Kimball Physics, Inc.
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THE RELATIONSHIP BETWEEN LaB_6 CATHODE LIFE AND GUN VACUUM

1. Introduction

Lanthanum Hexaboride (LaB_6) cathodes are now widely used in a large number of commercial electron optical instruments. However, in spite of some 20 years of experience with this type of cathode, there is still a poor appreciation for the need of good vacuum in electron guns employing LaB_6 cathodes. Instruments used for surface analysis are the exception, since the vacuum requirements for reliable analysis are generally better than 10^{-9} torr, which is already an ideal environment for LaB_6 cathodes.

In scanning and transmission electron microscopes, the requirements for good vacuum at the specimen level are less stringent. These instruments are still primarily sealed with elastomer O-rings, and vacuum levels generally range between 10^{-5} to 10^{-7} torr. Instrument manufacturers have attempted to improve the vacuums in the cathode region by adding ion pumps to the gun chamber, hoping to provide an operating pressure of about 10^{-7} torr at the level of the cathode. Even though improvements have been made, it is still rare to find an instrument that reliably achieves a pressure of 10^{-7} torr in the region of the cathode during normal operation.

Additionally, very few instruments monitor the pressure in the vicinity of the cathode itself. Most pressure indications come from the pump current in a remotely located ion pump, or from an equally distant gauge. The pressure in the region of the cathode is a balance between the local pumping speed and numerous gas sources such as internal outgassing, gas permeation through the O-ring seals, and gas flow along the electron optical column from the region of the specimen. In the absence of assembly leaks, and after many days of pumping, the major remaining constant gas source is generally permeation through elastomer O-ring seals. A significant variable gas source comes from the specimen chamber and can be dominated by the outgassing characteristics of the specimen itself.

What suffers most, from the poor and variable vacuum in the region of the gun, is the life (and reputation) of the LaB_6 cathode. The electron optical performance of the LaB_6 cathode is significantly affected only after the vacuum pressure rises above about 5×10^{-6} torr. However, as the pressure rises to this value, the material loss from the cathode, and hence its useful life, is dramatically affected. Experimental results show that the best indicator of the real gun vacuum is the life of the cathode itself.

2. Experimental Results

These experimental results come from the observation of cathode life in a series of wafer inspection SEM's. In one particular series of instruments, the same electron optical column is matched to several different specimen chamber sizes. In addition, instruments with similar chambers were devoted to problems with markedly different gas loads. The pumping for the cathode region on all the instruments was identical, with the vacuum pressure being indicated by the ion pump current. An isolation valve separating the electron gun and the specimen chamber was included in all instruments. Some instruments were used continuously on a 24 hour per day basis, while others were used intermittently (the gun chamber being isolated during the periods of non-use). From this wide sample of operating conditions with the same basic gun design, pertinent information regarding the life of the cathodes as a function of pressure has been obtained.

All cathodes examined were type ES-423E manufactured by Kimball Physics, Inc. The body diameter of an unused LaB_6 crystal is 320 ±10 microns. The general profile of the cathode is shown in Figure 1. The tip has a cone semi-angle of 45° and a microflat truncation diameter of 15 microns. The operating temperature of the cathode is known since calibration curves of temperature versus current were available, and the SEM's used for the tests are all equipped with reasonably accurate cathode current meters. The cathodes were nominally operated at 1850 K. Following known periods of operation, the cathodes were removed from the instruments, and the body diameters measured by optical microscopy to within ±1 micron. From the loss of body diameter, the radial loss was determined and hence the material loss in μm/hr from the free side surface of the cathode.

From this series of experiments, material losses over the range from 0.025 μm/hr to 0.35 μm/hr were observed. The results are shown in a generalized form in Table 1.

If the useful cathode life is considered to be a reduction of the body diameter to about 25% of the original diameter, the corresponding radial loss is 120 μm.

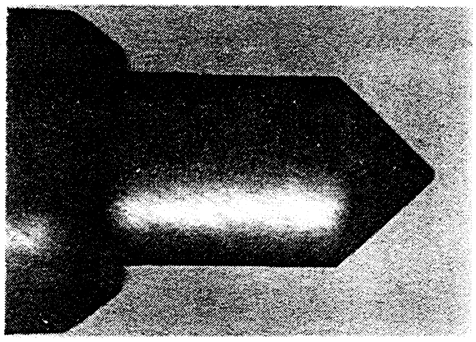

Figure 1. New cathode type ES-423E by Kimball Physics Inc. Body diameter 320 microns. Truncation 15 microns diameter.

