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Meeting Report

2nd International Workshop on Ambient Pressure X-ray Photoelectron Spectroscopy

The 2nd International Workshop on Ambient Pressure X-ray Photoelectron Spectroscopy (APXPS) was held at Lawrence Berkeley National Laboratory (LBNL) in Berkeley, CA, from December 7–9, 2015. It brought together more than a hundred participants from 17 countries. The workshop followed the inaugural meeting at the French synchrotron SOLEIL in December 2014, which was organized by François Rochet. The strong interest in these workshops reflects the growth of the APXPS community over the last decade, with instruments now operational at more than 12 synchrotrons around the world (see SRN, Vol. 27, No. 2, pp. 14–23) and a steady increase in the number of laboratory instruments. APXPS has established itself as an important method for the investigation of surfaces and interfaces under *in situ* and *operando* conditions, including liquid/vapor and liquid/solid interfaces.

This workshop featured 3 plenary lectures, 7 invited and 15 contributed talks, as well as a poster session with more than 30 contributions. The meeting opened with two plenary lectures by Robert Schlögl (Fritz Haber Institute, Berlin) and Miquel Salmeron (LBNL). Robert Schlögl's talk described the application of APXPS to studies of real catalysts, in particular those used in the Haber-Bosch process and the opportunities for using hydrogen from renewable sources in this reaction, which would reduce global carbon dioxide emissions by about 3%. Miquel Salmeron's plenary lecture took the prize for the most original title: "Chemistry and Surface Science in the 21st Century: Warm, Dense and Wet." In it, he described APXPS experiments on the adsorption of carbon monoxide on well-defined single-crystal metal surfaces, in particular, the mechanisms that lead to the restructuring of the surface in the presence of CO. Both plenary speakers showed the benefits of a combination of APXPS with other *in situ* techniques, such as electron microscopy and scanning tunneling microscopy. The third plenary talk was given at the beginning of the second day by David Prendergast (LBNL), who presented first-principles calculations of electrochemical systems and compared them to experimental results from X-ray absorption and photoemission experiments, showcasing the benefits that a close collaboration between experimentalists and theoreticians has for a deeper understanding of fundamental processes at interfaces.

The invited and contributed talks were grouped according to their scientific scope. One traditionally strong area of APXPS research is heterogeneous catalysis, where the relation between the surface composition of the catalyst and its activity are of particular interest. Edvin Lundgren (invited talk; Lund University) reported on the application of laser-induced fluorescence, combined with APXPS and multiscale modeling, to investigate mass flow limitations and their potential implications for carbon monoxide oxidation reactions studied in a typical APXPS reactor. Mark Greiner (FHI Berlin), Benjamin Reinecke (Lund University) and Ryo Toyoshima (Keio University) presented results of investigations on ethylene epoxidation on AgCu alloys, V_2O/TiO_2 selective reduction catalysts and carbon monoxide oxidation on PdAu alloy surfaces, respectively. The last session on the opening day of the workshop was dedicated to new experimental approaches in APXPS. Andrei Kolmakov (invited; NIST Gaithersburg) described advances using ultrathin, X-ray- and electron-transparent membranes for the investigation of heterogeneous interfaces, where the electrons from the interfacial region are collected through the membrane film. This is an exciting

new direction in particular for the investigation of liquid/solid interfaces and is under development in several laboratories. Luca Gregoratti (Elettra, Trieste) provided an update on advances in the application of scanning photoemission microscopy for heterogeneous reactions, an important direction for APXPS due to the inherent spatial heterogeneity of realistic systems under study in APXPS. A pathway towards increased spatial resolution across heterogeneous interfaces was presented by Osman Karslioglu (LBNL), who discussed the use of standing-wave XPS for subnanometer resolution in the determination of the chemical and electrical potentials across interfaces.

The second day of the meeting focused on the application of APXPS to electrochemistry and electrocatalysis. Talks by Bryan Eichhorn (invited; University of Maryland), Kelsey Stoerzinger (invited; MIT), Andreas Nenning (TU Wien) and Zixuan Guan (Stanford) addressed the complex surface chemistry of mixed metal oxides in the presence of gases, such as oxygen and water vapor, often in the presence of a potential gradient either along the sample surface or from the surface to the bulk. Daniel Friebel (invited; SSRL) and Rosa Arrigo (invited; Diamond Light Source) discussed the oxygen evolution reaction at the platinum cathode of a model proton exchange membrane fuel cell. New results on this system were also presented by Yasumasa Takagi (Inst. Mol. Sci., Japan) who used hard X-rays (~8 keV) for the investigation of the surface chemistry under operating conditions.

