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MAGNETRON SPUTTERING CLUSTER APPARATUS FOR FORMATION AND DEPOSITION OF SIZE-SELECTED METAL NANOPARTICLES

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The experimental setup utilizing a DC magnetron sputtering source for production of metal clusters, their size (mass) selection and following deposition in high vacuum is described. The source is capable to form clusters of various metals, for example, copper, silver, gold, *etc.* Cluster size selection is achieved using an electrostatic quadrupole mass selector. The deposited silver clusters are studied using atomic force microscopy. The height distributions show typical relative standard size deviation of 9-13% for given sizes in the range of 5-23 nm. Thus, the apparatus demonstrates good capability in formation of supported size-selected metal nanoparticles with controllable coverage for various practical applications.

1. Introduction

In the past two decades there has been an increasing interest in the deposition of size-selected metal clusters on various surfaces. Properties of clusters depend on the number of constituent atoms or molecules. Therefore, controlling the size and structure of a cluster allows tuning electronic, optical, magnetic and chemical parameters of the synthesized nanostructures. Cluster ion beam technique provides a number of advantages such as a precise control of the composition and size of clusters, surface coverage as well as kinetic energy which defines the cluster-surface interaction regimes [1,2]. Clusters (nanoparticles) on surfaces define a new class of systems highly relevant for practical applications such as biosensing, nanoelectronics, non-linear optics, catalysis and formation of nanocomposites for various applications [1-5].

Clusters can be produced by different methods [2]. Sources based on plasma sputtering using a magnetron provide vaporization of metal atoms from a target and their efficient agglomeration into clusters of various sizes [6]. Advantage of such sources is relatively high intensity of cluster beams as well as a possibility to produce nanoparticles of different metals. In the current paper, the design and capabilities of a newly build cluster deposition apparatus based on the DC magnetron sputtering source is described. Results of the first test experiments on the formation and deposition of size-selected silver clusters are presented.

Experimental

Schematic drawing of the magnetron sputtering cluster apparatus (MASCA) is shown in Fig. 1. The setup consists of several vacuum chambers. For cluster production a commercial source, NC200U from Oxford Applied Research, is connected to the source chamber. In the source, target material is sputtered into an aggregation region where clusters are formed and then expanded into the source chamber. More details about the process of magnetron sputtering can be found elsewhere [7,8]. Thereafter, the clusters are collimated into a beam by the conical in shape skimmer. After the skimmer the cluster beam enters the ion optics section. By Einzel lens and two pairs of deflectors the beam parameters are adjusted to enter the electrostatic quadrupole mass selector (EQMS) where clusters are size selected. EQMS consists of four equally distance hyperbolic electrodes surrounded by a grounded shield. Four electrodes are divided into two pairs which can be biased (U_{QP}) with opposite polarity, thus, bending the beam of charged clusters of desired masses for 90° into the deposition chamber. For details of the procedure see [6]. To measure intensity of the beam, Faraday cups are used. All chambers are evacuated by turbomolecular pumps (from 230 to 50 l/s) backed by rotary vane pumps. Using differential pumping a background pressure of 1.0×10^{-7} mbar is reached in the deposition chamber.

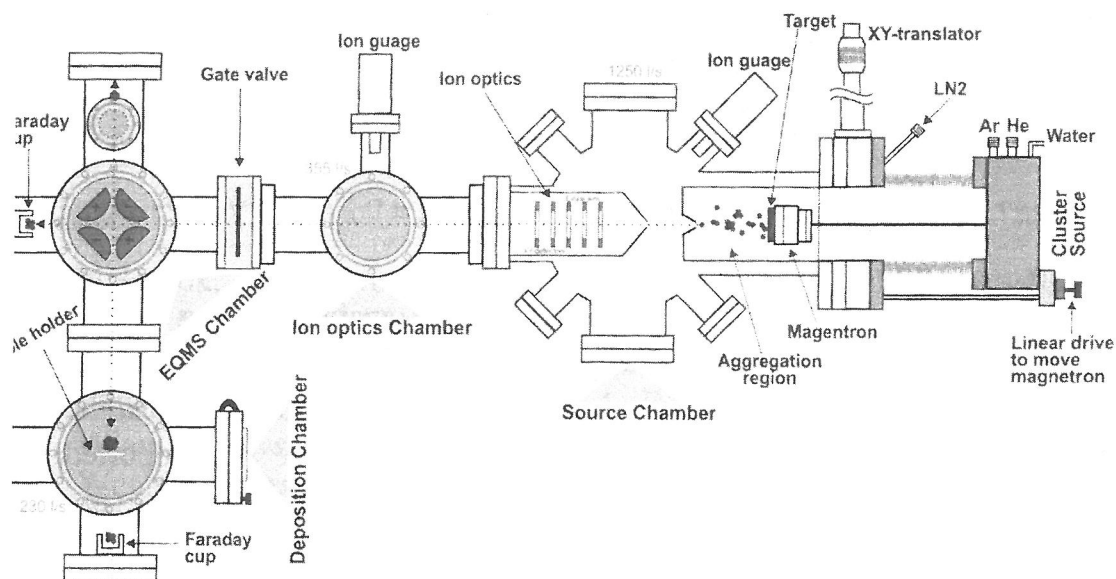


Figure 1. Schematic drawing of MASCA.

