Design and optimization of directly heated LaB₆ cathode assemblies for electron-beam instruments

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A description is given of the design and testing of a directly heated, stable, electron source utilizing a single-crystal lanthanum hexaboride (LaB₆) cathode. The emitter mounting fixture consists of an adjustable molybdenum base unit supported on gas-impervious alumina or machinable glass. Single-crystal cathode rods are securely clamped and positioned between vitreous carbon jaws that are resistively heated. The complete assembly is designed to be a direct “plug-in” substitute for the conventional tungsten thermionic filaments used in electron-beam instruments. The cathode current density for ⟨110⟩ axial orientations is found to be ten times higher than that for ⟨100⟩ orientations under equivalent conditions, a value of 50 A cm⁻² being measured at 1500°C with an observed lifetime in excess of 300 h. Optimum vacuum conditions for high lifetime and stable operation are in the range 1×10⁻⁶ Torr and lower. Comparison values for the emission at various temperatures from other borides, and tungsten, are also given.

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INTRODUCTION

The performance of most electron-beam instruments is limited by the brightness of the electron source, since this in turn sets an upper bound on the maximum current density which can be produced in any beam formed by the instrument. The most widely used source, the tungsten thermionic filament, has a cathode current density Jₑ in the range 0.1–10 A cm⁻² and an evaporation-limited lifetime of about 30/Jₑ h. For an acceptable lifetime, of say, 30 h, Jₑ is thus only about 1 A cm⁻².

The most promising approach to simultaneously improving the brightness and lifetime, without going into the technological problems set by field emission, is the use of thermionic emitters with a lower work function than tungsten. Of the possible candidates, the most interesting is lanthanum hexaboride, LaB₆, originally investigated by Lafferty.¹ Although the electrical parameters of LaB₆ are satisfactory it is a highly reactive material when held at a high enough temperature to be an efficient thermionic emitter. For this reason the first practical LaB₆ sources developed by Broers²,³ used long (1 cm) rods of sintered LaB₆ which were held at one end in an oil bath to cool them and heated in the region of their other end by radiant energy, and electron bombardment, from a tungsten coil placed close to, but not touching, the rod. This design was shown to be capable of producing Jₑ ≥ 5 A cm⁻² with lifetimes of 500 h. However, the gun assembly was bulky, it needed two extra power-supplies and generated about 60 W of heat.

More recently Crawford⁴ and Ramachandran⁵ have designed directly heated LaB₆ cathodes in which a small rod of LaB₆ is attached directly to a material with which it is not reactive such as graphite⁴ or rhenium.⁵ The rod is then resistively heated by the current passing through the support material. Such a design needs only a low amount of heating power (typically 5 W) because of the small mass of LaB₆ used. It is therefore possible to tailor the resistance and thermal mass of the assembly so that it can be driven from the normal filament current supply of an electron microscope, and the cathode unit can be made as a plug-in replacement for the standard tungsten hairpin. Such devices are capable of providing a good level of performance⁶ but are fragile and inherently unstable.

The purpose of the work described here was to design and optimize a directly heated LaB₆ cathode assembly to function as a plug-in replacement for the tungsten hairpin units currently used on electron-beam writing and imaging machines. The units were to

(1) give a worthwhile increase in brightness (at least 5X);

(2) provide substantially improved operating lifetimes at this brightness, with an aim of at least 300 h;

(3) be very stable to allow for long production runs on electron lithography systems without the need for realignment;

(4) be compatible in all respects with the standard tungsten assembly to facilitate the changeover and subsequent operations.

These requirements were achieved by two separate projects. Firstly, a variety of cathode emitter materials were examined and characterized and secondly, a rugged and stable cathode mounting assembly was devised and tested. These are described in the remainder of this text.
CATHODE EMITTER MATERIALS

The cathode current density \( J_c \) of a thermionic emitter is given by Richardson's expression as:

\[
J_c = AT^2 \exp\left(-\frac{e\phi}{kT}\right) \text{ (A/cm}^2) \tag{1}
\]

where \( T \) is the cathode temperature in K, \( e \) the electronic charge and \( k \) the Boltzmann constant. \( \phi \) is the work function of the radiating surface (in volts), and \( A \) is the Richardson constant.

