Title: Characterization of Osmium-Ruthenium Thin Films for Cathode Coatings

CHARACTERIZATION OF OSMIUM-RUTHENIUM THIN FILMS FOR CATHODE COATINGS

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Abstract

Osmium-Ruthenium (OsRu) alloy thin film coatings were characterized to understand the effects of film structure on thermionic emission from dispenser cathodes. Electron microscopy and x-ray diffraction of coated dispenser cathodes were used to characterize the various film structures, and life testing was conducted to gauge the improvement in cathode emission due to each film structure. In collaboration with industrial partner Semicon Associates, three novel film structures were studied in addition to the standard Semicon film. Different film architectures were created, with varying levels of substrate bias during film deposition. The films were simultaneously magnetron sputtered on porous tungsten pellets for characterization and on cathode assemblies for life testing. The cathode assemblies were life tested for 1000 hours. Cathode performance will be discussed in light of the microstructural features of each film.

Introduction

Dispenser cathodes make use of thermionic emission to produce a stream of electrons. The basic design consists of a tungsten pellet, a barium source (either a reservoir or a compound impregnated within a porous tungsten pellet), and a heating element. These cathodes have applications in travelling wave tubes, klystrons, magnetrons, and other various vacuum devices. Dispenser cathodes were developed in the 1940’s and made use of a reservoir of barium (Ba) that was dispensed to the cathode surface gradually over time [1]. Later, the reservoir of Ba was replaced by a Ba compound impregnated within a porous tungsten pellet. The natures of the devices that utilize dispenser cathodes require a long life and high reliability. One of the main lifetime limiting factors of dispenser cathodes is thought to be depletion Ba [2].

In the 1960’s, the so-called “M-Type” cathodes was developed by Philips [3]. An M-Type cathode is essentially the same as an impregnated dispenser cathode but in addition a coating of an osmium-ruthenium (Os-Ru) alloy is applied to the tungsten pellet. The Os-Ru coating acts to lower the work function of the cathode and thereby reduce the operating temperature which prolongs cathode life over non-coated cathodes [4, 5]. In M-Type dispenser cathodes, the lifetime are not typically limited by Ba depletion, but rather by excessive interdiffusion of tungsten (W) with the Os-Ru coating [6]. Furthermore, the interdiffusion of W-Os-Ru is accelerated at high temperatures which are experienced during operation. Minimizing the interdiffusion of W-Os-Ru is a key aspect in prolonging the life of M-Type cathodes.

To minimize W-Os-Ru interdiffusion, modifications to the microstructure of the Os-Ru thin film coating were made. Varying certain processing parameters during deposition of the Os-Ru film can induce changes in the microstructure of the film. Previous studies [7, 8] identified a few film architectures which showed potential for inhibiting excessive W-Os-Ru interdiffusion. These films were used in life tests of cathode assemblies and compared against a standard commercial coating used by industrial collaborator Semicon Associates.

Experimental Methods

Os-Ru coatings were deposited simultaneously onto porous tungsten pellets and full cathode assemblies which were used for characterization and life testing, respectively. Three novel films were deposited via DC magnetron sputtering in an AJA ORION sputtering system with a base pressure of less than 1.0×10^{-7} torr. A sputter-up configuration was used with substrate rotation to ensure even film thickness. Substrate bias was applied to create different microstructures. The Semicon Associates films were prepared by RF magnetron sputtering with no substrate bias in a sputter-down configuration using an Omega sputtering system. Table I summarizes the films and their associated processing parameters.

Table I. The Os-Ru films and their associated deposition parameters are listed. Also listed, are the reasons why each film was chosen for the study.

Film	Thickness [nm]	Substrate Bias [W]	Deposition Rate [nm/min]	Reason
10W-150nm	150	10	16.7	• Similar texture to Semicon films
5W-550nm	550	5	16.7	• Similar to Semicon film thickness • Basal texture {0002} • Excellent compositional stability
Multilayer	Layer 1: 25 Layer 2: 150 Layer 3: 25	Layer 1: 20 Layer 2: 5 Layer 3: 0	16.7	• Layer 1: coarse grains, good adhesion • Layer 2: basal texture • Layer 3: fine grains, improved emission
Semicon	550nm	0	10	• For comparison

After deposition, the cathode pellets were characterized via x-ray diffraction (XRD) and scanning electron microscopy (SEM), including cross sectional and plan-view imaging. The cathode assemblies were sent to a testing agency where they were life tested in a closed-space diode test for 1,000 hours. The closed-space diode test operates the cathodes at 1100 $°C_B$ and applies $5mA/cm^2$ to the cathodes. At various times, the cathodes are tested in pulse-duty and the currents at different temperatures are recorded.

