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# Characterization of scandia doped pressed cathode fabricated by spray drying method

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#### ABSTRACT

Scandia doped pressed cathode was prepared by a new method of spray drying combined with twostep hydrogen reduction process. The Sc<sub>2</sub>O<sub>3</sub> and barium–calcium aluminate co-doped powders have sub–micrometer size in the range of 0.1–1  $\mu$ m and scandium oxide and barium–calcium aluminate are distributed evenly in the powders. The cathodes sintered by powder metallurgy at 1600 °C<sub>b</sub> have a smooth surface and sub–micrometer grain structure with homogeneous distribution of scandium, barium, calcium and aluminum which are dispersed over and among the tungsten grains. This cathode has good emission, e.g., the current density of this cathode reaches 31.50 A/cm<sup>2</sup> at 850 °C<sub>b</sub>. After proper activation, the cathode surface is covered by a Ba–Sc–O active substances layer with a preferable atomic ratio, leading to its good emission property. The evaporation activation energy of SDP cathode with 4.58 eV is the highest among the Ba–W, M-type and SDP cathodes, and the average evaporation velocity  $v_t$  of SDP cathode with 1.28 × 10<sup>-8</sup> g cm<sup>-2</sup> s<sup>-1</sup> at 1150 °C<sub>b</sub> is the lowest one.

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#### 1. Introduction

Vacuum electron devices have a wide application in civil and military fields, such as communication, broadcastings and aviation industry [1–3]. An increase of current density at a given working temperature is a main trend in the development of thermionic cathodes for the application of high power vacuum devices. Among all the electron emitters, scandate cathodes have aroused great attention among all the thermionic cathodes due to their high current density [4–9].

In the past, the research on the scandate cathode mainly focused on the preparation of scandate impregnated cathode. During this preparation, a series of complicated procedures, including powder manufacturing, pressing, sintering, impregnating and water cleaning, should be taken to obtain the impregnated cathode. If the powder containing  $Sc_2O_3$ , impregnants (Ba, Ca aluminate) and tungsten was used for the preparation of scandate cathodes, the pressed cathode could be obtained by pressing and sintering. However, previous pressed cathodes fabricated by mechanical mixing have been characterized with a low-emission current density and non-uniform emission distribution due to the non-uniform distribution of scandium and barium–calcium aluminate [10]. To improve emission density and emission uniformity, it is desirable to find a novel method to fabricate the scandia doped pressed cathodes.

Scandia doped pressed (SDP) cathodes have been prepared by a new method of spray drying combined with two-step hydrogen reduction in this work. The microstructure, evaporation behavior and electron emission properties of the SDP cathodes have been studied in this paper.

#### 2. Experimental

Sc<sub>2</sub>O<sub>3</sub> and barium-calcium aluminate co-doped tungsten powders were prepared by a spray drying method combined with a two-step hydrogen reduction process. First, the precursor powders were prepared by spray drying (EYELA Spray Dryer SD-1000), calcined at 600 °C for 2 h in air to remove organic salt components, then the obtained scandium and barium-calcium aluminate co-doped tungsten oxide powders were reduced in scandia and barium-calcium aluminate co-doped tungsten metallic powders by hydrogen at 850 °C for 1 h in tube reduction furnace and cooled to room temperature with the furnace in the dry hydrogen atmosphere. The co-doped tungsten powders were pressed and sintered  $(1600 \circ C_{\rm h}, 2 \min)$  into a pellet of about 3 mm in diameter and 1 mm in thickness. Morphology, particle size of the powders and the microstructure of the cathodes were characterized by scanning electron microscopy (SEM Hitachi S-3400 Japan) with energy dispersive spectrometer (EDS). The particle size distribution was analyzed with a laser diffraction particle size analyzer (Malvern Nanosizer UK) with ethanol as the dispersant. "In situ" Auger

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**Fig. 1.** SEM images of (a) pure tungsten oxide powders, (b) scandia and barium–calcium aluminate co-doped tungsten oxide powders, (c) pure tungsten powders prepared with powder (a) through reduction at 850 °C for 1 h, and (d) scandia and barium–calcium aluminate co-doped tungsten powders prepared with powder (b) through reduction at 850 °C for 1 h, (e), (f) and (g) corresponding EDS results of powder (d).

electron spectroscopy (AES) analysis was carried with VG MICRO-LAB MK-II and PHI 550 systems. Electron emission properties of the cathodes were tested in a close-spaced diode configuration in ultrahigh vacuum system with a Mo-anode by a computer-controlled automatic emission-testing device. The average evaporation rate of cathodes was measured by quartz crystal microbalance. The cathode temperatures were measured by KELLER (PB 06/M BG01) infrared thermometer and calibrated into brightness temperatures.



Fig. 2. Size distribution curves of (a) pure tungsten powders, (b) scandia and barium-calcium aluminate co-doped tungsten powders.

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