The most efficient electron sources will be those which have the highest \( A \) number and the lowest \( \phi \) value. The relative importance of \( A \) and \( \phi \) varies with the cathode temperature. However, for the best stability and lifetime \( T \) should be as low as possible and for \( T < 2000 \text{ K} \), \( \phi \) is the prime parameter in determining \( J_c \). While there is a considerable literature on the thermionic properties of lanthanum and other borides\(^7\), the data are unreliable for several reasons. In general, they were obtained for sintered powder material of unknown quality, and of variable density compared to the theoretical value. Secondly, the figures were mostly deduced from room temperature photoemission measurements and may not be valid at high temperatures.

An experimental diode gun was therefore set up in an ion pumped UHV vacuum chamber as shown in Fig. 1. The cathode rods were mounted in the cathode support assembly described later and heated by an externally adjustable ac supply. The cathode temperature could be measured directly by a pyrometer focused on to the emitting tip. An emissivity of 0.8 is listed in standard tables, but a calibration run on a LaB\(_6\) “black body” yielded a value close to 1.0 and this was used for all further measurements. The cathode was held at from 500 to 1000 V negative with respect to the anode. This took the form of a large fluorescent screen deposited on to metal. By this means the emission distribution of the cathode could be viewed directly. The cathode itself was rotatable with respect to the screen and could be turned through a calibrated range of ±50°. In the center of the anode a small hole allowed a fraction of the incident electrons to pass through and fall into a Faraday cup connected to a precision pico-ammeter. Electrostatic alignment was provided to maximize the current into the cup.

The current entering the cup \( I_F = J_F \pi r^2 \), where \( J_F \) is the current density at the entrance to the cup and \( r \) the radius of the entrance aperture. The cathode current density \( J_c \) is then

\[
J_c = J_F \frac{r^2}{r_0^2} = \frac{I_F}{\pi r_0^2} \tag{2}
\]

where \( r_0 \) is the effective tip radius of the cathode. Since \( J_c \propto I_F \) for any given cathode, a plot of \( \ln(J_F/T^2) \) against \( 1/T \) can be seen from Eqs. (1) and (2) to give a straight line of slope \( e\phi/k \).

Substituting values for \( e \) and \( k \), a plot of \( \ln(J_F/T^2) \) against \( 1.16 \times 10^4/T \) gives \( \phi \) directly in volts. By assuming a value of \( r_0 \) the absolute value of \( J_c \) can be estimated. From previous measurements\(^8\) \( r_0 \) was taken to be 3 \( \mu \text{m} \) for cathodes prepared by the electropolishing techniques described later.

The system pressure was approximately \( 5 \times 10^{-7} \text{ Torr} \) generated by an oil-free pumping system and monitored near the emitter mounting assembly.

RESULTS

Initial observations were made of the emission patterns from cathode rods of sintered LaB\(_6\) powder of the type used for the original measurements.\(^6\) Patterns of the type shown in Fig. 2 were obtained. These show a diffuse central bright lobe with a second broad bright ring at about 35° to the rod axis. Over periods of the order of an hour the relative brightness of portions of the pattern showed marked changes and this effect became more pronounced for temperatures in excess of 1300°C. This is clearly due to the processes of grain growth and recrystallization and the resultant variable crystal orientation of the tip which are inherent in any polycrystalline compact of this type. Because these variations give rise directly to fluctuation in the emission from the cathode, no sintered-powder cathode can be considered suitable for use as a stable source. All further investigations were therefore made on single crystal rods in which such an effect is totally absent. It is also to be expected that \( A \) and \( \phi \) values will depend on the choice of rod orientation and thus a variety of orientations were examined to find which was the best for each material.

Crystals of LaB\(_6\) were prepared by cutting suitable rods from melted boules of LaB\(_6\) (in which case the orientation could be chosen) or by using the needlelike crystals of LaB\(_6\) produced from the aluminum flux process\(^8\) (which were generally of (100) type).

Figure 3 shows the emission pattern observed for (100) crystals. There is no strong bright central spot, but four major lobes are visible forming a square around the center. Farther out from the center, additional bright spots were observed.

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This type of pattern became visible as soon as the cathode was activated (see later) and showed little variation with temperature up to around 1600°C. No changes in the pattern were noted for running periods in excess of 24 h at 1550°C. Only under conditions of poor vacuum (pressure greater than \(5 \times 10^{-6}\) Torr) were substantial changes noted.

