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P.B. Sewell

GENERAL GUIDELINES FOR OPERATING ES-423E LaB_6 CATHODES

1. Introduction

Detailed instructions for the set-up of LaB_6 cathodes are usually included in the specific instrument operating manual. However, occasionally it is necessary to determine the optimum operating conditions for LaB_6 cathodes when such information is not readily available. This is frequently the case in modified equipment or where the ES-423E LaB_6 cathode is not the one specified in the instrument instruction manual.

These notes are offered as a general guideline for the set-up of ES-423E Extended Life LaB_6 cathodes (and also the predecessor ES-423B cathodes) where detailed instructions are not available for the electron gun in question. Some general information is also included.

2. LaB_6 Cathodes - General

Cathodes of different styles differ mainly in the method of mounting and heating of the LaB_6 crystal. The methods of mounting have been reviewed in the literature[1] and vary with the evolution of the various technologies. They may be divided into three basic groups, namely: Vogel mounts, wire mounts, and carbon rod mounts. The ES-423E extended life cathode is a carbon rod mount. There are major performance and lifetime differences between the three groups, but for the purposes of this discussion, only the difference in the electrical characteristics of the various mounts needs to be addressed. This may result in dramatically different settings on the heater control when using different cathodes.

The overall power requirements for heating the various cathodes are quite similar. However, carbon-rod heater cathodes made by Kimball Physics are of higher resistance than, for example, the wire heater cathodes Model 3 from Denka. Hence, if the control of the heater is voltage related, as in Philips Microscopes, then the control knob setting for Kimball Physics cathodes will be much higher than that for wire heated cathodes at the same operating temperature. However, if the control is power related, as for many of the JEOL microscopes, then the setting of the control knob is likely to be quite similar for the two types.

Unfortunately, very few instruments can measure cathode current directly, thus specific calibrations of cathode current versus cathode temperature are of lesser value in determining set-up parameters. If the cathode current is measurable, then this information will help to determine the optimum operating conditions.

It will be assumed in the following discussion that no direct measurements of cathode voltage or current are available, and that the operator has only the total emission current and the auto-bias settings as guides to cathode operation.

3. Basic Gun Parameters

For fixed gun geometry, the only mechanical variable available to the operator is the setting of the filament height within the Wehnelt aperture. If a replaceable Wehnelt aperture is fitted in the gun, then this aperture diameter may also be changed. As most electron guns in commercial TEM's and SEM's use auto-bias operation, the only electrical variable other than the cathode heating, is the value of the auto-bias resistor. Experimenting with the set-up of LaB_6 cathodes in an auto-bias supply with automatic bias compensation is difficult. Manual fixed bias settings should be used if possible.

A brief review of the inter-relation of gun variables will help to understand the prime features that control the set-up of a cathode for optimum operation:

3.1 Cathode Height: The cathode height, h, is defined as the distance between the cathode emitting surface, and the outer surface (anode side) of the Wehnelt aperture, see Figure 1. The setting of the cathode height in the Wehnelt is the principal adjustment required of the operator. This height setting is generally recommended by the manufacturer for their specific gun. The height setting can be best specified in terms of a ratio of the aperture diameter to the height setting as shown in Figure 1. For low brightness cathodes, such as tungsten hairpins, the setting is such that this ratio is between 1 and 2. However, for high brightness cathodes, a much higher electric field is required to overcome space charge at the cathode tip, such that this ratio is generally between 5 and 7.5. Hence, for a Wehnelt aperture diameter of 750 μm, an operating height setting of 150 to 100 μm is required.

[1] C.K. Crawford, "Mounting Methods and Operating Characteristics of LaB_6 Cathodes," SEM (1), (1979), pp. 19 - 30.

