Preparation of a Au SERS substrate and its application in the in-situ Raman spectroelectrochemistry study of $\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3$ molten salt

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Molten carbonate is often used as a medium for the decomposition of carbon dioxide to produce carbon and oxygen. In-situ Raman spectroelectrochemistry is an effective means to study species changes and the electrochemical reaction mechanism at the electrode-electrolyte interface. In this paper, a Au surface-enhanced Raman spectroscopy (SERS) substrate was prepared by an electrochemical oxidation-reduction cycle (ORC) process, and the reaction mechanism and intermediate species on this kind of Au SERS electrode in a $\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3$ molten salt were studied. The prepared Au SERS substrate has a SERS factor of 1.37×10^4 , and it presents excellent uniformity and long-term stability. Although the SERS effect of the Au substrate gradually weakens as the temperature increases, it maintains an acceptable SERS factor of 8.76×10^2 after a high-temperature treatment at 873 K. The cyclic voltammogram result shows that carbon reduction preferentially occurs on an Au electrode during the cathodic electrochemical process in $\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3$ molten salt. In-situ Raman spectroscopy confirms the existence of the intermediate peroxide ion (O_2^{2-}) during the electrochemical reduction process.

Keywords: In-situ Raman spectroscopy; SERS; Carbonate molten salt; Peroxide ion

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