Figure 4 shows the typical results of a plot of \(\ln(J_c/T^2)\) against \(1/T\) for (100) orientation \(\text{LaB}_6\) as the temperature was cycled from about 900 to 1500°C and back again. In the initial region \(AB\) the effective work function is very low with \(\phi \approx 1.5\) eV, but no recognizable emission pattern is produced by the cathode and the output from the cathode is small \((J_c \leq 0.1\) A cm\(^{-2}\)). At about 1100°C an emission pattern showing the expected symmetry forms on the screen and this is accompanied by a large increase in output from the cathode. Small increases in the amount of power supplied to the cathode heater cause large changes in the emitted current and measured cathode temperature. This is the process of “activation.” It seems that in this region \(BC\), the surface contaminants are removed and free \(\text{LaB}_6\) is available on the cathode face. The resultant increase in emission leads to an increase in the cathode temperature and as a result the true work function is very difficult to measure over this range. The apparent value is high, typically 4.5 eV, but it is not possible to hold the cathode at any temperature within the range \(BC\) because the emitter will either build to full activation (i.e., the temperature will rise over a period of minutes) or deactivate entirely (the temperature falls) and so the value is of no significance. This wide region of instability is a major problem with (100) oriented \(\text{LaB}_6\) and stable operation of the source will only be possible when the temperature is high enough to keep well out of this portion of the characteristic. Use of a current-controlled power supply may be desirable for improved stability and temperature control for emitter operation near to the activation temperature range.

Above point \(C\) (about 1280°C) the cathode behavior stabilizes and the emitter shows a characteristic work function of about 3 eV. It was, however, found that in many cases the output from the cathode “saturated” at about 1450°C and no further improvement in performance was obtained by further increase of the temperature. This effect did not appear to be dependent on the sharpness of the emitting tip and consequently it does not seem to be due to space-charge limiting. Similar effects have been observed by other workers.9

On reducing the temperature the emission follows the line \(DC\) and \(C'B\) but continues to deactivate for a further time \(BE\) before eventually rejoining the original line \(AB\). An increase in temperature leads to the cathode following the same general form of behavior.

At 1350°C the estimated value of \(J_c\) was 5 A cm\(^{-2}\). This is consistent with a value of \(\phi \approx 3\) eV and a Richardson’s number of \(\sim 100\). While this is an acceptable performance it follows from the appearance of the \((100)\) emission pattern that the \(\{100\}\) face is not the optimum, lowest \(\phi\), orientation. Preliminary field ion microscopy studies by Swanson and Dickinson10 also support this observation. We have confirmed the nonoptimum aspect of \((100)\) by measuring the axial current output of the cathode while rotating it about an axis normal to the \(\{100\}\) direction in the emitter rod. The result shown in Fig. 5 and normalized to the \((100)\) output indicate that, with the angular resolution of the sampling technique, the output passes through a minimum when the \(\{510\}\) axes are selected and through maxima when the \(\{110\}\) axes are chosen.11 The fourfold symmetry of the emission pattern is the consequence of the four \(\{110\}\) faces lying at 45° to the \(\{100\}\) axis. The results indicated that for the same cathode temperature the cathode current density of \(\{110\}\) faces was an order of magnitude higher than for the \(\{100\}\) faces.

This was further verified by preparing rods of \((110)\) orientation and performing a similar sequence of measurements on these. It was found in addition that faces of this orientation activated at a lower temperature (\(\sim 1200°C\)) than for the \(\{100\}\) case. The activation process was accomplished rapidly and for any temperature in excess of 1220°C the emitter was fully activated and stable. Deactivation did not start until about 1100°C. The narrower region over which activation occurs and the resistance of the emitter to deactivation makes cathodes of this orientation much easier to control than those of the \(\{100\}\) type.
The results of the measurements performed on the [110] material are listed in Table I together with similar data for a variety of other emitters. All the data refer to fully activated cathodes. The accuracy of the techniques (as checked by the value determined for tungsten) is good when the emission pattern is essentially diffuse as is the case for a polycrystalline cathode. However, in the case of single-crystal emitters there is a possible problem. Because of the limited angular resolution of the measuring system more than one emitting face may contribute to the result. If these faces have widely different work functions the data may only give a poor fit to a Richardson plot, and the calculated value of $A$. While every precaution was taken to avoid this difficulty it is probable that the accuracy of the results are affected by this, and in the case of (110) orientation LaB$_6$ it was found that the use of a Wehnelt grid cap (which removes off-axis emission lobes) gives a substantially lower value for $\phi$ (Table I). Additional properties of LaB$_6$ are listed in Table II.