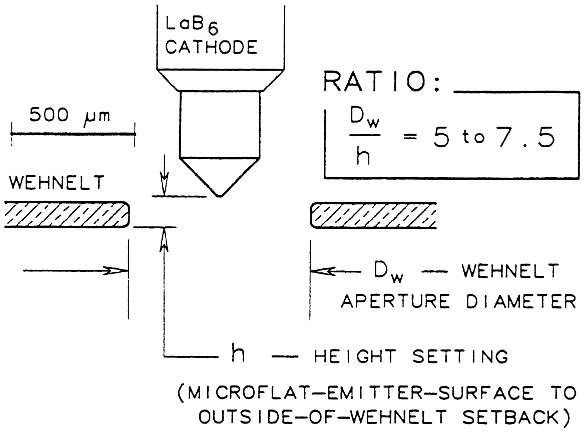

Figure 1. *Scale drawing of KPI ES-423E LaB_6 Cathode set at height 125 μm in a Wehnelt of 100 μm thickness with an aperture diameter of 750 μm. The ratio of D_w to h is 6, typical of that required for high brightness performance.*

This ratio does not apply to guns having tapered conical Wehnelts, such as those used in JEOL instruments. With such a Wehnelt configuration, adequate brightness from LaB_6 is obtained with a D_w/h ratio of between 1.6 and 2 or a height setting of 0.6 to 0.5 mm in a 1 mm diameter aperture.

The initial cathode height setting is done with the cathode cold. However in operation, the tip of the cathode expands forward by amounts which vary with the type of mount. This expansion ranges from about 30 μm for carbon-rod heater cathodes and 50 μm for wire mounted cathodes. Expansion of the filament mounting posts can also contribute to the forward expansion of the cathode tip. Long mounting posts on some bases can contribute an additional expansion of some 40 μm for a total forward expansion of the cathode by about 80 μm.

3.2 <u>Cathode Centering</u>: Following the height setting, the centering of the tip of the cathode in the Wehnelt is most important. The standard emitting area of a conical ES-423E LaB_6 cathode is 15 μm in diameter. Both the size of the emitting area and the conical shape of the LaB_6 cathode make the centering of this type of emitter much more critical than the regular tungsten cathode. Pre-centered cathodes are rarely centered with the accuracy required for optimum operation of LaB_6 cathodes. It is recommended that centering be done with the aid of a metallurgical microscope with normal incidence illumination. The flat tips on truncated LaB_6 cathodes make this operation straightforward. (Radiused tips are more difficult to center and adjust in height using this method.) Centering should be done to an accuracy of about ±25 μm.

Poor centering will show up as an asymmetric development of the cross-over image and/or the beam profile during the saturation of the cathode.

3.3 <u>Gun Bias</u>: At the point of saturation or optimum operation of a cathode, the zero equipotential intercepts the truncated tip of the cathode near the edge of the microflat, as shown in Figure 2. Thus electron emission occurs only from that part of the cathode surface on the anode side of this equipotential. To a first approximation, total emission can be considered to come from the flat alone. Making this assumption and the further assumption that uniform emission comes from the area of the truncation, an estimate of the total emission current as a function of cathode temperature can be made. Table 1 shows this for a <100> flat of 15 μm diameter, assuming a work function of 2.69 eV. It is seen that between operating temperatures of 1700 and 1900 K, the total emission would be expected to vary between about 7 and 60 μA.

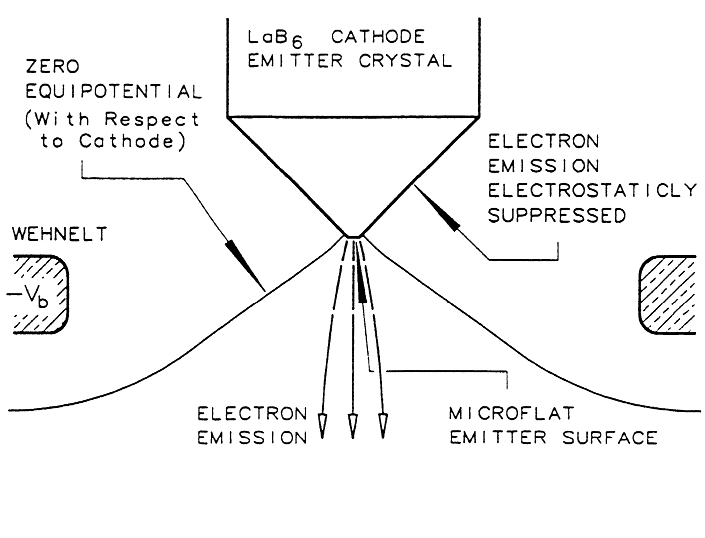

Figure 2. *Intercept of the zero-equipotential(measured with respect to the cathode) on the tip of a truncated LaB_6 cathode, defining the area of emission at saturation (optimum) bias.*

TABLE 1

Emission Characteristics of Truncated LaB_6 Cathode
KPI Type ES-423E
<100> Single Crystal with 15 μm diameter flat
(Area: 177 μm^2)

Temperature [K]	Cathode Loading [A/cm²]	Emission [μA]
1700	3.8	6.7
1750	6.8	12.0
1800	10.4	18.4
1850	20.0	35.3
1900	33.0	58.3
1950	50.0	88.4
2000	80.0	141.4

Zero-field cathode loading based on work function of <100> surface as 2.69 eV.

