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Photocorrosion and protection of CuBi2O4 electrodes monitored by operando surface-sensitive x-ray scattering and impedance spectroscopy

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The low stability of most semiconducting materials is a key challenge in the development of efficient photoelectrochemical (PEC) water splitting cells. Monitoring the semiconductor-electrolyte interface during operation is essential to understand all the underlying photocorrosion processes and, in turn, establishing appropriate mitigation strategies.
We developed a custom-built PEC cell that enables real-time assessment of the crystalline and morphological evolution of the semiconductor surface by operando grazing-incidence X-ray scattering. We applied the technique to monitor the evolution of CuBi2O4 films, a promising p-type semiconductor for the cathodic compartment of a PEC cell.
Our operando technique, combined with complementary X-ray absorption near edge spectroscopy (XANES) and inductively coupled plasma mass spectroscopy (ICP-MS) measurements, uncovers multiple degradation pathways affecting CuBi2O4 films performance during PEC operation. We found that CuBi2O4 undergoes reduction to metallic Bi and Cu, with the first one being the fastest process. Additionally, Cu ions are released in the electrolyte during long-term stability tests, while BiPO4 forms at the surface of the CuBI2O4 film, due to the presence of phosphate ions in the electrolyte. This study provides a comprehensive view of the degradation mechanisms at the CuBi2O4 electrodes surface under operation and establishes a methodological foundation for investigating the photocorrosion of a wide range of PEC materials.
Additionally, using Electrochemical Impedance Spectroscopy (EIS) under light illumination, we also monitored the charge transport properties of CuBi2O4 films protected with TiO2 via atomic layer deposition (ALD). By fitting the EIS data with an appropriate equivalent circuit model, we extracted the charge transfer resistances, capacitances, and time constants that influence the PEC performance of the electrode as a function of the TiO2 layer thickness. Our findings reveal that a band-mismatch between the two materials leads to the accumulation of photogenerated electrons at their interface, resulting in a performance decline for TiO2 thickness greater than 15 nm

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