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P.B. Sewell

OXYGEN ACTIVATION OF LAB6 CATHODES - THE DOUBLE SATURATION EFFECT

Introduction - The Symptom

In the relatively high background pressure in the gun of an electron microscope, it is possible to observe the activation of an LaB_6 cathode by residual water vapor. This effect may be noticed during the first heating of a cathode after exposure to air and also after the cathode has been heated in vacuum for any length of time at a temperature below 1600 K, at which oxides are stable and not rapidly desorbed or dissociated. This value, may be somewhat higher or lower than 1600 K, depending on the partial pressure of water vapor and the times involved.

This oxide activation leads to the appearance of a "double saturation" sequence as the cathode is brought to temperature in an auto-biased gun, and is seen while observing the cross-over image in the TEM or the beam profile image in the SEM.

As the temperature of the cathode is raised, the cross-over or emission pattern initially contracts from the large hollow beam, towards a final, near-gaussian image at saturation. As this saturation image is approached, the emission may suddenly alter and the beam again becomes extremely hollow. A further closing sequence to the final saturation is observed as the cathode temperature is further raised and finally comes to a stable saturation condition. The behavior is influenced by the rate of increase of temperature and the residual gas pressure.

If the temperature is held at the position of the first saturation image, this condition may be retained for long periods, depending on the local pressure in the gun. There is an equilibrium established between the loss of oxygen by dissociation and the replacement of oxygen by reaction with the background gas. However, very unstable behavior may result. As the pressure improves at a low fixed cathode temperature, the emission and brightness will rapidly fall to a very low level due to dissociation of the oxide. The unstable behavior is remedied by increasing the temperature of the cathode to the final saturation, where the cathode is in a clean surface condition.

The cathode temperature at which dissociation of the oxide is likely to be observed is between 1550 to 1650 K which corresponds to a cathode current of about 1.5 to 1.65 A for the Kimball Physics ES-423E LaB_6 cathode. This region of operation should be avoided in conventional electron microscopes.

Experimental Study

This oxygen activation is illustrated in an experimental study of an LaB_6 cathode in the gun of an Auger system. Here the base pressure could be reduced to the 10^{-9} torr level, and the gun brightness observed indirectly as a target current. The experiment is illustrated in Figure 1. At position A, time 0, the cathode is operating at 1850 K in UHV with a target current near 4 μA. As the temperature of the cathode is lowered, new emission plateaus are established associated with equilibrium temperatures of 1750 K, 1650 K and 1550 K, each with correspondingly lower target currents. These values fall in the ratio expected from the known thermionic emission current densities for clean LaB_6.

At a low temperature of 1500 K, the cathode is exposed to oxygen at a pressure of 2×10^{-7} torr for a period of about 50 minutes, between B and C. During this period, oxides form on the surface of the LaB_6 and are stable at this temperature and pressure. With this oxide in place, the temperature of the cathode is carefully raised to 1550 K at D. Here it is seen that the emission is similar to that for clean LaB_6 at 1650 K, and nearly 10 times greater than that for clean LaB_6 at 1550 K. As the temperature is further increased to 1610 K, the emission begins to increase rapidly to E and then falls slowly back until, at 1650 K, it is now comparable to that previously observed in UHV. The region around 1600 K is very unstable as oxides are forming, evaporating, and dissociating.

As the temperature is further increased in the oxygen background to 1750 K at F, the emission is the same as in UHV, as the oxide has totally disappeared. Here its rate of oxide formation in this low pressure of oxygen is low compared to the dissociation rate so the surface remains "clean". Again at 1850 K, at G, the emission is as observed for clean LaB_6 in UHV.

In the above sequence, the observed behavior in the region from C to F is much the same as is expected in the

high water vapor pressures in the gun of an electron microscope. The sequence is observed as the "double saturation" described above, or might be observed in terms of the total emission current or the current at the screen of the TEM or specimen of the SEM, all of which are directly related to the emission current density from the tip of the cathode.

Operating Procedure

During the turn-on of an LaB_6 cathode, care should be taken to avoid operation at the "first" saturation peak. If the first apparent saturation shows any sign of instability, it is desirable to continue to increase the temperature of the cathode. If the cathode is operating on the first peak, then the emission should suddenly drop and the beam image open up to the hollow mode. The temperature should be increased to the second saturation, which will be that of stable clean LaB_6.

