated by resistive heating and its temperature is increased through volumetric heating. The heat transfer from filament to crucible is by the action of radiation. The heat emitted from filament will be proportional to area of filament exposed and fourth power of the temperature of the filament. Mathematically, the heat transfer will be proportional to [215]:

$$Q \propto A_{\text{filament}} \, T^4_{\text{filament}} \tag{18.1}$$

To achieve a temperature of 2000 ℃ in crucible, the filament should be operating at higher temperature. This will result in further complexities in maintaining both filament and crucible at high temperature. The electron bombardment method takes the advantage of thermionic emission of electron from the surface of a metal at high temperature. Metals like Tantalum or Tungsten emit sufficient number of electrons at temperature greater than 1700 ℃. Thus, one can make filament of these metals to generate thermionic electrons. The current density of the thermionic emission from material surface follows Richardson-Dushman equation as [216, 217]:

$$J = AT^2 exp\left(-\frac{\phi_0}{kT}\right) \tag{18.2}$$

Here, ϕ_0 is the work function of the material and A is the material constant. The thermionic emission current increases rapidly with temperature. By applying appropriate voltage on the crucible, desired power can be delivered to the crucible for heating. The major advantage of this method is that filament can be kept at a lower temperature than the crucible. However, this method has disadvantage of control. Since, the thermionic current increases exponentially with filament temperature, control of the power dissipated becomes difficult. Moreover, since crucible is at higher temperature, reducing temperature becomes complicated, as hotter crucible keeps feeding heat energy to the filament through radiation. This problem can be overcome by limiting the thermionic current in the space charge region. The electron beam evaporation is perhaps one of the widely used methods to evaporate variety of materials to generate vapour beam. This method is popularly used in physical vapour deposition process. The material contained within a water-cooled crucible is heated from the top using electron beam. The electron beam having intense power density in the order of few kW/cm^2 increases the temperature of the material and causes it to melt and then evaporate. In this method, the molten pool remains confined within its own solid material which is called skull. This method produces cleanest vapour as it remains virtually within its solid self and does not react with the crucible. However, this method requires elaborate setup for electron beam focusing and bending. Further, full material loaded in the crucible cannot be used for evaporation. Some material has to be left in the crucible to avoid puncturing of the crucible by electron beam. This method is advantageous for large scale evaporation in the order of 100s of gram per hour. As the crucible size is small, material inventory is in the order of few tens of grams and operating temperature requirement of 2000 °C electron bombardment is the method of choice for evaporation.

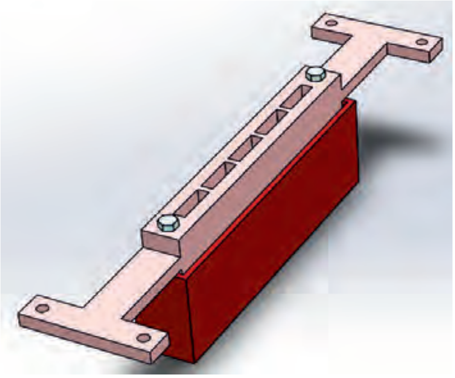

Figure 18.1: Schematic of a crucible-collimator setup.

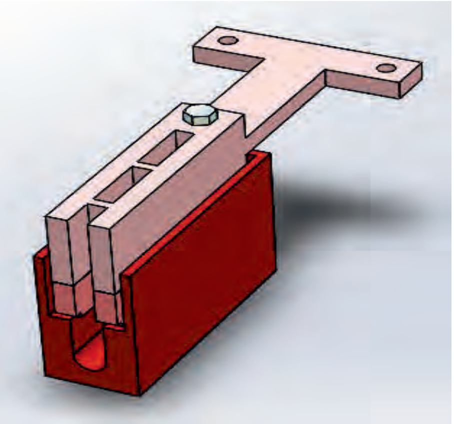

Figure 18.2: Section view of the crucible showing crucible (red), and 2 collimators (pink and light pink).

