Fabrication of a gold single crystal microelectrode annealable up to 980 °C.

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Ultramicroelectrodes (*umes*) are important in electrochemistry and electroanalysis because of the special mass transfer conditions observed with respect to those at electrodes of normal dimensions. This property has been used to facilitate the measurement of very fast electron transfer kinetics, a well-known example being the electrooxidation of ferrocene. On the other hand *umes* made from metals in the usual way are necessarily polycrystalline in nature. As a result, the orientation of gold grains on the surface of the electrode and therefore its double layer properties can vary from electrode to electrode. This fact prevents assessment of the role of interfacial properties in the kinetics of very fast electron transfer processes.

A method for fabrication of gold single crystal ultramicroelectrodes (*scumes*) has been recently outlined [1] . This technique has been used to fabricate gold (111) and gold (100) ultramicroelectrodes with effective diameters from 10-50 μm. Cyclic voltammograms for these microelectrodes in $HClO_4$ show the same features as single crystal electrodes of more conventional sizes. This confirms that single crystallinity was maintained during all preparation steps. Although gold *scumes* fabricated in this way can be cleaned electrochemically by cycling through the so called gold oxide region, annealing especially if one would apply the same technique to fabricate platinum single crystal ultramicroelectrode, seems to be an essential step to prepare a clean surface prior to electrochemical measurements. Use of an epoxy resin in this case would prohibit this step to be done.

In this paper, the fabrication of gold (111) *scumes* is reported using a glaze as an insulation material. The glaze used in this fabrication contains finely ground aluminum and silica oxides together with a flux, which is usually sodium or potassium oxide. Unfired glaze also contains a binder such as dextrin. During the firing process the mixture of these oxides forms a glass insulation with melting point around 980 °C. The properties of the unfired glaze allow the previously described setup [1] to be used for the fabrication with only minor modifications. The body of an electrode is composed of ceramic material compatible with a given glaze. Electrical contacts are all gold-gold thereby eliminating problems with contamination of the electrode at elevated temperatures by diffusion from the microwire-crystal interface.

Scumes fabricated in this way (see fig. 1) can be annealed up to the melting point of the glaze. Gold (111) *scumes* were characterized using cyclic voltammetry. The results of our experiments with these systems are described in this paper.

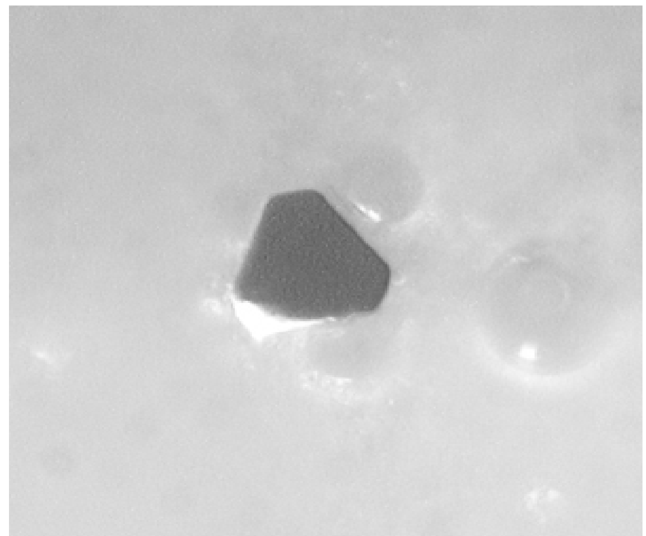

Figure 1: Gold (111) *scume* embedded in the glaze. Electrical contact with the crystal was made from below the crystal. The glaze during the firing process leveled leaving only one face of the crystal exposed.

References:

1. V. Komanicky, W. R. Fawcett, *Angewandte Chemie,* 40, Issue 4, **2001**, 563-566