Ambient pressure photoelectron spectroscopy and practical *operand* measurement

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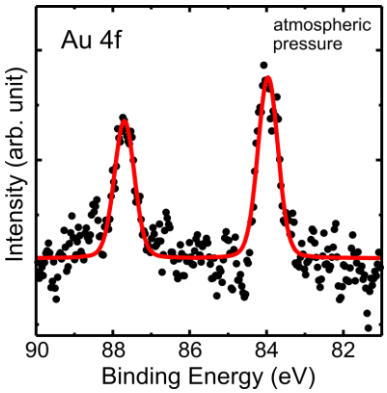
Conventional photoelectron spectroscopy (XPS) can be performed only under vacuum, but the recent development of a differential pumping type photoelectron analyzer and a high brightness synchrotron radiation have allowed us to measure a spectrum under a gas pressure of about 3000 Pa. We have developed a near ambient pressure hard X-ray photoelectron spectroscopy (NAP-HAXPES) system at the BL36XU of SPring-8 [1] and have observed a gas reaction on a solid surface using the system [2]. However, the XPS measurement under higher gas pressure is desired because many practical chemical reactions occur under atmospheric pressure. Therefore, we have conducted research and development to further raise the pressure limit in the NAP-HAXPES measurement.

In order to raise the upper limit of the measured pressure in NAP-HAXPES, it is necessary to reduce the diameter of the aperture to decrease the amount of gas flowing into the photoelectron analyzer. Furthermore, it is also necessary to shorten the distance between the sample and the aperture (working distance: WD) to suppress scattering of photoelectrons by the atmospheric gas. We fabricated an aperture with a diameter of 30 μm by a focused ion beam and attached it to the NAP-HAXPES equipment. We set the WD to 60 μm, twice the diameter of the aperture, using an *XYZ* stage with a position resolution of 1 μm or less and a θ stage with an angular resolution of 0.1 ° or less. The NAP-HAXPES measurement of Au(111)/Mica was carried out using hard X-ray of 7.94 keV as excitation light. The spectra of Au 4f and Au $3d_{5/2}$ were measured with increasing the gas pressure from 1 Pa to the atmospheric pressure. As the result, we confirmed that the accurate XPS measurement was possible even under atmospheric pressure [3].

By raising the pressure limit of NAP-HAXPES, it is possible to measure various samples *in operando*. For example, a sufficient humidity is necessary to increase a reaction efficiency in a fuel cell electrode, and oxygen gas is necessary as a fuel at the same time. Therefore, it is possible to measure the electrode reaction at the practical operation of the fuel cell only under a gas pressure of 30 kPa or more. It is also possible to directly observe changes in electronic states of hydrogen adsorption metal under the hydrogen atmosphere at near atmospheric pressure.



Au 4f spectrum under atmospheric pressure.

Bibliography

[1] Y. Takagi et al., Appl. Phys. Lett., **105**, 131602 (2014).
[2] Y. Takagi et al., Phys. Chem. Chem. Phys., **19**, 6013-6021 (2017).
[3] Y. Takagi et al., Appl. Phys. Exp., **10**, 076603 (2017).

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