Although the value of $\phi$ is of major significance the constant $A$ is also of interest because this will determine the magnitude of $J_e$ at a given temperature. "$A$" can be deduced from the Richardson plot (see Table I), but for comparative purposes we have found it more instructive to plot the axial emission current (which is directly proportional to $J_e$ and hence to $A$).

**Table I.** Properties of LaB$_6$.

| Material          | Orientation | $\phi$ (eV) | $A$  | Comments                           |
|-------------------|-------------|-------------|------|------------------------------------|
| LaB$_6$           | (100)       | $\geq 3$    | 100  |                                    |
| LaB$_6$           | (110)       | 2.5         | 60   |                                    |
| LaB$_6$/BaCO$_3$  | (100)       | 2.1         |      | Surface coating of BaCO$_3$,       |
|                   |             |             |      | Very unstable at                   |
|                   |             |             |      | temperature however lower          |
|                   |             |             |      | observed activation and            |
|                   |             |             |      | deactivation temperature            |
| LaB$_6$ sintered  |             | 4.5         | 100  |                                    |
| YB$_6$            |             | $\sim 1$    |      | Very unstable                      |
| CeB$_6$           | (100)       | 3           | 1    |                                    |
| PrB$_6$           |             | 2.3         |      | Unstable                           |
| NdB$_6$           |             | 1.6         | 0.1  |                                    |
| La$_{0.8}$Y$_{0.2}$B$_6$ |             | 2.5        |      | Unstable, are melted               |
| W                 |             | 5.5         | 100  |                                    |

* Value using Wehnelt (see text). *b Polycrystalline.

**Table II.** Properties of LaB$_6$.

| Property                  | Value                  | Remarks                          |
|---------------------------|------------------------|----------------------------------|
| Melting point             | 2530°C (congruently melting) |
| Crystal structure         | Cubic-Pm3m (221)       |
| Density                   | $4.714 \pm 0.001 \text{ g/cm}^3$ |
| Color                     | Pinkish-purple         |
| Electrical resistivity    | $120 \mu\Omega \text{ cm at } 1600^\circ \text{C}$ |
|                          | $15 \mu\Omega \text{ cm at } 20^\circ \text{C}$ |
| Temperature coefficient   | $4.9 \times 10^{-6}$   |
| Hall coefficient          | $-5 \times 10^9 \text{ cm}^2/\text{V s}$ |
| Thermal emf               | $0.1 \mu\Omega/\text{degree}$ |
| Mobility of carriers      | $33.1 \text{ cm}^2/\text{V s}$ |
| Number of conduction      | 0.9                    |
| carriers per metal atom   |                        |
| Work function (thermionic)| $2.5-3.0 \text{ eV}$   |
| Chemical solubility       | v. soluble in HNO$_3$  |
|                          | insoluble in H$_2$O, HCl, KOH, NaOH, alcohol |
| Single-crystal emitter    | (100) 1280°C C        |
| activation temperature   |                        |
|                          | (110) 1200°C C        |

* For (110) single crystal at 1545 K.
CATHODE emitter will then be that one which provides the highest and the inverse exponential of $\phi/kT$ against $T$. The optimum emitter will then be one which provides the highest current at the lowest temperature. Figure 6 shows such a plot for all the systems tested. In each case the values refer to the activated cathode only, and the graph only covers that temperature range over which the emitter was usefully stable.

It is clear from this that (110) oriented LaB$_6$ is superior over the entire temperature range of interest, with (100) LaB$_6$ next. Although other materials display lower work functions they either have a smaller $A$ value, or are insufficiently stable in the desired temperature range, to make their use practical. We therefore conclude that single-crystal LaB$_6$ of (110) axial orientation is the optimum choice for the cathode material.