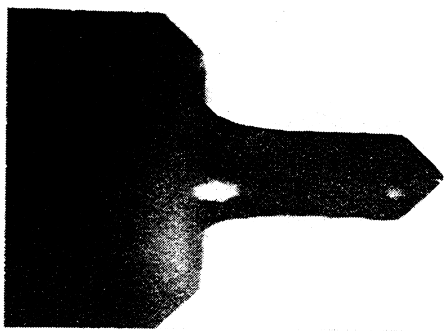

Figure 2. ES-423E Cathode, C1155, after 4500 hours at 1850 K in Bio-Rad DL-3006 SEM. Material loss at 0.025 μm/hr.

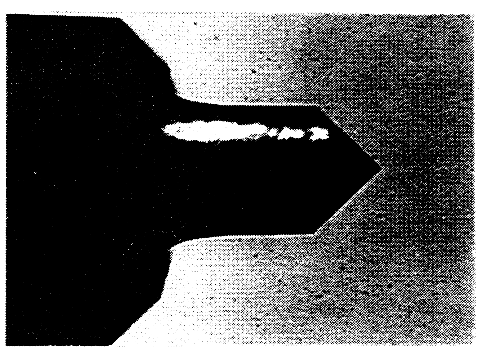

Figure 3. ES-423E Cathode, B1065, after 1105 hours at 1850 K in Bio-Rad SR-118 Wafer inspection SEM. Material loss at 0.04 μm/hr. Marked sharpening of tip.

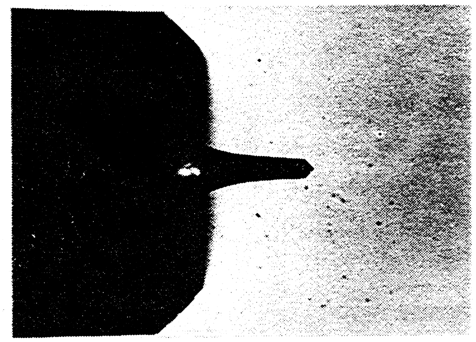

Figure 4. ES-423E Cathode, B1058, after 900 hours at 1850 K in Bio-Rad SR-118 Wafer Inspection SEM. Material loss 0.15 μm/hr.

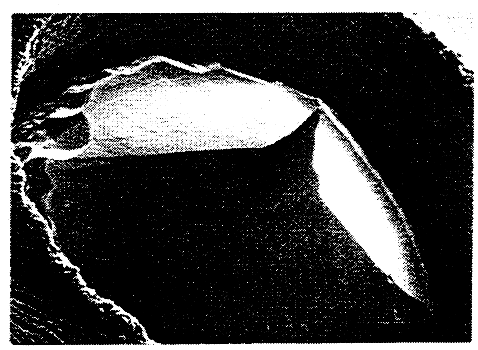

Figure 5. ES-423E Cathode after 10,000 hours in Philips EM-420 TEM at around 1750 K. Crystal almost completely gone. Heater structure unchanged.

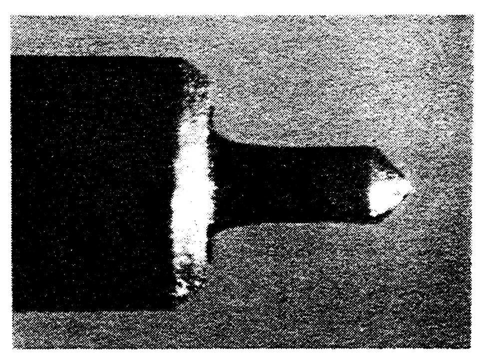

Figure 6. ES-423E Cathode, P1085, after 4750 hours continuous operation at about 1750 K in Philips EM-430 TEM/STEM. Material loss at 0.017 μm/hr.

TABLE 1

Material loss from LaB_6 Cathodes as function of Gun Pressure

Radial Loss [μm/hr]	Estimated Gun Vacuum [torr]	Vacuum Condition	Cathode Life* [hours]
0.025	$<10^7$	Excellent	4800
0.06	5×10^{-7}	Average	2000
0.2	1×10^{-6}	Poor	600
>0.3	$>2 \times 10^{-6}$	Unsuitable	<400

*For KPI ES-423 Cathode: 120 μm radial loss. Temp 1850 K.

Thus the range of useful life for the cathode has been observed to vary from about 4800 hours to only about 340 hours. The single variable causing this wide range of useful cathode life was the pressure in the cathode region, since all instruments were operated with identical electron optical parameters.