18.2 Calculation of Heating Power Requirement

The first consideration in the design of furnace is determination of power requirement. This can be known by calculating heat loss from the system. The typical design of the crucible along with the collimator is shown below in Figs. 18.1 & 18.2. The crucible is having volume capacity of 23 cubic-centimetres. The collimator structure is located above the crucible to provide directionality to the vapour beam. The complete crucible-collimator assembly is made up of Tungsten to sustain high temperatures. From Fig. 18.2 it can be understood that the material (charge) to be evaporated will be kept in the lower portion of the crucible. The design requirements stipulate that charge (Lutetium) should be evaporated at a temperature of 2000 ℃ and top surface of the collimator to be maintained at 1700 ℃ to avoid solidification. The mass of Lutetium loaded in the crucible will be approximately 200 g, therefore, one can safely assume that temperature gradient within crucible and charge will be negligible. The various parameters are listed in table 18.1.

Table 18.1: Important Parameters.

Parameters	**Values**
Outer surface area of the crucible, A_c	245.25 sq. cm
Surface area of the top surface of the collimator, A_t	22.37 sq. cm
Crucible Temperature, T_c	2000 ℃
Collimator top surface temperature, T_t	1700 ℃
Ambient Temperature T_{ambient}	27 ℃
Emissivity of Tungsten, ϵ [215]	0.35
Heat loss from the crucible and top slit	$\sigma\epsilon\ A\left({T_c}^4 - T^4_{\text{ambient}}\right)$=14.3 kW (T in K)

The above calculation for heat loss does not take into account of heat loss from the middle collimator, side faces of top collimator and from the wing area of the top collimator. However, this number 14.3 kW gives us an approximation of the power required to maintain the temperature of the crucible. Using heat shields will reduce the heat loss from the crucible. For this operating range temperature, heat shields of molybdenum/tantalum/tungsten can be used. Molybdenum has an emissivity of 0.6, tantalum and tungsten has emissivity of 0.35 each [215]. For cost effectiveness, molybdenum heat shields will be used. It is known that radiation heat transfer decreases as the number of shields are increased and this is generally given by relation [215]:

$$Q \propto \frac{1}{1 + \text{no. of shields}} \tag{18.3}$$

Depending upon material availability and fabrication ease, simulation design with 6 number of radiation shields was taken up. The sketch of the design is shown below in Fig. 18.3. The key feature of the above design is that crucible assembly and heat shield assembly is supported through alumina insulators. This allows us to provide different electrical potentials to the components. The thermal radiation analysis is done in simulation, using Steady-State

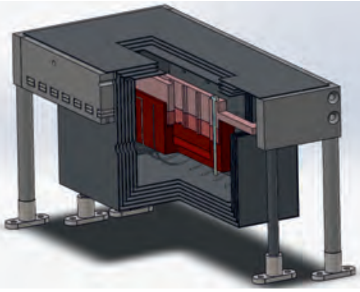

Figure 18.3: Sketch of the crucible and heat shield assembly. The assembly also shows the filament wire.

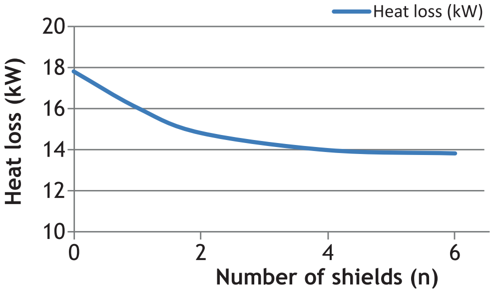

Figure 18.4: Variation of heat loss from the crucible-collimator assembly as number of shields is increased.