A silver target of 99.99% purity (from Goodfellow Ltd) is used for cluster production. Size-selected Ag_n clusters were deposited on clean Si(100) substrates at room temperature. Cluster kinetic energy is kept in so-called thermal regime providing good conditions for soft-landing without significant distortion of cluster shape which is assumed to be close to spherical in the gas

phase prior the deposition. Supported nanoparticles are studied by atomic force microscopy (AFM) in tapping mode using Ntegra Aura nanolaboratory from NT-MDT.

3. Results and discussion

The clusters inside the source are formed in different sizes from a few up to 1000's of atoms; significant fraction of them is ionized. The cluster sizes can be tuned by varying the discharge power, flows of sputtering (Ar) and aggregation (He) gases as well as aggregation length in the source. All these parameters were optimized in order to maximize beam intensity for the required cluster sizes.

Size-selection is carried out using following EQMS voltages $U_{QP} = \pm 100, \pm 300, \pm 500, \pm 900, \pm 1400$, and ± 2000 V. Clusters are deposited for 15-30 min to achieve considerable surface coverage.

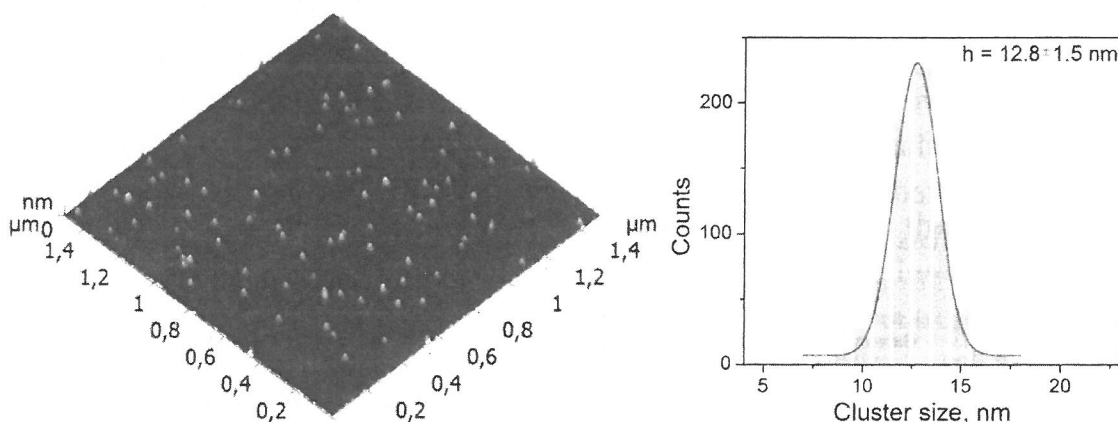


Figure 2. AFM image of silver clusters on silicon deposited at $U_{QP} = \pm 300$ V and histogram of the height distribution.

Heights of the deposited nanoparticles are analyzed using AFM. Earlier experiments [6] showed that shape of the deposited clusters is only slightly deviate from the spherical; the particles became a bit flattened. Thus, the height is almost equal to the diameter. Typical AFM image of supported silver nanoparticles with mean height $h = 12.8 \pm 1.5$ nm is presented in Fig. 2. Histogram of the height distribution for this sample is also shown, demonstrating relative standard deviation of *ca.* 12%. By varying the voltage we achieved deposition of the clusters with good size selection in the range between *ca.* 5 and 23 nm (see Table 1). By changing the deposition time the cluster surface coverage can be easily controlled. Further optimization of the sputtering conditions can allow fabrication smaller cluster or to go to very large ones depending on requirements of particular applications.

Table 1. Mean heights and standard deviations for the clusters deposited at different EQMS voltages.

Voltage, V	100	300	500	900	1400	2000
Height, nm	5.2±1.1	12.8±1.5	13.5±1.7	18.2±1.6	19.9±2.3	23.0±1.3

MASCA is also tested for the formation of copper clusters showing the capabilities very similar to those for silver ones.

4. Conclusion

Operation parameters of MASCA are optimized for the production of intense beams of silver and copper clusters with required sizes. The performance and efficiency of the electrostatic quadrupole mass selector is tested. Silver clusters of 5-23 nm are deposited on clean Si(100) substrates in high vacuum. Analysis using AFM shows typical relative standard deviations of cluster sizes within ca. 9-13%. Thus, the apparatus demonstrates good capability in formation of supported size-selected metal nanoparticles with controllable coverage that can be utilized for various practical applications, for example, for production of nanostructures with well-reproduced parameters of surface plasmon resonance which are attractive for optical sensing.

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