Total emission based on uniform emission over surface of truncation at 177 μm^2.

Experimental emission expected to be higher due to slight field reduction of work function.

The bias value at which this optimum emission occurs is a function of the gun geometry, the Wehnelt to anode field, and the height setting of the cathode within the Wehnelt. Computed cut-off voltages for a gun with parallel Wehnelt and Anode electrodes are shown in Table 2, for the specific value of Wehnelt diameter of 500 μm, a Wehnelt to Anode spacing of 10 mm, and an accelerating voltage of 50 kV. Note the strong dependence of the cut-off bias voltage with height setting. For small height settings, the bias voltage for optimum emission is about 95% of the cut-off bias voltage.[2] Thus, for a specific height setting of 100 μm, the required bias is about -600 V, or some 20 V less than the cut-off value.

With the auto-bias circuit shown in Figure 3, this voltage is established from the product of the total emission current and the value of the bias resistor. For high brightness emission at 60 μA, from an LaB_6 cathode at about 1900 K, the bias resistor required is about 10 MΩ. For lower brightness operation, with a total emission of 7 μA from a cathode at about 1700 K, a bias resistor of 85.7 MΩ is required.

4. Practical Considerations

For a gun operating correctly, there is a direct relationship between the total emission current at optimum bias or saturation and the cathode temperature. However, if the correct value of bias resistor is not selected then the

TABLE 2

Computed Values of Cut-off Voltage for a Triode Gun
Parallel Anode and Wehnelt Geometry
Wehnelt Aperture: 500 μm diameter; 100 μm thick
Wehnelt to Anode Spacing: 10 mm
Accelerating Voltage: 50 kV

Height Setting h [mm]	Cut-off Bias $-V_e$ [V]	Diameter/Height Ratio
0.0	1055	∞
0.05	816	10
0.10	620	5
0.15	467	3.3
0.20	352	2.5

cathode can run at an excessive temperature resulting in a reduced life.

For the above situation with a -600 V bias voltage, if the bias resistor selected was only 2 MΩ, then a total emission current of 300 μA would be required. From Table 1, it is seen that the cathode temperature would need to be in excess of 2000 K for this emission to be achieved from a 15 μm diameter truncation.

In practice it is therefore desirable to test new cathode configurations with the highest bias values available. Saturated emission should then occur at a low cathode

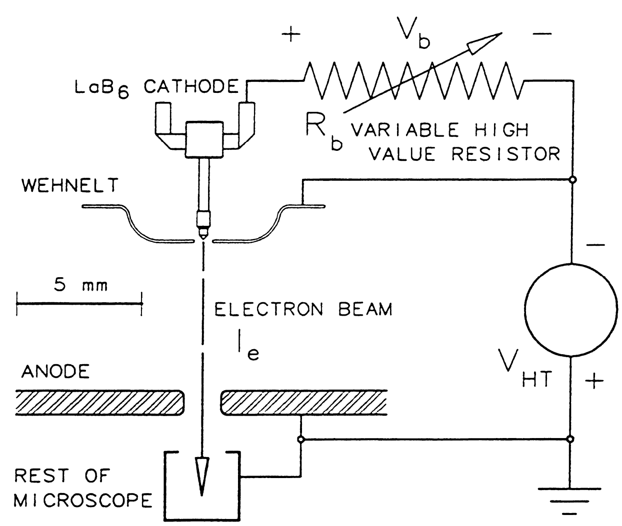

Figure 3. Basic electrode geometry of triode electron gun, showing auto-bias resistor R_b and operating bias $V_b = R_b \times I_e$ where I_e is the total emission current.

