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Activation of binary Zr–V non-evaporable getters: synchrotron radiation photoemission study

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Abstract

Zr–V alloy getter films were prepared on stainless steel substrates by magnetron sputtering. The thermal activation behavior of these getters was investigated by synchrotron radiation photoelectron spectroscopy using photon excitation energies of 600, 250 and 73 eV. Depth resolved results were compared to the results of the SIMS profiling. The measurements confirmed the disappearance of the superficial oxide layer covering the air-exposed Zr–V surfaces via its progressive reduction during the thermal activation. The depth sensitive results showed that the activated getter surface is covered by a residual zirconium sub-oxide.

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1. Introduction

Non-evaporable getters (NEGs) permit the achievement of ultra-high vacuum conditions with pressure of the order 10^{-11} mbar and lower. They can be used for efficient pumping of low-aperture and sealed-off vacuum devices [1–3] or for construction of getter pumps as a complement of conventional pumping equipment [4–6]. An especially important application of NEGs is reduction of ultimate pressures in collider facilities [1,7–10]. The pumping properties of NEGs are determined largely by their surface characteristics since gettering is essentially a surface chemical reaction.

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Zr–V binary alloys have been found to exhibit an activation temperature below 200 °C [10,11], and lowering of the activation temperature is an important way to extend the range of NEGs' practical use. Recently, several studies have been devoted to the characterization of Zr–V based getters by means of surface sensitive techniques, such as Auger electron spectroscopy (AES) [12,13], X-ray photoelectron spectroscopy (XPS) [14–16] and static secondary ion mass spectrometry (SSIMS) [17]. XPS investigations showed that oxide reduction proceeds through formation of sub-oxides with a simultaneous formation of carbides [16].

In this paper, we report a synchrotron radiation photoelectron spectroscopy (SRPES) study of the activation process of Zr–V NEG thin films, in which we use the tunability of the photon energy to vary the surface sensitivity.

2. Experiment

The Zr–V getter films studied in this work were deposited by the DC magnetron sputtering technique on stainless steel substrates (10 mm × 10 mm × 1 mm) in an argon atmosphere, using the deposition system previously described in [16].

The SRPES experiments were performed at the Materials Science Beamline at the Elettra synchrotron light source in Trieste. It is a bending magnet beamline with a tuning range from 40 to 800 eV equipped with a plane grating monochromator based on the SX700 concept [18]. The UHV experimental chamber with a base pressure of 1×10^{-10} mbar is equipped with a 150 mm mean radius electron energy analyser Phoibos 150 made by Specs with a multichannel detection. The analyser was used in a constant energy resolution mode and high point transmission lens mode with an acceptance area of 2–3 mm^2 and an acceptance angle of 8°. Core-level and valence band spectra were recorded at an emission angle of 30° from the surface normal and 30° incidence of photons. For the SRPES experiments we used primary photon energies of 600, 250 and 73 eV. The 600 and 250 eV photon energies were chosen in order to maximize surface sensitivity for the V 2p and Zr 3d core level photoelectron peaks. The energy of 73 eV corresponded to the Cooper minimum for Zr 4d. If the beam energy is tuned to the Zr 4d Cooper minimum the V 3d/Zr 4d photoionization cross-section (CS) ratio is about 60. On the other hand, for 250 eV the Zr 4d, V 3d and O 2s CS are of similar values. The binding energies and corresponding CS of core and valence band levels used in this study are summarized in Table 1. The CS data are taken from [19].

Table 1
Binding energies and photoionization cross-sections of the measured peaks

Level	BE	CS (73 eV)	CS (250 eV)	CS (600 eV)
Zr 4d	0.2	0.05	0.07	
V 3d	0.2	3	0.17	
O 2p	7	2.5	0.09	
C 2s	18	0.6	0.06	
O 2s	23	0.75	0.12	
Zr 4p	28	0.62	0.24	
V 3p	37	0.8	0.63	
Zr 4s	50	0.27	0.09	
Zr 3d	180	–	4.6	1.26
V 2p	512	–	–	1.41
O 1s	530	–	–	0.41

The samples were heated using a resistively heated Ta wire mounted in the sample holder. The pressure during the experiments did not exceed 8×10^{-10} mbar at the beginning of the activation and the typical value was about 4×10^{-10} mbar.

The reference SIMS experiments were performed in an UHV chamber equipped with a Perkin-Elmer spectrometer PHI 06-600 [17].

3. Results and discussion

Several samples of the same composition of $Zr_{44}V_{56}$ were deposited simultaneously by magnetron sputtering, and the sample composition was determined ex situ by XPS in the same way as described in [16]. Three samples were investigated by SRPES at different excitation energies, one sample was used for the reference SIMS measurement.

The air-exposed Zr–V getters were degassed first at 120 °C for 4 h. Then a thermal activation process was performed in five consecutive heating steps at 160, 200, 240, 280 and 320 °C. For each step the sample was kept at the indicated temperature for 2 h and SRPES spectra were recorded at this temperature.