Characterization of Material Phases on the Surface and in the Near-Surface Region of Scandate Cathodes

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Abstract: *The impressive electron emission capabilities of scandate cathodes continue to receive significant research attention, although there remain gaps in understanding the mechanistic reasons for their performance. This is partly due to lingering questions about the materials and microstructure of the cathode emitting surface. In the current study, scandate cathodes fabricated using related but distinct processes were emission-tested and then characterized using advanced electron microscopy and analytical spectroscopy techniques. The cathode surfaces were consistently observed to consist of faceted tungsten grains decorated with ~100 nm oxide ($BaAl_2O_4$ and Sc_2O_3) particles and ~20 nm Ba-containing particles.*

Keywords: scandate cathode; surface; characterization; electron microscopy; spectroscopy.

Introduction

Due to their intriguing electron emission performance and promises of lower operating temperatures and/or extended lifetimes, scandate cathodes have long been viewed as next-generation vacuum electron devices. Scandate cathodes offer the potential for significant improvement over the currently dominant M-type cathode [1-5]. In order to more effectively implement scandate cathodes in functional devices, it is necessary to reliably model their behavior. In turn, it is also necessary to understand what materials exist on the emitting surface, so that their combined emission performance can be modeled and so that scandate cathodes can be consistently fabricated.

Methods

Cathode surfaces were imaged using scanning electron microscopy (SEM; FEI/Thermo Helios NanoLab 660) in both secondary electron (SE) and backscattered electron (BSE) image modes. X-ray energy dispersive spectroscopy (EDS) was performed in the SEM, in order to map the distribution of elements on cathode surfaces. The focused ion beam (FIB) capabilities of the Helios NanoLab 660 were utilized to extract an electron-transparent lamella from the cathode surface, for additional imaging and EDS mapping in the transmission electron microscope (TEM; FEI/Thermo Talos F200X G2).

Results and Discussion

The surfaces of scandate cathodes consistently exhibited faceted tungsten grains, as shown in Figure 1. As described in a separate publication [5], these crystallographic facets are primarily {112}, along with diamond-shaped {110} and square or rectangular {100}. These facets are apparent in the SE images (left side of Figure 1). The BSE images (right side of Figure 1) reveal ~100 nm diameter nanoparticles with two distinct gray levels ($BaAl_2O_4$ is light gray, Sc_2O_3 is dark gray). The identity of these oxides was confirmed using EDS. As seen in the lower left SE image of Figure 1, ultrafine nanoparticles (~20 nm diameter) also decorate the tungsten facets; these nanoparticles contain barium and are believed to be BaO.

In order to characterize the near-surface region of the scandate cathodes, cross-section samples were extracted from a cathode using FIB. This lamella is shown in Figure 2, which presents an SEM image along with EDS elemental maps showing the distribution of all relevant elements. The original scandate cathode surface is at the top of each image in Figure 2. Note that the gallium (Ga) signal is an artefact of the FIB milling process, which utilizes a Ga beam for material removal.

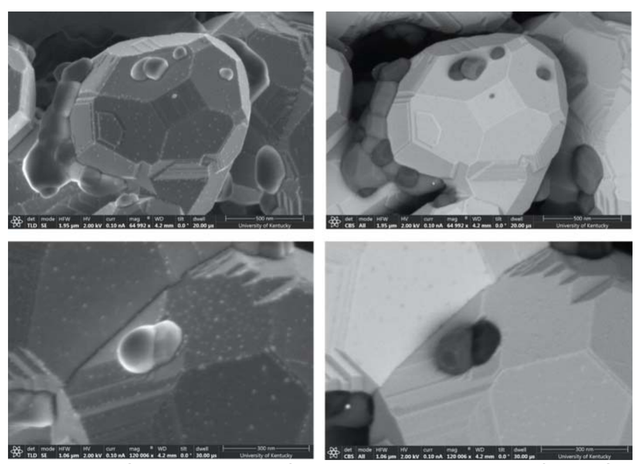

Figure 1. SEM images of scandate cathode surface. SE images are on the left, with BSE images showing atomic number contrast on the right. In the lower-right image, different oxide particles are visible: light gray particles are $BaAl_2O_4$ and darker gray particles are Sc_2O_3. In the high-magnification image at bottom left, ultrafine white particles are visible.