Results and Discussion

The Os-Ru films exhibited microstructures consistent with those from previous works [5, 6]. The grains in the films were columnar through the thickness and uniformly covered the pellet surface. Figure 1 shows SEM images of the films both in plan- and cross-sectional views. The closed-space diode test revealed that the 10W-150nm film yielded a very stable cathode over 1,000 hour test. The multilayer film showed improved performance with time over the course of the test and the 5W-550nm film showed little improvement over the course of the test. Figure 2 shows current versus temperature plots of the cathodes at different points in the life test.

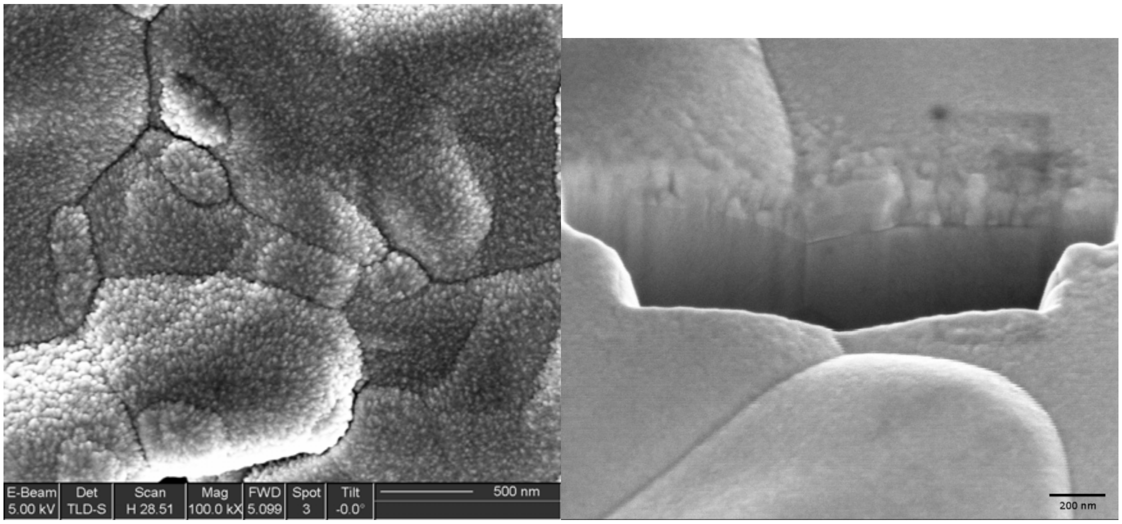

Figure 1. SEM images of the 5W-550nm film in plan-view (left) and 10W-150nm film cross-section (right). Notice the fine grain structure of the Os-Ru film superimposed on the larger grain structure of the tungsten substrate.