temperature module. Initially for the analysis, the crucible temperature is kept fixed at 2000 ℃ and collimator top surface temperature is kept at 1700 ℃. Only side heat shields are kept in the analysis and top heat shields were removed. The radiation is heat loss is checked in the crucible and collimator assembly. The simulation is done for different number of heat shields. The results obtained are shown in Fig. 18.4. From above curve, it is clear that on using 6 nos. of heat shields the heat loss is 14 kW. It is also observed that increasing number of heat shields beyond 4 does not offer much advantage. Therefore, the initial design is retained. The final configuration is further refined by putting some of the radiation heat shields on the top side. This further reduces the heat loss from the crucible-collimator assembly. In the final configuration, the heat loss from the crucible-collimator assembly comes out to be 11.6 kW. The simulation is run again in the final configuration. This time the crucible is given heat flow as 12 kW distributed equally over two side surfaces and bottom surface of the crucible. The emissivity of the tungsten crucible is taken as 0.35 and of molybdenum shields as of 0.6. The frame is made of SS 304 and its emissivity is taken as 0.8. The ambient temperature is taken as 30 ℃. No thermal contact is assumed between any of the surfaces. The section view of the geometry taken is shown in Fig. 18.5 and the simulation results obtained are shown in Fig. 18.6. The temperature of the crucible bottom and side surface is 2100 ℃, and temperature of inner shield is 1550 ℃. The temperature of the outer shield is 800 ℃. The frame which supports heat shields reaches to a peak temperature of 340 ℃, with average temperature sustained at 300 ℃. Also, the plot of the various temperatures with the power supplied to the crucible is as shown in Fig. 18.7.

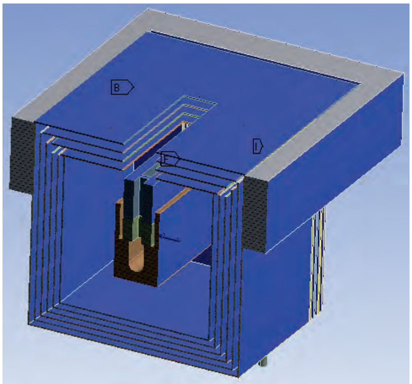

Figure 18.5: Section view of the geometry taken in simulation workbench.

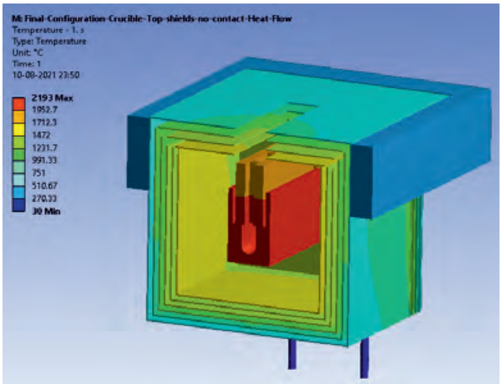

Figure 18.6: Section view of the temperature distribution of the assembly.

18.3 Design of the Filament

The filament in this setup will supply the thermionic current also known as emission current to the crucible. The crucible will be kept at positive potential so that thermionic electrons will attract to it. The power dissipated to the crucible will be equal to the product of thermionic current and voltage of the crucible. The thermionic current from the filament will be dependent upon the temperature of the filament and surface area of the emission. The emission current density is given by Richardson-Dushman Eq. (18.2) [216, 217]. This emission current is however limited by high density of the electron in the filament region. This phenomenon is called space-charge effect and maximum current density through this

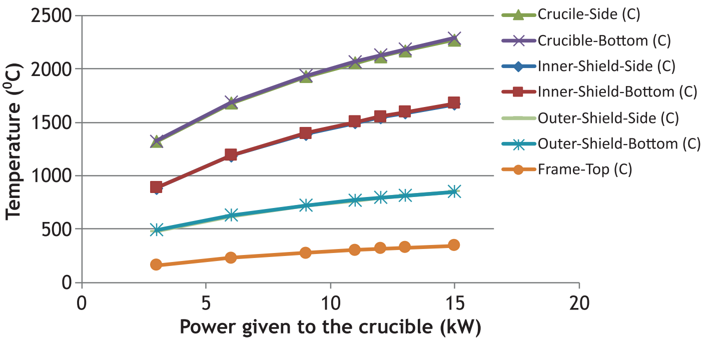

Figure 18.7: Temperature of the various surfaces as function of power supplied to the crucible.

