

# Distribution of Desorption Products on Interior Surfaces of Scandate Cathode Test Vehicle

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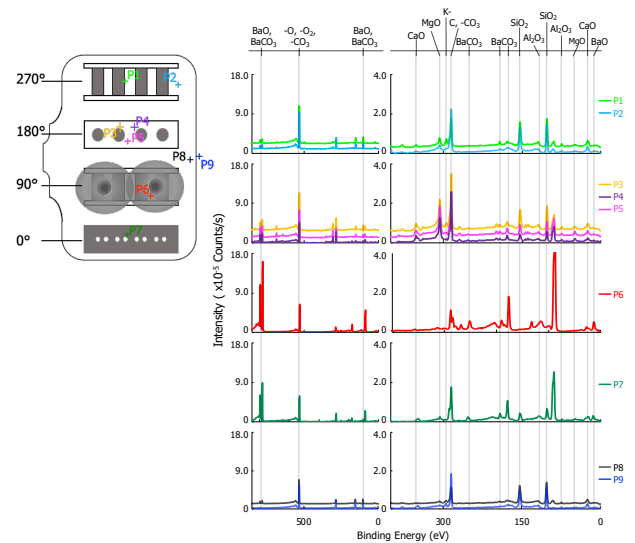
**Abstract:** Scandate cathodes have shown a higher emitted current density than any of its predecessors, which is hypothesized to stem from chemical complexes on the W matrix surface. Therefore, it is crucial to examine the cathodes' desorption behavior to understand how surface chemistry evolves during cathode lifetime. In this work, the distribution of desorption products of scandate cathode activation and operation mapped with XPS and EDX are reported. The compositional studies were conducted on the interior of the glass envelope wherein the cathodes were activated and operated, as well as the anode resting directly atop the cathodes. The results from the glass envelope indicate that Ca is expelled from the surface during pre-activation, not activation. Results from the anode yield insight into the composition and volume of material expelled throughout cathode activation and operation. Lastly, within the detection limits of XPS and EDX, no appreciable Sc was found on the anode nor the glass envelope, suggesting it is not evaporating in great amounts at any stage of cathode lifetime.

**Keywords:** scandate cathode; desorption; XPS; EDX

## Introduction

Early porous thermionic cathodes, B-type cathodes, were fabricated by impregnating a porous W body in a hydrogen atmosphere with a melt containing BaO, Al<sub>2</sub>O<sub>3</sub>, and CaO [1]. Further refinement saw the addition of Sc to the system, as either another component of the impregnate or as part of the W matrix itself; these are scandate cathodes. Scandate cathodes have shown a higher emitted current density than any of their predecessors, which is hypothesized to stem from chemical complexes on the W matrix surfaces. Therefore, it is crucial to examine the cathodes' desorption behavior to understand how surface chemistry evolves during cathode lifetime. In this work, the distribution of desorption products of cathode activation and operation are reported. Particular attention is given to Ca, since previous compositional analysis performed by Liu *et al.* [2] did not find Ca present in the cathodes post-activation. In this work, the distribution of desorption products of cathode activation and operation mapped with X-Ray Photoelectron Spectroscopy (XPS) and Energy-dispersive X-ray Spectroscopy (EDX) are reported. The compositional studies were conducted on the interior of the glass envelope wherein the cathodes were activated and operated, as well

as the anode resting directly atop the cathodes. The test vehicle was provided by eBeam, inc. and arrived to the University of Kentucky sealed; the scandate cathodes within said vehicle were fabricated, activated, and operated according to eBeam's proprietary process.



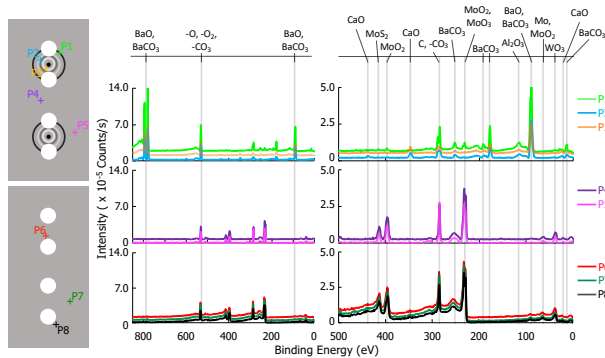
**Figure 1.** XPS results for the interior of the glass envelope as a function of position. **(left)** A diagram of the glass envelope. Each point examined is plotted (P1-P9); it is important to note that P8-P9 are controls. **(middle)** The full XPS survey of each point scan with the three tallest peaks identified. Note that there is an arbitrary vertical shift applied to P1, P4, P5, and P8 to improve visibility. **(right)** A portion (binding energies of 0-400 eV) of the full spectrum of each point, taken to improve the visibility of shorter peaks.