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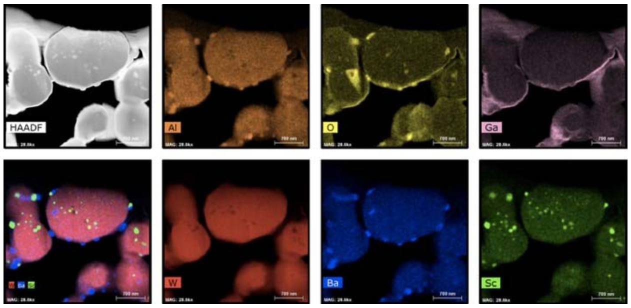

Figure 2. SEM image and EDS elemental maps for a cross-section TEM lamella extracted from a scandate cathode surface. The composite EDS map at lower left shows the distribution of oxide nanoparticles.

In Figure 2, the elements contained in the impregnate material are seen to be distributed heterogeneously amongst the porous tungsten matrix. As shown in the composite elemental map at lower left, $BaAl_2O_4$ particles (blue color) and Sc_2O_3 particles (green color) decorate the tungsten grains, which is consistent with the observations made during SEM imaging (Figure 1). Note that these larger nanoparticles are distinct, i.e. they are not mixed Ba-Sc-Al-oxides.

Further characterization of the larger oxide nanoparticles was performed using electron backscatter diffraction (EBSD) in the Helios NanoLab. This technique utilizes electron diffraction patterns to determine crystallographic orientation and can be used to verify the crystal structure of a material at high spatial resolution (~50 nm). An EBSD scan of a cathode cross-section is shown in Figure 3. A cluster of larger (~100 nm) surface oxide nanoparticles are seen near the middle of the SEM image (left side of Figure 3), along with a phase map (right side of Figure 3) showing the material phases as determined by EBSD. The prevalence of red grains is consistent with tungsten grains occupying most of the sample volume. The oxide nanoparticles are colored blue in the phase map and were confirmed to be $BaAl_2O_4$ through their crystal structure and composition. Note that the Ba_2ScAlO_5 phase was specifically sought in this study, as that phase is predicted to exist in scandate cathodes. However, it was not found in Figure 3 nor in other regions characterized with EBSD.

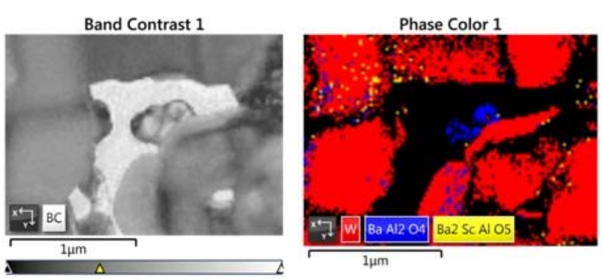

Figure 3. SEM image and EBSD phase map of a TEM cross-section specimen, showing the near-surface region of a scandate cathode.

Summary

Detailed characterization was performed on scandate cathodes, using a combination of electron microscopy and analytical spectroscopy to determine the material phases that exist on the cathode surface as well as in the near-surface region. The oxide nanoparticles decorating the faceted tungsten grains were identified. Importantly, the distribution of elements originating from the impregnate (Ba and Al) was found to be heterogeneous, as was the Sc that was part of the original pellet matrix. Additionally, certain phases that some researchers expect to exist (Ba_2ScAlO_5) were not found in the cathode surface region. The material phases that occupy the emitting surface region of scandate cathodes are complicated, and work is ongoing to determine their detailed structure and distribution.

Acknowledgment

This work was financially supported by the Defense Advanced Research Projects Agency (DARPA) Innovative Vacuum Electronics Science and Technology (INVEST) program, under grant number N66001-16-1-4041.

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.

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