Cathodes representative of the range of material losses measured are shown in Figures 2, 3, and 4. It is generally noticed that under the best vacuum conditions the cathode remains rather blunt, retaining the general profile of the initial truncation. At higher loss rates the tip sharpens, sometimes quite rapidly. This sharpening is generally undesirable from an electron optical standpoint, since the correct operating bias for the gun becomes more difficult to establish. At very high loss rates, indicative of pressures in excess of 10^{-6} torr, the tip is often blunt; this effect may be due to ion bombardment at high pressures.

3. Discussion

The above observations are not new and have been predicted and reported by several authors.[1-4] The anticipated loss of LaB_6 due to evaporation alone at 1850 K is about 0.022 μm/hr taken from evaporation data by Storms and Mueller.[5] Table 2 is representative of results reported in the literature.

It is unfortunate that this information is frequently not appreciated by either the designers or the operators of instruments. The effort to produce improved vacuum in most commercial instruments has been marginal. Great efforts are often expended on the improvement of gun vacuums where the use of thermal or cold field emitters is anticipated. However, LaB_6 cathodes also offer increased brightness and vastly longer life than the more widely used tungsten hairpin cathodes, but the need for good vacuum with LaB_6 is still ignored. In the early days of LaB_6, lifetimes of some 500 hours were considered satisfactory. Some other commercially available LaB_6 cathodes are still limited to such lifetimes by thermal degradation of the cathode heating structure. But with increasing use of 24 hour operation, and the trend to leave cathodes on continuously to stabilize guns in both TEM's and STEM's, 500 hours represents only some 3 weeks of use. With improved gun vacuum, this 3 week period could be extended to some 6 to 7 months of continuous instrument use with the cathode at full operating temperature.

For the Kimball Physics cathodes, type ES-423E investigated here, the carbon heater assembly and the crystal position remain stable during the entire life of the cathode. Cathodes with some 10,000 hours of operation at

TABLE 2

Material Loss in μm/hr from LaB_6
(Literature Data)

	Swanson[1]		Denka[2]		Sewell[3]	
Pressure [torr]	1750 K	1850 K	1750 K	1850 K	1750 K	1850 K
$2\text{-}3 \times 10^{-6}$			0.8	1.0	1.5	1.5
1×10^{-6}	0.09	0.11			0.5	0.5
5×10^{-7}	0.026	0.056				
3×10^{-7}					0.15	0.2
1×10^{-7}	0.015	0.056	0.02	0.06	0.01	0.07
$<1 \times 10^{-8}$	0.0025	0.020				

1750 K show heater structures with no visible sign of material degradation, other than the loss of LaB_6 as shown in Figure 5. In most cases where poor performance of the cathode is reported, the culprit is poor gun vacuum.

Excellent cathode performance has also been recorded in the Philips EM-430 and EM-420 TEM's indicating excellent gun vacuum in these instruments. In high voltage instruments like these, much of the cathode life is spent at a temperature of about 1750 K. A cathode with 5740 hours of continuous operation in an EM-430 is shown in Figure 6. The loss rate is 0.017 μm/hr, still above the 0.003 μm/hr anticipated from congruent evaporation loss of LaB_6 at the given temperature. Much of the higher loss is still due to oxidation evaporation, as evidenced by the appearance of the deposit on the Wehnelt aperture.

In conclusion, ES-423E Extended Life cathodes are capable of providing up to 5000 hours of operation at 1800 to 1850 K in the high brightness mode, providing the gun vacuum is at 10^{-7} torr or better. Failure to achieve such long lifetimes is most often a result of poor gun vacuum.

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[1]P.R. Davis, G.A. Schwind, and L.W. Swanson, "Experimental study of LaB_6 in partial pressures of oxygen," J. Vac. Sci. Technol. B4(1), (1986), p. 112.

[2]Denka Kagaku Kogyo Kabushiki Kaisha, "Technical Information Bulletin No. 2," Experimental Data on LaB_6 Cathodes in Electron Microscopes (August 1, 1983).

[3]P.B. Sewell, "High Brightness Thermionic Electron Guns for Electron Microscopes," Scanning Electron Microscopy 1, (1980), pp. 11 - 24. (Calculated from oxidation model assuming unit sticking coefficient for oxygen and evaporation of La_2O_3 and B_2O_3 and congruent evaporation of LaB_6.)

[4]K.H. Loeffler, "Corrosion of Lanthanum Hexaboride Emitters", Septième Congrès International de Microscope Electronique Grenoble II, (1970), pp. 381 - 382.

[5]E. Storms and B. Mueller, J. Phys. Chem. Vol 82, (1978), p. 51.