At temperatures of 1100°C and higher, a value for $f_P$ of about 50 A cm$^{-2}$ is available. The projected lifetime, as set by evaporation, under these conditions is in excess of 2000 h for a cathode power input of about 2.5 W.

**CATHODE ASSEMBLY DESIGN**

In the early designs for directly heated LaB$_6$ cathodes the emitter was supported by what was essentially a modified “hairpin” filament made of either molybdenum or rhenium. These materials have sufficiently low reactivity with hot LaB$_6$ to permit reasonable secure operation. The simplicity of the mount is however offset by two drawbacks. The mechanical stability of these assemblies is inherently poor because of the stress in the support resulting from the attachment of the LaB$_6$ block. This leads to considerable drift over a time scale of hours which makes such a system of little value for application to electron lithography systems where prolonged periods of operation without the necessity for realignment are required. Secondly, the thermal cycling which occurs during switch-on and switch-off rapidly causes the failure of any mounting system which relies on a chemical bond (such as LaB$_6$-Re) rather than a mechanical bond. This imposes the necessity of continuous constant-temperature operation which is not practical on most instruments.

In a later design the LaB$_6$ block was held between two graphite strips which gripped grooves in the emitter rod. While this design offers improved stability and reliability, it is fragile and liable to destruction during installation. For both this, and the previous designs there is also the problem that the rupture of the support leads to loss of the crystal, and mounting and aligning the crystal on the support calls for considerable manual dexterity.

It was therefore decided to adopt a rigid type of mount in which the emitter rod is held under pressure from two graphite pads which are supported by metal clamps. Various structures of this type have been previously reported by several workers. Such an arrangement has considerable advantages—it can readily be made rigid and free from thermal drift; no chemical bond is required and so thermal cycling is not a potential problem; insertion and alignment of new emitters is rapid and easy; the basic design is adaptable to fit most existing filament assemblies and there is sufficient design flexibility to make it possible to tailor the electrical and thermal loading of the cathode assembly to match the power output available from standard power supplies.

Figure 7 shows the configuration finally adopted for use. The materials specified for the assembly have been critically selected on the basis of considerable experimentation, as it is necessary to avoid combinations of reactive components and also to obtain desirable mechanical, electrical, and thermal properties. The main clamp is fabricated from molybdenum as this has a higher electrical conductivity and thermal conductivity than stainless steel as well as lower thermal expansion and reactivity. This is tensioned by molybdenum screws to minimize the effects of differential thermal expansion at the jaws. The carbon pads holding the emitter are constructed of V-25 grade vitreous carbon. This form of graphite contains a microstructure which consists of randomly oriented interwoven platelets of turbostratic basal planes and combines properties of low thermal and electrical conductivity. Unlike most graphites or carbon compounds it contains no filler, is nearly gas impervious, has good resistance to thermal shock and undergoes minimal reaction with LaB$_6$. It is stable thermally to $\sim$2500°C and is shaped with diamond tooling. The base of the assembly is made from alumina or Dow Corning machinable glass. In addition to being mechanically stable and resistant against thermal shock it is desirable that the base be nonporous and impervious to gases to prevent trapping and the formation of a virtual leak in the vacuum system.

During construction the metal surfaces were ground rather than milled as this was found to reduce the residual stresses in the assembly. Nevertheless, as a precaution, the completed units were backed out at 1500°C for 24 h at $10^{-6}$ Torr by closing the jaws without an emitter present and passing a suitable ac current through the assembly. This served both to stress-anneal and degas the units. In use the structures have proven reliable and stable up to 1650°C for periods of continuous operation of several hundreds of hours. Some detailed points of design remain to be completed in order to ensure even better long-term stability—especially the provision of a cross-tie tensioning spring to guarantee positive clamping. The basic principles of the design, however, have been sufficiently proven to be correct.