[2]M.E. Haine and P.A. Einstein, "Characteristics of the Hot Cathode Electron Microscope Gun," Brit. J. Appl. Phys. (3), (1952), pp. 40 - 46.

temperature. Reduce the bias resistor systematically and observe the new current at saturation. The product of the bias resistor and the saturation current should be a fairly constant voltage, the optimum bias value. Since the bias resistor is generally stepped, then nearest approximations occur. Table 3 shows this behavior for the Philips EM-430 bias circuit with five settings of brightness or bias resistor. The corrected emission of the Table is the observed emission less 5 μA of high-voltage bleeder-resistor leakage current, which occurs in the absence of electron emission. This value will vary with different instruments, and with time in any specific instrument. It should be checked with the high voltage ON, and the cathode heater OFF (or set at its minimum value).

5. Axial Brightness

Relative axial brightness in TEM's can usually be measured by observing the screen current at high magnification as a function of the total emission current. In SEM's the information can come from a measurement of the specimen current at small spot size settings. Such data are shown in Figure 4 for the Philips EM-420 TEM using tungsten and LaB_6 cathodes. If the brightness does not increase with increasing total emission at the optimum bias setting, then the gun is space charge limited. To move away from the space charge limit, the tip of the cathode needs to be moved further forward in the Wehnelt.

6. SUMMARY: Set-up Guidelines for LaB_6 Cathodes

(a) Truncated <100> cathodes with a tip diameter of about 15 μm are recommended for stability and uniformity of emission in the cross-over.

TABLE 3

Operating data from Philips EM-430
KPI type ES-423E LaB_6 Cathode

Emission Setting	Bias Setting [MΩ]	Corrected Emission [μA]	Bias Voltage [V]
1	99.5	5	497
2	49.5	10	495
3	24.9	19	473
4	12.0	37	444
5	6.0	75	450

Operating Voltage: 250 kV
Wehnelt Diameter: 500 μm
Height Setting Cold: 150 μm

(b) Set the tip-height to Wehnelt-diameter ratio to about 1/5 (tip height = 0.2 x Wehnelt diameter). Center the tip as accurately as possible, preferably using normal-incidence illumination in a metallurgical microscope.

(c) At the desired operating kV select the highest value of bias resistor available. If possible, it is desirable to determine these values from the circuit diagram of the gun power supply. The bias resistor control is often referred to as "Gun Brightness," or just "Brightness." Low Brightness uses high bias resistors and High Brightness uses lower value bias resistors. Check specific instrument manuals for details, or check with service engineers.

(d) Check the emission meter for leakage current. Note the value. Proceed to increase the heater current until emission is observed on a meter, on the microscope screen, or on a video-display. Optimize the emission by procedures recommended in the instrument manual. This usually requires adjusting the gun alignment and the beam alignment controls while observing the cross-over image or the beam profile (emission pattern).

If there are serious problems with the symmetry of the cross-over or the beam profile, then the cathode tip should be realigned within the Wehnelt.

With a symmetric beam, observe the emission current at saturation as a function of bias resistor setting; the result should be similar to that outlined in Table 3.

Note: If saturation of the cross-over or beam profile, at the maximum bias setting, can not be obtained with a total emission current less than 100 μA, then open the gun and increase the cathode height setting. Subsequent operation should then require a lower bias value.

(e) Measure axial beam current as a function of bias settings with increasing emission. If the beam current does not increase with total emission, then consider reducing the cathode height setting. Check the instrument manual for the minimum height settings recommended by the manufacturer. Note that the operating bias is the product of the emission current times the bias resistor. For small emission currents, the maximum bias resistance available in the instrument may not be

