

Considerations for the Cyclic Voltammetry of Gold in Sulfuric Acid Solutions

*Xingxing Xu*¹, *Asta Makaraviciute*¹, *Jean Pettersson*³, *Shi-Li Zhang*¹, *Leif Nyholm*², *Zhen Zhang*^{1*}
¹*Division of Solid-State Electronics, Department of Engineering Sciences, The Ångström Laboratory, Uppsala University, P.O. Box 534, SE-751 21 Uppsala, Sweden*
²*Department of Chemistry, The Ångström Laboratory, Uppsala University, P.O. Box 538, SE-751 21 Uppsala, Sweden*
³*Department of Chemistry-BMC, Analytical Chemistry, Uppsala University, P.O. Box 599, SE-751 24 Uppsala, Sweden Uppsala, Sweden*
*Corresponding author: Zhen.Zhang@angstrom.uu.se

A comprehensive understanding of the cyclic voltammetry (CV) for gold surfaces is essential for advanced applications. In the present study, a series of experiments were designed to investigate CV for gold under different experimental conditions when using a conventional configuration of a Ag/AgCl/sat. KCl reference electrode and a platinum wire counter electrode. The interferences introduced by the configuration were reflected in the three fingerprint regions of the voltammograms. It was found that the shape of the voltammograms was less reproducible at a lower sample volume when the cycle number was increased. This observation could be explained by different concentrations of Cl⁻ leaking from the reference electrode and platinum dissolved from the counter electrode. The reproducibility of the gold oxidation and reduction (Ox/Re) region in the voltammograms was improved when gold dissolution and re-deposition caused by Cl⁻ leakage was eliminated by using a bridge. In the hydrogen evolution and oxidation reactions (HER/HOR) region the catalytic performance of the gold electrode could be minimized by replacing the platinum counter electrode with a graphite rod. Alternatively, it could be enhanced by increasing the surface ratio of the co-deposited platinum to gold. In the electric double layer (EDL) region, peaks dependent on the concentrations of Cl⁻ and SO₄²⁻ were observed. To account for the occurrence of these peaks, a new mechanism based on the formation of neutral gold (I) complexes at very low Au⁺ concentrations, was proposed.