**PREPARATION OF THE EMITTER RODS**

Emitter rods were prepared to be of square section, approximately 0.25 mm square and about 1.75 mm long. Their mass is thus of the order of one milligram. These rods were
The emitter is then placed in the jaws while they are held to a spacing to finalize alignment and stabilize. After a period of two or three hours at a temperature of around 1100°C while monitoring the axial beam current. As activation takes place the beam current will increase by 50–100X for relatively small changes in filament current. Once activation is completed and the beam current has stabilized the emitter can be run up to the desired temperature. For a clean cathode, activation is normally completed in 2–3 min, but a contaminated cathode may require as much as 20 min. Because of the tip on the emitter no saturation knee in the emission current characteristic will be found, and care must therefore be taken not to overrun the emitter. If possible the emission pattern of the gun should be observed during this process, as the optimum running temperature will be the lowest one above activation which produces a single bright lobe on axis. Running the tip at too low a temperature or incorrectly biased or adjusted in the Wehnelt may all lead to multiple emission lobes which can readily be identified and correct if the emission pattern can be viewed. Figure 8(a) shows the inhomogeneous emission pattern obtained just above activation and Fig. 8(b) the pattern of a tip at 1150°C and correctly biased.

In operation the gun vacuum must be maintained at 10⁻⁶ Torr or better if stable emission is to be achieved. Above 10⁻⁶ the emission pattern will be affected.

**FINAL ALIGNMENT AND OPERATION**

Before the emitter is inserted in the holder the jaws are set to a spacing 0.10 mm less than the width of the rod. Thus for a 0.25-mm rectangular rod, the jaw spacing is set to 0.150 mm. The emitter is then placed in the jaws while they are held apart with a suitable tool. With the crystal in position the jaws are allowed to close on to the rod. Because of the adjustment described above, the rod is now held under a positive pressure which is sufficient to clamp it without causing damage to the graphite pads.

When in position the emitter should be accurately normal to the base of the cathode structure and centered between the two jaws. A tilt of as little as 3° will be sufficient to cause a loss in performance and problems with alignment of the source when used in the microscope. Slotted grooves on the jaw faces may aid in future designs for absolute crystal alignment. This feature, however, remains untested at this time.

Immediately after assembly the resistance of the completed unit as measured across the filament pins at the mounting base will be of the order of 3 Ω. With the assembly in a clean vacuum, the unit is then heated by passing a current through it. This current is gradually built up so that a tip temperature of about 1000°C is attained in about 30 min. During this period the resistance of the unit will fall to 1 Ω or less and should stabilize. After a period of two or three hours at 1200°C, the assembly can be removed and the tip checked for alignment and any signs of relative motion between the crystal, the carbon pads and the jaws. Assuming that the support assembly has been properly stress-annealed as described above no problems should be encountered. If any motion has occurred, the jaws must be retensioned, the crystal must be remounted and the running-in process repeated until stability is achieved.

In operation the cathodes perform in a similar way to pointed tungsten cathodes but with the additional complication of the activation time. The preferred mode of use is as follows. The cathode is slowly run up (55 min) to a temperature of around 1100°C while monitoring the axial beam current. As activation takes place the beam current will increase by 50–100X for relatively small changes in filament current. Once activation is completed and the beam current has stabilized the emitter can be run up to the desired temperature. For a clean cathode, activation is normally completed in 2–3 min, but a contaminated cathode may require as much as 20 min. Because of the tip on the emitter no saturation knee in the emission current characteristic will be found, and care must therefore be taken not to overrun the emitter. If possible the emission pattern of the gun should be observed during this process, as the optimum running temperature will be the lowest one above activation which produces a single bright lobe on axis. Running the tip at too low a temperature or incorrectly biased or adjusted in the Wehnelt may all lead to multiple emission lobes which can readily be identified and correct if the emission pattern can be viewed. Figure 8(a) shows the inhomogeneous emission pattern obtained just above activation and Fig. 8(b) the pattern of a tip at 1150°C and correctly biased.

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Torr a stable oxide will form on the emitter and this leads to a marked decrease in performance. It is believed that this is because the efficient operation of the tip depends on the surface being of stoichiometric composition. Below $10^{-6}$ Torr the oxide is not stable and will not form for temperatures of $1450^\circ C$ or above.

**SUMMARY**

A stable, high brightness electron source using a single-crystal LaB$_6$ emitter has been designed. With an emitter of (110) orientation a cathode current density of the order of $50 \, A \, cm^{-2}$ is available at $1500^\circ C$ with an experimentally observed lifetime in excess of $300 \, h$. Improved resolution might be obtainable in those devices presently utilizing tungsten emitters as electron sources in view of lower operating temperatures for LaB$_6$.

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