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Scanning tunneling microscopy of sputtered aluminum particles

B. Kaiser, K. Sattler, U. Müller, N. Venkateswaran, J. Xhie, and G. Raina

Department of Physics and Astronomy, University of Hawaii at Manoa, 2505 Correa Road, Honolulu, Hawaii 96822, USA

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Abstract. The scanning tunneling microscope is an ideal tool to study the local geometric and electronic structure of single supported metal clusters. Our experimental setup consists of an extraction type ion gun combined with a quadrupol mass spectrometer to deposit mass-selected metal cluster ions. First results showing scanning tunneling microscopy pictures of sputtered aluminum clusters are presented.

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Introduction

Small particles and quantum size systems have been of increasing interest in the fields of surface science, catalysis, microelectronics and photography during the last decade. All of these domains are basically concerned with the properties of particles at the surface of some kind of bulk material. The particles at this interface may have the size of only one atom, but they may also consist of several thousands of atoms and their properties are dependent on their size. Whereas many sophisticated investigations have been and are currently conducted on mass-selected clusters in the gas phase, traditional surface science methods have always been too insensitive to investigate a single particle, and most information available regarding clusters on surfaces is an average over a cluster size distribution. With the aid of the scanning tunneling microscope (STM) it is for the first time possible to study the local geometric and electronic structure of these particles on an Angstrom size scale [1].

The ideal experimental setup would provide for the possibility to generate an intense beam of cluster ions, which can be mass-selected and then deposited onto different types of substrates, which in turn can then be transferred under ultra high vacuum (UHV) conditions to an UHV-STM. In this paper we present the experimental setup to deposit mass selected clusters, and we show first STM results taken at room temperature and in air of sputtered aluminum particles on highly oriented pyrolytic graphite (HOPG).

Experiment

The experimental setup is shown in Fig. 1 and consists of an extractor type ion source for noble gas ion sputtering

