ESF Short Visit Grant - Scientific Report

Reference Number : 4619

Activity Title

New Approaches to Biochemical Sensing with Plasmonic Nanobiophotonics (PLASMON-BIONANOSENSE)

Title of the research project

In-situ SEM investigation of thermal annealing of SERS substrates

Applicant's Name and address

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Host name and address

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1 - Purpose of the visit

In recent months, the applicant has been fabricating patterned substrates for surface-enhanced Raman scattering (SERS) experiments that have been carried out by the group of Janina Kneipp at the Humboldt University in Berlin. The fabrication process consists of a unique combination of electron-beam lithography, metal deposition and a sequence of high temperature annealing steps to obtain the final structure. This circumvents the limitations of conventional e-beam patterning where the minimum achievable interparticle spacing is set by proximity exposure effects.

In order to get a more detailed picture of the formation of the final structure, a short visit was arranged to the University of Linköping in Sweden, where a scanning electron microscope (SEM) with a heatable stage (up to 750°C) was available. Such equipment is not commonplace, and not available at the applicant‘s home university. The visit was made possible with the kind help of Assoc. Prof. Johanna Rosén. Observing the annealing *in-situ* on 100 nm length scales allows for a more accurate determination of the required annealing temperatures and times, as well as their effect on the detailed form of the final structure. Pre-patterning of samples, including e-beam lithography, metal deposition and lift-off was carried out at the applicant‘s home institute, prior to the visit. Furthermore, a precise thickness calibration of a short-working-distance sputter coater for gold deposition at the host institute was carried out using x-ray reflection measurements.

Zeiss Leo 1550 microscope at the University of Linköping

2 - Description of the work carried out during the visit

Half a day was spent exchanging the normal sample stage of the SEM with the heatable stage. Temperature controller and water-cooling circuit were installed and tested. A significant effort was made to reduce vibrations due to cooling water flow to improve picture quality. Two days were used for sputter deposition of gold, thermal annealing and *in-situ* SEM investigations at elevated temperatures. The final products were SERS substrates containing triangular arrays of closely spaced gold particles, with a range of lattice constants, heights, and interparticle spacing. 1½ days were used for travelling.

3 - Description of the main results obtained

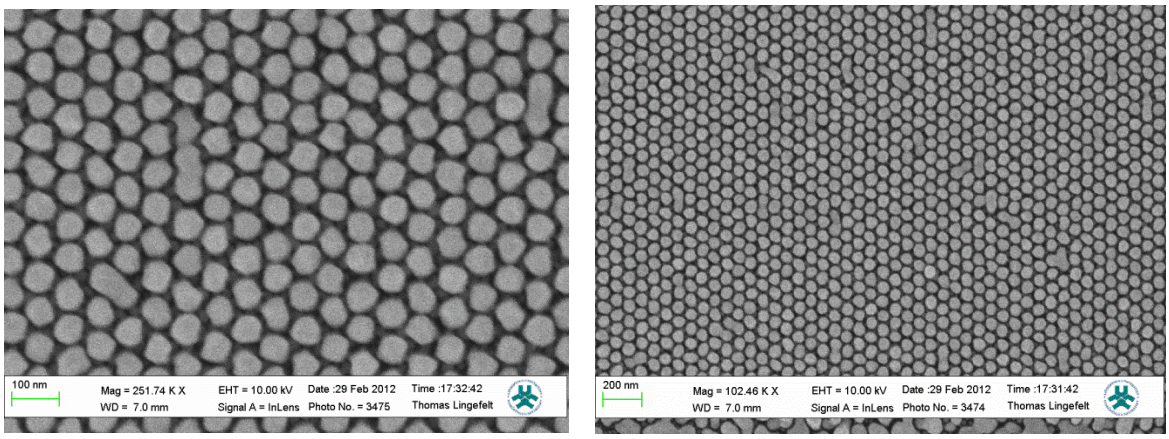

Fig. 2. SEM images of the fabricated structures

The above images show examples of annealed structures, consisting of nanoparticle arrays with a lattice constant of 70 nm and an interparticle spacing substantially lower than what can be obtained by direct electron-beam lithography. Live investigations of annealing behavior allowed for a precise

determination of the required temperatures for annealing (in vacuum) which were found to be substantially lower than temperatures used previously by the applicant (in air). Partly, this difference is due to a slowing down of diffusion in the presence of oxygen. Carefully controlled annealing conditions also result in completely different structure of the metal film in unpatterned regions. This structure will also be tested with respect to SERS enhancement, as it is a self-organized structure that can be readily fabricated on large-area substrates.

4 - Future collaboration with host institution

With some modifications, it should be possible to use the heated sample stage without water cooling, in the temperature range of interest. This would result in more stable imaging conditions. It would be interesting to continue the collaboration with the host institution to make further investigations along these lines. In addition, the host institute has a wide range of x-ray characterization tools which may be used to monitor changes in the crystal structure of the gold particles(also at elevated temperatures). It is clear that annealing significantly improves the crystallinity of the patterned particles, with positive effects on their plasmonic properties.

5 - Projected publications/articles resulting or to result from your grant

Samples fabricated during the visit will be characterized by Raman imaging at the Humboldt University in Berlin at the earliest opportunity. Results obtained on previously fabricated samples have given strong indication that the work might be of sufficient impact to warrant publication in journals such as Nano Letters, ACS Nano, or similar.

6 - Other comments

The applicant gratefully acknowledges assistance from Arni S. Ingason, Thomas Lindefeldt and Anna-Karin Eriksson.