region is given by Child-Langmuir's Law [218, 219]:

$$J_{CL} = \frac{4}{9}\varepsilon_0 \sqrt{\frac{2e}{m_e}} \frac{V^{3/2}}{d^2} \tag{18.4}$$

where V and d are the potential difference and the distance between anode and cathode, respectively; m_e and e are the electron mass and charge, respectively and ϵ_0 is the permittivity of free space. For electron in vacuum, the above Eq. (18.4) reduces to [220]:

$$J_{CL} = 0.0002334 \frac{V^{3/2}}{d^2} \tag{18.5}$$

Here, J_{CL} is in A/cm^2; V is in volt and d is in millimeter. As it is evident, there are two competing phenomena occurring during emission current. The Richardson-Dushman equation tries to increase the emission current as an exponential function of filament temperature, while space-charge effect tries to limit the emission current as a function of Child-Langmuir law. It is always better to operate the filament in the space-charge limiting region. During operation, control of filament temperature is difficult, as to reduce emission current, temperature has to be reduced which is difficult due to presence of high temperature crucible in front of it. Thus, the positive feedback will be delayed and there are chances of runaway control. This will lead to:

$$P \propto \; exp(T_{\text{filament}}) \tag{18.6}$$

The emission current in this region can be controlled by changing the anode-cathode voltages. Thus, emission current in this case will be proportional to one and half the power of voltage. Further, change in voltage will also change the power to it. Hence, in mathematical notations:

$$I \propto \; V^{3/2} \tag{18.7}$$

$$P \propto \; VI \tag{18.8}$$

$$P \propto \; V^{5/2} \tag{18.9}$$

Thus, the design of the filament is done for space-charge limiting operation.

a) Power requirement: 12 kW

b) Design Power (1.25 x 12kW): 15 kW

Assumptions:

c) V_{Crucible} : 2000 V

d) Filament temperature: 2300 K
e) Filament Material: Tantalum
f) Distance between filament and crucible: 12 mm
g) Diameter of the filament: 1 mm

Calculations:

1. Emission (thermionic) current required: 7.5 Amp
2. Current density from Richardson-Dushman, Eq. (18.2): 0.1846 A/cm^2
3. Current density from Space-Charge effect: 0.145 A/cm^2
4. Max current density possible: 0.145 A/cm^2
5. Filament active surface area required: 51.73 cm^2
6. Filament active length required: 164.67 cm
7. Heat loss from the filament (P_f): 500 W (approx.)
8. Resistivity of the Tantalum at 2300 K [221]: 9 x 10^{-7} Ωm
9. Electrical resistance offered by the active length: 1.887 Ω
10. Line current required (using $P_f = I^2$ R): 16.27 A
11. Voltage Required: 30.7 V

The optimisation of the filament length and temperatures requires some iteration. The above are the values obtained after final iteration for acceptable range.

18.4 Conclusion

There are numerous methods available for design of the heating furnace for small evaporation. The electron beam bombardment method has been selected for this application. Initially, power requirement has been determined to keep the crucible operating at 2300 K. Heat shields were provided to keep the power requirement low and 6 numbers of shields are used. After determination and optimisation of the power requirement, filament design parameters are arrived. The filament is designed keeping in mind about availability of material onsite and availability of power supply. The parameters of the filament are optimised for operation in space charge limiting region to have better control on the process.

Frequently Asked Questions

Q1. What are different materials used for making crucible for high temperature?
Q2. Why does change in heat loss from the crucible become less as one increases the number of heat shields?
Q3. How can one ensure that simulation results from software are correct?
Q4. The resistive heating is involved in all the methods through one way or other. Find out where in the methods it is involved.