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Plasma Cleaning Applications for Surface Science and Model Catalyst Samples

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We used the Evactron plasma cleaning system, a device manufactured by XEI Scientific, Inc. (www.Evactron.com), for three applications in catalysis and surface science, described below. The Evactron produces low power RF O_2 plasma that oxidizes and removes hydrocarbon contamination. This type of contamination can be found on the surface of any sample transported in air and the contamination greatly hampers characterization of the sample by surface sensitive techniques such as X-ray Photoelectron Spectroscopy (XPS). Removal of carbon-like impurities without sample damage can be a key issue on some studies.

I. Catalytic application: Industrial catalysts are often complex systems in which the active phase is buried within a porous support. Fundamental surface studies of these catalysts can be difficult, if not impossible. Surface sensitive techniques provide limited information in this case because the catalytically active particles are buried inside the pores of the catalyst. To avoid this problem, a model catalyst is made by depositing the component on the surface of a flat, non-porous substrate. This sample can then be easily accessed by surface sensitive tools. One of the difficulties in working with a model catalyst is cleaning its surface without disturbing the subnanometer-sized catalytic structures and the flat support.

Platinum clusters with 7-10 atoms prepared at Argonne National Laboratory were deposited on an alumina film over a Si wafer and then were transported in air into our UHV system. Oxygen plasma oxidation created by the Evactron was used to rid the surface of carbon contamination. The amount of carbon decreased by 5-6 times after the catalyst was cleaned using the Evactron RF O_2 plasma. However, the treatment oxidized the Pt such that the Pt 4f peak shifted beneath the Al 2p peak, as shown in Fig. 1. Oxidized Pt can be reduced back to the metallic state in a H_2 atmosphere.

II. Surface Science application (I): In order to mimic a gold surface subjected to rigorous cleaning treatments with piranha solution, we have treated a gold film in O_2 plasma [1]. Oxygen plasma removed the carbon contamination and produced a metastable oxide layer, which is sufficiently long-lived under vacuum conditions to permit its characterization by XPS (see Fig. 2). This data was used to identify the assembling mechanism of dithiocarbamate-anchored monolayers on Au substrates in aqueous solutions [1].

III. Surface Science application (II): The wide-band gap semiconductor zinc oxide (ZnO) is a material with much potential for future electronic devices. Despite significant interest in its fundamental electronic properties, there are few spectroscopic studies of the ZnO surface. The surface properties of ZnO are dependent on the amount of surface contamination. In this case, the commonly used methods of Ar^+ sputtering and annealing should be avoided to prevent damage to the sample. Oxygen plasma cleaning could clean the surface without damaging it.

References

[1] H. Zhu, D.M. Coleman, C.J. Dehen, I.M. Geisler, D. Zemlyanov, J. Chmielewski, G.J. Simpson, and A.Wei, *Langmuir*, 24 (2008) 8660.
[2] We thank XEI Scientific for the loan of the Evactron plasma cleaning system and Dr. Stefan Vajda of Argonne National Laboratory for preparing the Pt clusters.

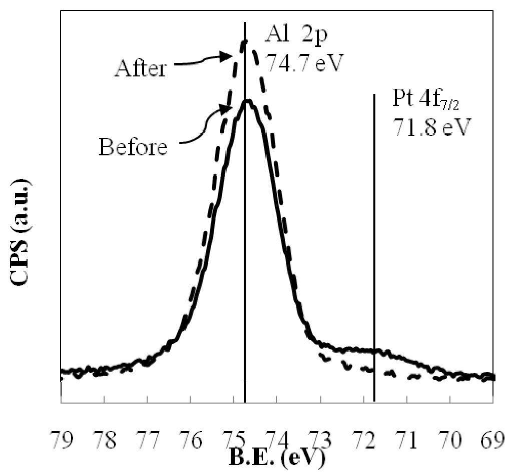

FIG. 1. Al 2p/Pd4f core level spectra obtained from a $Pt/Al_2O_3/SiO_x$ catalyst before and after treatment with Evactron oxygen plasma. After the plasma treatment the Pt $4f_{7/2}$ shifts to higher BE because it oxidizes and can no longer be resolved from the Al 2p peak.

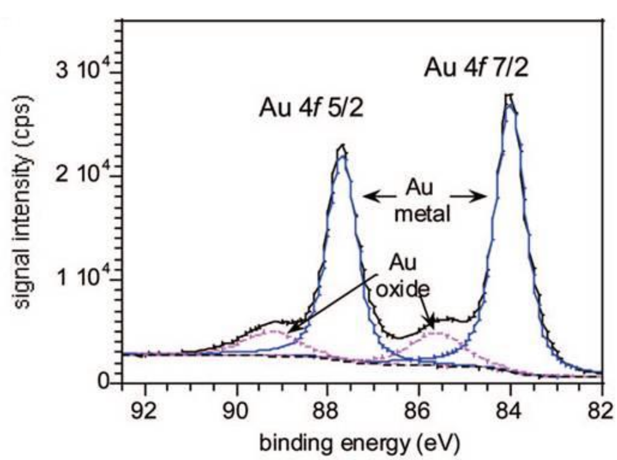

FIG. 2. The Au 4f core level obtained at a 60° photoemission angle from Au substrate cleaned by the Evactron plasma. Deconvolution of the Au 4f peaks (solid black) enabled peak assignments to Au metal (solid blue) and Au-oxide (dashed purple) after subtraction of the background signal (dashed black).

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