An exciting new development is the investigation of liquid/solid interfaces using APXPS, which is in particular relevant for electrochemistry, geochemistry and atmospheric/environmental science. The challenge in these experiments is to detect electrons from the interfacial region through the adjacent condensed phases. At the meeting, two approaches to this problem were presented: Takuya Masuda (invited; NIMS Tsukuba) and Juan Velasco-Velez (Fritz Haber Institute, Berlin) described the use of ultrathin solid membranes in contact with a bulk liquid, where the photoelectrons from the interface are detected through the membrane. This method was used to investigate the electrochemical growth of Si in the presence of water (Masuda) and iridium nanoparticles deposited onto a graphene membrane in the presence of a potassium hydroxide solution (Velasco-Velez). Another approach is to form a 10 to 30 nm thick liquid layer by dipping and then partially removing a solid substrate from a solution while maintaining the partial pressure of the solution in the background. Examples of this method were provided by Marco Favaro (LBNL), who studied the corrosion of cobalt oxide supported on silicon, and Andrey Shavorskiy (MAX-IV, Lund), who investigated light-induced changes in the band alignment at hematite surfaces in contact with a potassium hydroxide solution.

The utility of APXPS for the investigation of heterogeneous chemical reactions in the atmosphere and environment was highlighted in talks by Michael Makowski (UC Irvine), describing measurements of ion concentration gradients at liquid/vapor interfaces using a combination of APXPS and a liquid micro-jet. Anthony Boucly (UPMC Paris) presented studies of ion concentration gradients on static droplets, in addition to the swelling of clays in the presence of water vapor, and Astrid Waldner (Paul Scherrer Institute) reported on measurements of the uptake of trace gases on ice surfaces. Luca Artiglia (Paul Scherrer Institute) presented measurements of the reaction of ozone with aqueous solutions using a combination of APXPS and a liquid micro-jet.

As in the past meeting, this workshop also featured talks by the two leading suppliers of commercial APXPS instruments. Andreas Thissen (SPECS Surface Nano Analysis GmbH, Berlin) and

John Åhlund (ScientaOmicron, Uppsala) presented updates on the latest developments of APXPS instruments and related equipment.

The final presentations of the meeting were brief updates on recent developments and upcoming installations of APXPS instruments both at synchrotrons and in the lab. Facilities that were discussed included MAX-IV (Joachim Schnadt, Lund), Diamond Light Source (Rosa Arrigo), NSLS-II (Ira Waluyo), Soleil (Anthony Boucly), Imperial College, London (David Payne) and Oregon State University (Gregory Herman).

The workshop concluded with an open discussion of a broad range of topics related to APXPS, including the all-too-common problem of cross-contamination between experiments using the same apparatus, which is in particular an issue for shared instruments at synchrotrons. A very lively debate ensued on the appropriate name and acronym for the technique, that has variously been called high pressure, near ambient pressure and ambient pressure XPS. While there was no unanimous agreement on a single acronym, the attendants of the session agreed that "ambient" or "near-ambient" pressure are the most appropriate terms, with the added benefit that literature searches for "ambient" apply to either of the acronyms. We also took advantage of the opportunity to introduce the development of a new website, www.apxps.org, which will serve as a centralized location for archiving APXPS publications and a portal to share other information on experimental best practices such as, e.g., contamination mitigation strategies, as well as workshop, meeting and other announcements relevant to the APXPS community. This is in the very early developmental stages but we hope it will grow and develop into an important and useful tool for the community.

The discussion session concluded with the planning of future workshops. The participants agreed that, for the foreseeable future, these workshops should be held annually, with the next three workshops taking place at Diamond Light Source/U.K. (December 12-14, 2016; organizer: Georg Held, Diamond/Reading University), Shanghai Light Source (2017; Zhi Liu, SLS/Shanghai Tech University) and Berlin (2018; Robert Schlögl, Fritz Haber Institute, Berlin). The conference site for the 2019 workshop will be decided during the meeting in Shanghai, and after that, always two years in advance of the workshop date. The great interest in hosting these workshops bodes well for the growth and vibrancy of the APXPS community over the coming years.

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Robert Schlögl (Fritz Haber Institute, Berlin) presenting the opening plenary lecture. © 2010 The Regents of the University of California, through the Lawrence Berkeley National Laboratory.

Miquel Salmeron (LBNL) giving a plenary talk during the opening day of the workshop. © 2010 The Regents of the University of California, through the Lawrence Berkeley National Laboratory.