For the KPI ES-423E cathode, the emission at the second saturation should be in the range of 10 to 80 μA, depending on the axial brightness required. The cathode is then operating in the range of 1700 to 1880 K. High values of bias resistor with low emission currents at saturation reflect low operating temperatures, while high values of emission and lower values of bias resistor are associated with higher temperatures. Table 1 gives an indication of the currents to be expected at saturations for various filament temperatures, for a KPI ES-423E Extended Life cathode which has a 15 μm microflat truncation on a 90° cone on the LaB_6 crystal (a style 90-15 cathode). At saturation, emission is restricted to approximately the microflat region of the cathode.

Comment

While the "double saturation" is frequently observed during the start-up of an LaB_6 cathode in the TEM or SEM, it is not always seen. The appearance of the effect depends on the condition of the cathode surface, the partial pressure of water vapor in the gun chamber, the rate of heating of the cathode and the previous idle time at a temperature below 1600 K.

When a normal single saturation sequence is observed during the start-up of the cathode, it is assumed that surface activation by oxidation has not occurred, or that the heating rate of the cathode has been sufficiently rapid that the dissociation sequence of the oxide is missed. This note is an attempt to explain the occasional observation of a "double saturation" during the start-up of auto-biased triode electron guns using LaB_6 cathodes, a sequence of events which otherwise may result in some operator confusion when first observed.

Table 1

Temperature [K]	Cathode Current* [A]	Cathode Loading [A/cm^2]	Emission Current [μA]
1700	1.75	3.8	7
1750	1.8	6.8	13
1800	1.9	10.4	20
1850	2.0	20	38

*KPI ES-423E Cathode

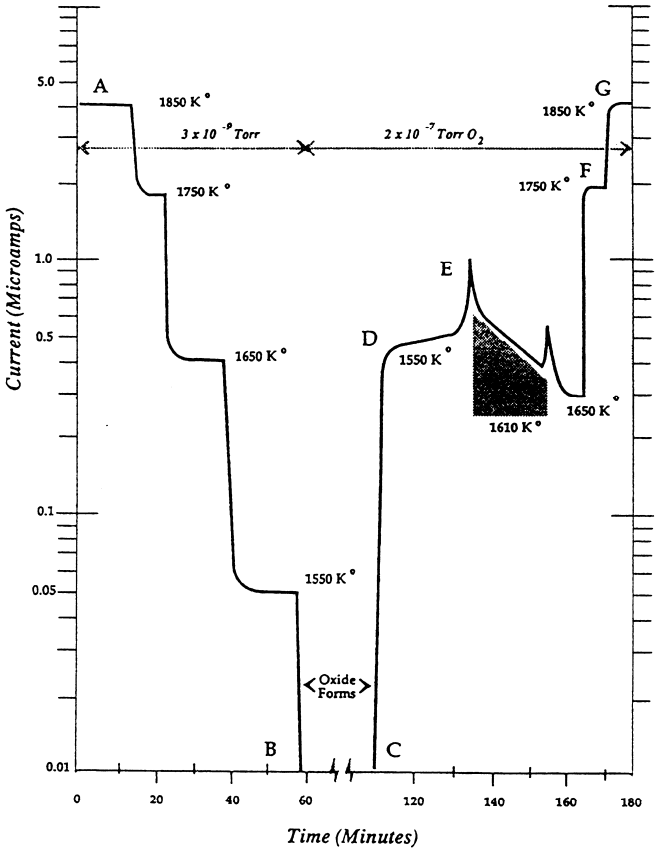

Figure 1. Measurements of emission from LaB_6 cathode in ultra high vacuum with controlled oxygen exposures. Cathode temperature from current calibrations. Clean LaB_6 in base pressure of 3 x 10^{-9} torr, between A and B. Oxygen exposure at 2 x 10^{-7} torr from B to C. Activated LaB_6 emission from C to D to E. Desorption of oxide around E and clean LaB_6 in oxygen at temperatures above 1650 K at F and G.