## Surface Analysis

### A. Glass Envelope

XPS results for the glass envelope are plotted as a function of position in Fig. 1. Using XPS analysis, strong evidence of Ca was present throughout the glass envelope, except atop the Ba flash region. It was then hypothesized that Ca is beneath the Ba flash, which was then confirmed by EDX on the TEM sample taken from near the glass substrate. This suggests a timeline wherein the Ca is expelled during pre-activation, not activation or operation. The cathode assembly is heated, Ca evaporates from the impregnate, condenses on the glass, and is then buried by the Ba flash. This suggests that not only is the Ca absent during operation, but

activation as well. This further complicates the role Ca has in improving cathode performance. It is also possible that Ca simply acts to lower the eutectic temperature of the impregnate mixture. Ba is thought to be responsible for the improved electron emission, but the Ca allows the impregnate mixture to further penetrate the cathode. There was no Sc found in any region of the glass envelope. This suggests that it is not expelling during pre-activation, nor is it emitting from the cathode during subsequent stages of fabrication/operation. The strong presence of SiO<sub>2</sub> suggests that in all regions except the Ba flash, the depositing layer is <10 nm deep.



**Figure 2.** XPS results for the anode surfaces as a function of position. **(left)** A diagram of the anode samples. The top portion of the diagram shows the anode surface facing the cathodes; the bottom portion shows the outward facing anode surface. Each point's location is plotted (P1-P9) **(middle)** The full XPS survey of each point scan with the three tallest peaks identified. Note that there is an arbitrary vertical shift applied to P1, P3, P4, P6, and P7 to improve visibility. **(right)** A portion of the full spectrum of each point, taken to improve the visibility of shorter peaks. Note the difference in axes scales compared to the charts in the middle. Labels for each relevant peak are included.

### B. Anode

XPS results as a function of position on the anode are shown in Fig. 2. Ba was found at all points except for P4 and P5. On P1, P2, and P3, the Ba likely originates from the cathode, rather than the Ba flash. It was hypothesized that Ba from the Ba flash would have a higher probability of reaching P6, P7, and P8 than P1, P2, and P3. Since the Ba peaks for P6, P7, and P8 are much smaller in magnitude than P1, P2, and P3, it follows that Ba present on the latter points was originally from the impregnate. Since the anode is known to be composed of Mo, its presence in the P1 scan suggest that any deposited layer is <10 nm deep. No Ba was discernible in the P4 and P5 results. This suggests that very little was emitted from the impregnate. A small Ba signal was measured on P6, P7, and P8. This is plausible, as this side of the anode is exposed to the glass envelope environment, wherein Ba is universally found (shown in Fig. 1).

Ca was present in P2 and P3, but not in P1. This is plausible, as P1 was furthest away from the pinhole at the center of the rings. It suggests that Ca is indeed expelled from the surface of the cathode, but not so much that a discernible concentration is present at P1. It is not currently possible to ascertain whether the Ca was expelled during pre-activation, activation, or operation. However, EDX conducted on a FIB lift-out of the anode showed distinct Ca concentrations within the deposit material. The Mo substrate is visible, topped by Ca-Ba rich layer. Another 500 - 1000 nm further from the Mo is a Ca-rich layer.

Within the detection limits of the techniques used here, no Sc was found at any point on the anode.

### Summary

A major purpose of this work was to identify how Ca moves throughout the test vehicle during cathode activation and operation. The results reported here suggest that Ca is expelled from the surface during pre-activation, not activation. XPS results from the Ba flash region do not find evidence of Ca, while EDX on a TEM lift out sample found Ca *beneath* the Ba layer. Since the Ba flash occurs prior to activation, it follows that species emitted during activation and operation would collect on, not beneath, the surface of the Ba flash, and thus be discernible with XPS. XPS studies of the anode yielded insight into the composition and amount of material expelled from the cathode throughout the process. Near the pinhole directly opposite the cathode, no Mo is detected, suggesting that the deposit is >10 nm. However, between 1.5-2 mm away from the pinhole, Mo is detected, indicating the amount of Ba and Ca reaching that distance is minute. Lastly, within the detection limits of XPS and EDX, no appreciable Sc was found at any points examined, on the anode or the glass envelope. This suggests that it is not evaporating in great amounts at any stage of the process.

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The views, opinions, and/or findings expressed are those of the author(s) and should not be interpreted as representing the official views or policies of the Department of Defense or the U.S. Government.

### References

- [1] R. Levi, *Journal of Applied Physics*, vol. 24, p.3., 1953
- [2] X. Liu *et al.*, *Materials Characterization*, vol. 148, pp. 188-200, 2019.