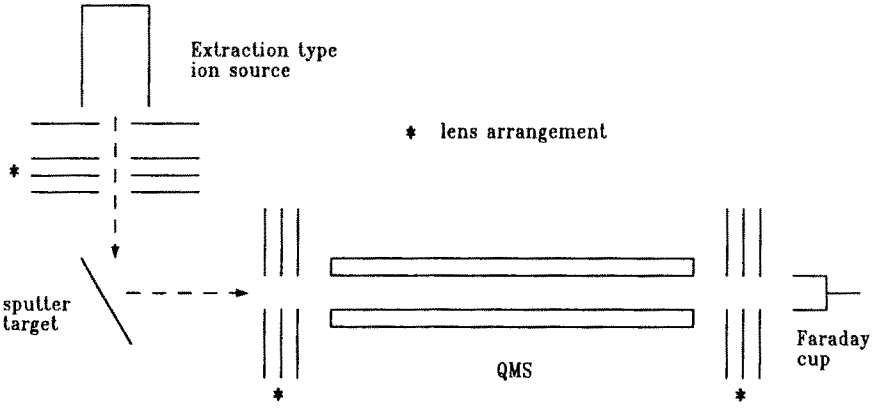
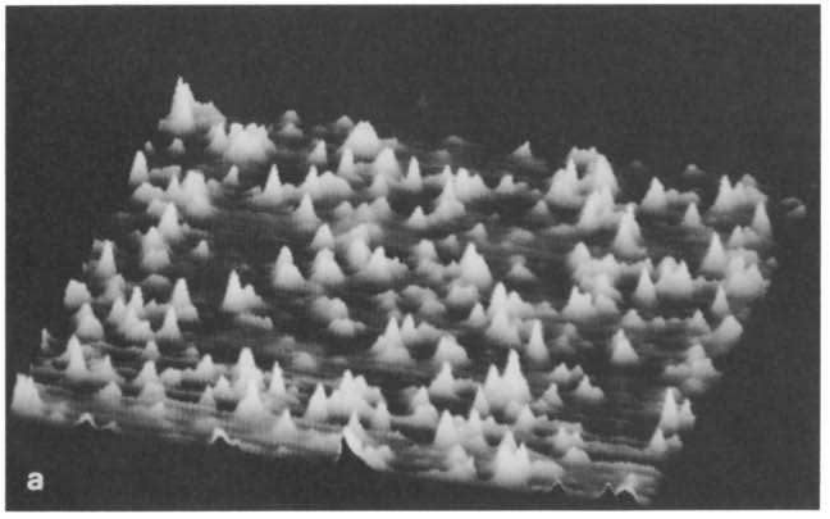
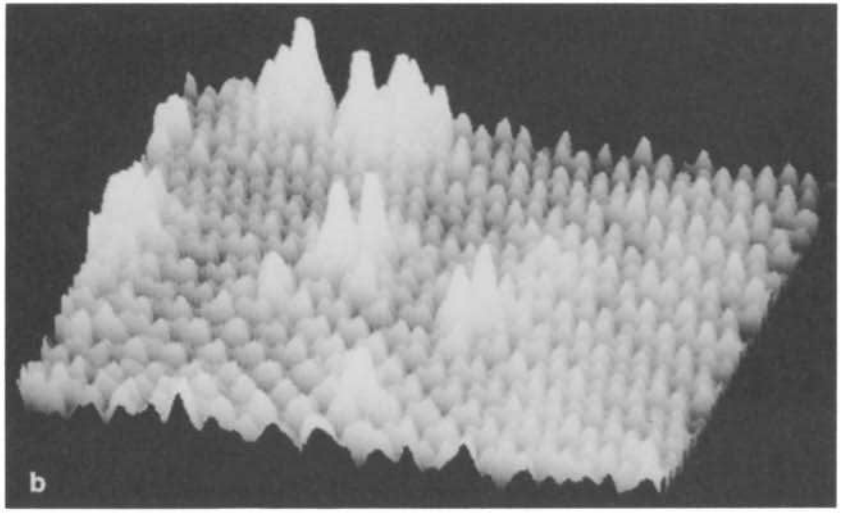
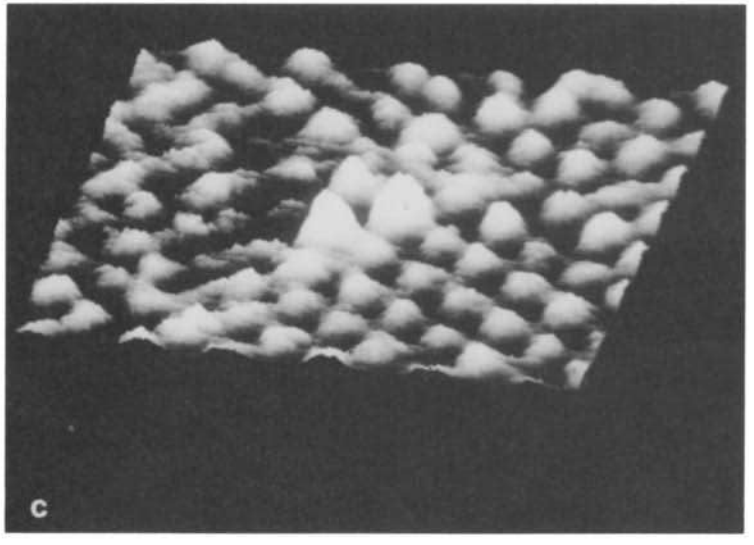