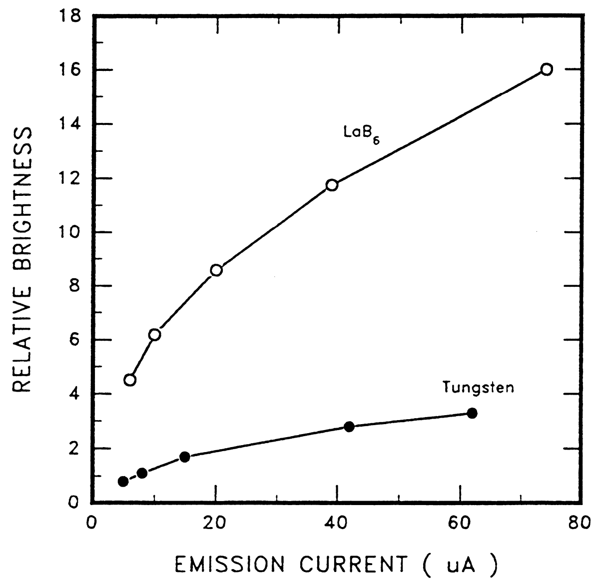

Figure 4. Relative brightness recorded from screen current versus total emission current for 5 Brightness settings with the emission at saturation. Data from Philips EM-420 TEM with LaB_6 and tungsten cathodes.

sufficient to achieve the bias required for saturation.

(f) A correctly set cathode should behave in the same general manner as shown in Table 3. Note that the absolute values of emission at saturation will vary with different gun geometries together with the computed value of the bias voltage. Axial brightness behavior similar to Figure 4 should also be observed.

(g) Failure to achieve good performance with an LaB_6 cathode will probably require re-evaluation of the gun geometry, particularly if the gun was not initially designed for LaB_6 operation. A common design fault is a range of bias resistor values which is too low with all possible settings. Older guns designed for tungsten frequently have a maximum bias resistor of 5 MΩ and large diameter Wehnelt apertures. The resistor range needs to be increased to at least 50 MΩ, and the Wehnelt aperture diameter reduced to 750 or 1000 μm, as first steps towards satisfactory LaB_6 operation.

7. Cathode Life

Cathode life is discussed in more detail in Kimball Physics Technical Bulletin #LaB_6- 02B.

Provided the gun vacuum is satisfactory, and provided the D_w/h ratio and bias resistance/voltage settings are properly adjusted (which in turn assure reasonable cathode operating temperatures), then the cathode life can be up to several thousand hours. Expected total emission currents can be roughly calculated from the emitting microflat area and the desired cathode emitted current density. For the standard 15 μm diameter microflats, optimum settings normally imply total-emission currents (at saturation) in the range of 60 to 80 μA for high brightness operation. For smaller 6 μm microflats, currents of about 10 μA are appropriate (again for high brightness operation). Incorrect settings of the D_w/h ratio and/or bias settings frequently result in excessive cathode temperatures, with a consequent rapid loss of LaB6 by direct evaporation.

It is useful to inspect the deposits on the inner surface of the Wehnelt. A dark, or dark purple, LaB_6 deposit suggests an excessive cathode temperature, unless the gun has been operating in truly clean ultra-high vacuum for a long period. The dark deposit indicates that direct LaB_6 evaporation, rather than the evaporation of oxides, is the predominant material loss mechanism. More generally, in 10^{-7} torr vacuums, the Wehnelt deposit is mostly a white or grey mixture of LaB_6 and its oxides, indicating that direct evaporation and oxidation evaporation are proceeding concurrently. Optical interference colors in transparent thin films indicate oxide formation without much LaB_6 evaporation; cathode temperature is close to normal, but the pressure is too high. Clearly, at best, deposit colors can give only a rough average of what the time-varying operating conditions have been; the more recent conditions tend to dominate.

A rough, white, powder-like deposit on the cathode itself indicates the cathode operating temperature is too low (not even the oxide can be evaporated), and the vacuum is excessively poor (too much oxide is being formed). A gun which is vented to air (dry nitrogen is preferred), without waiting for the gun structure to cool (a few minutes is appropriate), causes the cathode to become a dark blue to bluish silver color. Such a cathode can often be reactivated by over-temperature operation at 2000 to 2100 K for 10 to 15 minutes; the surface layer, presumably mostly LaB_9, evaporates away, exposing fresh LaB_6 underneath. As the emission reappears, it is important to reduce back to a normal 1750 to 1850 K operating temperature.

Short LaB_6 cathode life is frequently associated with a poor gun vacuum. Note that the pressure measured by the gun ion-pump current, or using an ionization gauge attached to a gun manifold, is most often more optimistic than the real gun pressure. Nearly all commercial electron microscopes have gun pumping systems that are not suitably designed for LaB_6 operation. The partial pressures of oxidizing gases, especially water vapor, are critical in determining gun performance and cathode life.