Electrochemically Controlled Preparation of Gold Particles on HOPG and Characterization by Scanning Tunneling Microscopy*

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It has been realized that nanometer-scale metal particles differ from their bulk in physical and chemical properties. For example, significant catalytic activity has been observed from supported gold particles of several nanometers.[1]. Among various ways of fine particle preparation, metal electrodeposition is unique from view points of ease of control in electrode potential, external light excitation, solution composition and therefore the particle size and micro-crystallography.

Highly oriented pyrolytic graphite (HOPG) has been regarded as an ideal substrate for STM since a clean and smooth surface is easily obtained. However, the almost defect free HOPG surface places difficulties both for the nucleation and stabilizing metal particles because of weak interaction between the metal particles and HOPG. By activating the HOPG with anodic oxidation or oxidation-reduction cycles, however, metal particles can be nucleated subject to the followed-by STM characterization. But the work is limited to only a few systems at present.

In the present work, gold particles have been electrochemically deposited onto HOPG surfaces with and without activation. Though, gold is a noble metal which should be easily deposited at controlled potential of –0.3 V vs. SCE (an overpotential of ca. 1 V), STM characterization shows that long induction period (~ 5 min.) is required before stable gold particles can be deposited and imaged by STM on smooth HOPG. Small layer by layer gold flakes of 150 nm are the characteristic of the deposit. If, however, the HOPG was activated prior to the deposition by oxidation in phosphate buffer solution[2] so that defects such as kinks and bumps can be generated, the nucleation of the gold particles is promoted which are stable to sustain for the STM tip rasting. Only very short time (~ 10 s) is sufficient for the same amount of gold particles to be deposited onto activated HOPG. Reduced particle sizes are observed upon decreasing the deposition time from 5 min to 10 s. Fig. 1 gives the STM image showing densely but isolated gold fine particles after electrodeposition at –0.3 V for 10 s on an activated HOPG. The gold particles are different in size ranging from 10 to 40 nm indicating the 3D progressive nucleation mechanism.

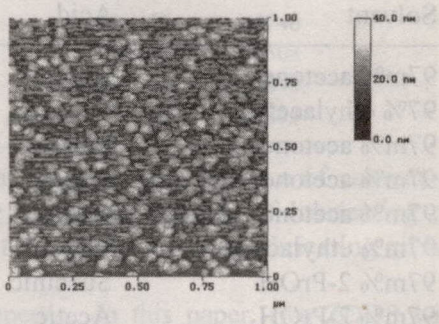

Fig1.Au electrodeposition on activated HOPG in 1mM $HAuCl_4$.

We have shown an electrochemical way of preparing fine gold particles which are expected to be catalytically reactive and to act as optically active centers (e.g. for SERS).

References

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