Fig. 1. Schematic drawing of the experimental setup







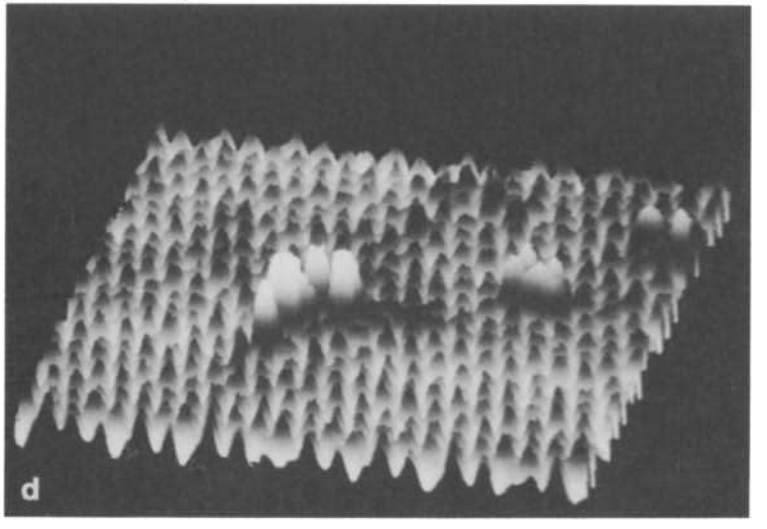


Fig. 2a–d. a 137 nm by 137 nm STM picture of sputtered aluminium clusters on HOPG, **b** zoomed portion of a, 8.6 nm by 8.6 nm, several particles of different sizes are highlighted on top of the graphite lattice, **c** dimer on HOPG, 3.14 nm by 3.14 nm, **d** tetramer and dimer on HOPG, 6.2 nm by 6.2 nm

up to a kinetic energy of 20 kV. The primary ion beam is focused onto a target and sputtered secondary ions are accumulated by the entrance lens of a quadrupol mass spectrometer (QMS) with a mass range of 1 to 1000 amu. At the exit of the QMS is a second lens to focus and decelerate the mass selected cluster ions. The size distribution of sputtered particles can either be analysed with a Faraday cup or secondary electron multiplier (SEM), or the Faraday cup can be replaced by a sample to collect mass-selected cluster ions. The sample can then be transferred to a UHV-STM under high vacuum conditions.

Results and discussion

First results regarding the deposition of aluminum particles have been achieved by using a simple experimental setup: Argon ions are accelerated in the ion gun to an energy of 4.5 kV and impinge without further focussing on an aluminum target 1.5 cm away from the exist aperture of the ion gun. The grounded aperture is also used as a holder for a graphite sample, which collects sputtered aluminum particles. The background pressure in the vacuum chamber is 10^{-6} torr and rises during sputtering to 10^{-4} torr of argon. The ion current at the aluminum target is of the order of 1 $\mu A/cm^2$. After a sputtering time of a few minutes the graphite sample is then transferred for further investigation to a STM operating in air and at room temperature [2]. The samples prepared in this way are stable at room temperature and under atmospheric pressure conditions for several hours. Figure 2 shows a series of STM pictures with sputtered aluminum particles of various sizes. Figure 2a) gives an overview over a large, 137 nm by 137 nm, area. Particles ranging up to several nanometers in diameter are clearly visible. Figure 2b) shows a zoomed portion of Fig. 2a) in a region between the large aluminum particles. The background shows the clearly resolved triangular graphite lattice and one finds small aluminum clusters, in this case two dimers in the center, a monomer in the front and part of a larger cluster in the background area. The picture also shows superstructures on the underlying lattice, which are often observed near large islands on graphite and which have been explained to be periodic charge density modulations [3]. Figure 2c) shows a single dimer on the graphite lattice. We find a distance between the two maxima of 2.6 Å, which is in good agreement with theoretical calculations of the bond distance in the aluminum dimer [4]. Figure 2d) shows a tetramer and a dimer.

So far, we assigned the observed features in the STM pictures to naked aluminum clusters, despite that one would expect this material to become oxidised easily under in-air conditions. But the longtime stability and the well resolved atoms in the particles having the right size make it reasonable to believe that we are indeed imaging the electron density of the metal particles. Because the clusters are produced under high vacuum conditions, they may use their free electrons to bind to the substrate, which therefore would not be available anymore to form oxides. Most everyday chemical reactions are not taking place in high vacuum. The STM is a specially suited tool to investigate chemistry and physics of surface reactions on an atomic scale under ambient conditions, and therefore might be valuable for the understanding of the underlying reaction mechanisms.

Further research will concentrate on the size dependent properties of these small particles and the experimental preparation of mass selected deposited metal clusters under UHV and ambient conditions.

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