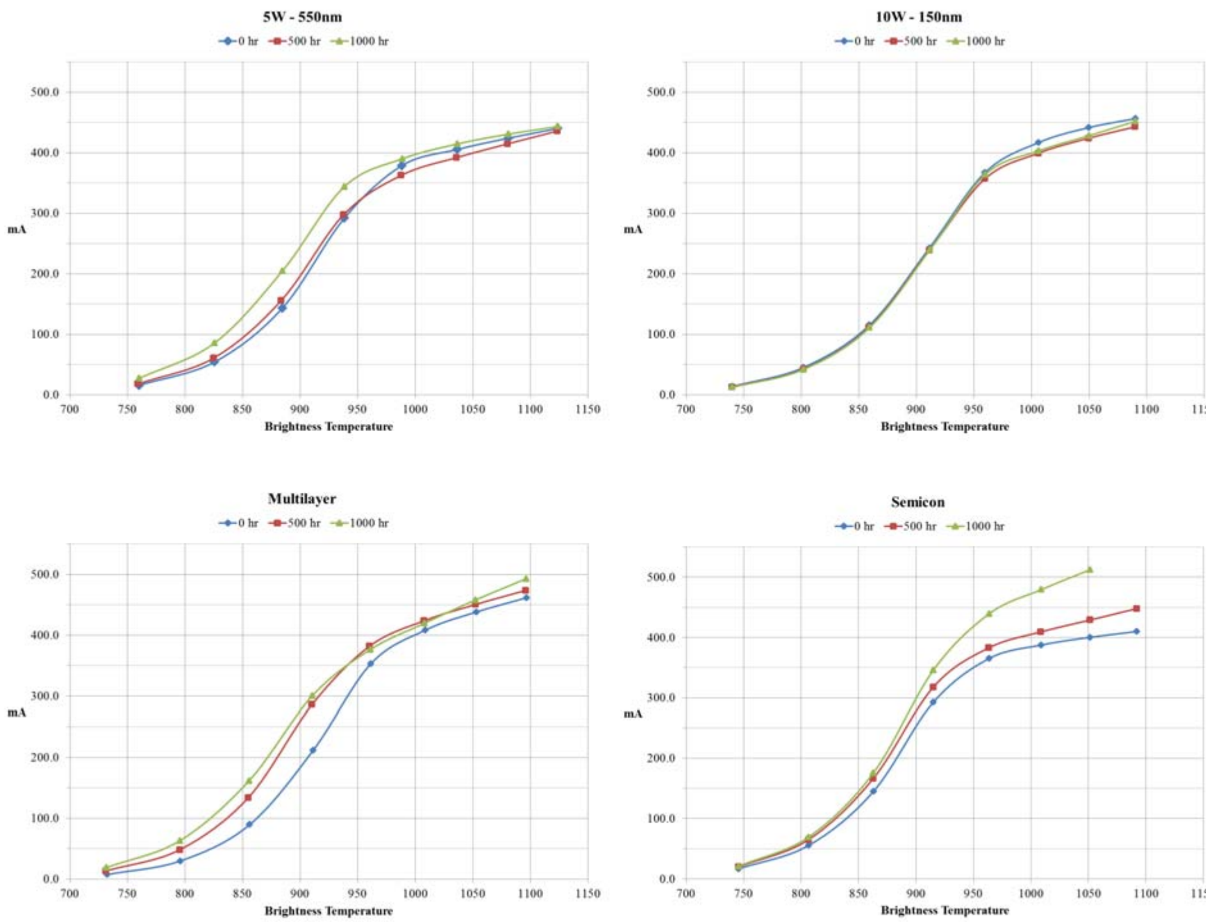

Figure 2. Current versus temperature plots for the different Os-Ru films during life testing. High current at low temperature is ideal.

The knee temperature is an indicator of cathode performance and defines the cross over point between temperature limited and space charge limited emission. The knee temperature can be used as a way to rank performance with the lowest knee temperature as the best. Table II lists the knee temperatures for all four cathodes in the life test. From the knee temperatures, it was clear that the Semicon cathode had the lowest activation (0 hr) knee temperature and maintained a low knee temperature throughout the test. All three of the novel films exhibited high activation knee temperatures but the multilayer film improved greatly and in the end rivaled the performance of the Semicon cathode. The 10W-150nm film stood out for its stability as the knee temperature changed by only 10 degrees after 1,000 hours of testing which also rivaled the performance of the Semicon cathode.

Low knee temperatures have implications for improving the lifetime of cathodes because the cathodes can operate at a lower temperature. Typically, M-Type dispenser cathodes are operated just above the knee temperature. Operating at a lower temperature may slow the W-Os-Ru interdiffusion process as well as the rate of Ba depletion.

Table II. Knee Temperatures for the Os-Ru films at different points during the 1,000 hour test.

Cathode	T_{knee} [°C]		
	0 hr	500 hr	1000 hr
5W - 550nm	969	961	952
Multilayer	976	946	934
10W - 150nm	975	969	965
Semicon	940	930	940

Conclusion

The ability to control and change Os-Ru film microstructures to achieve different cathode performance allows for the opportunity to optimize the coating for the best chance at increasing the life of M-Type dispenser cathodes. This research revealed a multilayered film microstructure that shows promise for increasing the life and prompts further investigation into film microstructures and electron emission mechanisms.

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