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Cross characterization by scanning electron microscopy and atomic force microscopy of Ag islands grown on Si (111) 7×7

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Résumé. — Deux types de microscopies, la microscopie électronique à balayage (SEM) et la microscopie à force atomique (AFM), ont été utilisées d'une façon comparative pour étudier des îlots d'Ag de très grande taille (10 - 40 μm) formés sur l'interface Si(111)$\sqrt{3} \times \sqrt{3}$-Ag. Les images sont de nature très différente dans les deux cas: Electrons secondaires émis par l'échantillon en SEM, lumière réfléchie par la sonde balayant la surface en AFM. Les deux microscopes ont été utilisés dans des gammes de grossissement comparables. Une morphologie plutôt irrégulière et une topographie de surface démontrant de grandes inhomogenéités ont pu être mises en évidence sur les îlots dont la distribution de taille est par ailleurs très large. Les résultats obtenus en AFM et SEM sont, mis à part des différences mineures, principalement identiques.

Abstract. — Two types of microscopy, scanning electron (SEM) and atomic force microscopy (AFM), have been used in a comparative way to study very big (10 - 40 μm) Ag islands grown on a Si(111)$\sqrt{3} \times \sqrt{3}$ interface. The signals used for image formation are of very different nature; secondary electrons emitted from the sample, in SEM and, in AFM, light deflected from the system probing the surface. In the present study, both microscopes were used in similar magnification ranges. A rather irregular morphology of the islands and a surface topography with large inhomogeneities on the top face were observed. The size distribution of the islands was found to be very wide. AFM and SEM, for minor differences, basically give access to the same results.

1. Introduction.

AFM has only recently been developped into a powerfull tool for examining the fine structure of surfaces [1]. Scanning an object gives direct access to height differences thus visualises the surface topography. An optical detection system senses the vertical deflection of a cantilever as features of the scanned sample are encountered by the pyramid mounted on the cantilever edge. These deflections are directly translated into variations in height across the sample. SEM, on the

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contrary, is currently used since several decades for examining surfaces or growth systems. The contrast in SEM images is due to differences in the secondary electron emission (SEE) from two neighbouring points of the sample. This SEE increases considerably with increasing tilt angle between the incoming electron beam and the sample surface normal. Those parts of the sample that have different inclinations relatively to the surface plane will therefore be contrasted in SEM images. We have used SEM and AFM to examine Ag islands grown on a Si(111)7×7 surface held at 400 °C. The island size beeing between 10 and 40 μm.

Ag grows on Si(111)7×7 in the layer plus island growth mode. In this mode, islands grow after the formation of a 2D intermediate layer. For substrate temperatures exceeding 200 °C, this intermediate Ag layer is ordered in a $\sqrt{3} \times \sqrt{3}$ structure [2]. Ag islands of sizes up to 10 μm, grown on top of this $\sqrt{3} \times \sqrt{3}$ interface have been found to be very flat with a height to width ratio between 0.01 and 0.04 when observed under UHV [3]. This shape may change either when the Ag/Si(111) system is exposed to air [4], when the surface is contaminated [5] or in some cases when the islands exceed a critical size [6]. With increasing size, more irregular island shapes and an increase in island size distribution has been observed [3]. A more detailed study of individual, very large Ag islands of several 10 μm in size, grown on Si(111), has not yet been published to our knowledge.

The present short note compares AFM and SEM images obtained on Ag islands on the same sample using similar magnifications. The same results have been confirmed with both techniques.

2. Experiment.

The experiments were performed both, in a commercial AFM [7] and in a SEM [8], after transfer through air. In the AFM, a tube-type piezoscanner was used for rastering the sample in air and a force of about 10^{-8} N was applied to the probe cantilever during measurements. The images are either displayed in top view or in a three-dimensional surface plot representation. The SEM images were taken with a primary beam energy, Ep, of 15 keV and an angle of sample inclination, Θ, of either 0° (plan-view) or 60° towards the surface normale. For the observations only those magnifications common to both types of microscopes (from the ten to the hundred μm range) were used. The samples were prepared in a separate UHV chamber equipped with a standard LEED-AES retarding field analyser. As substrates we used commercial Si(111) wafer (p-type, 1.5 - 2.5 Ω cm). The samples were cleaned by flashing to 1200 °C by passing a direct current through the specimen until the LEED pattern showed the 7×7 reconstruction, characteristic for clean Si(111). Silver was deposited from a tungsten wire onto a 7×7 reconstructed Si(111) surface held at 400 °C.

3. Results and discussion.

AFM and SEM images were taken at steveral random places on the Ag covered Si(111) sample. Overview images taken in the hundred μm range with SEM and AFM are shown in figure 1. The island shape and size distribution can readily be recognized in figure 1a, where an SEM image taken at $\Theta = 0°$ is represented. Very little contrast due to surfacte topography is visible in this image which displays also a wide size distribution of the islands. This image is representative for the whole sample. In figure 1b another part of the sample is visualized with slidly higher magnification. Θ has been increased to 60°, giving rise to surface topography contrast. Every island consists of large, flat parts as well as much higher elongated bumps; the latter beeing always located along the edges of the islands. The islands are in epitaxy with the Si(111) substrate and the edges follow the high symmetry directions $[\bar{1}10]$ of the substrate. Again this image is representative for any random part of the sample examined. An AFM image at similar magnification, displayed in figure 1c, contains at the same time all information concerning the shape of the island base, the size distribution of the islands and their surface topography. A top view of individual Ag islands is given in figure 2a and b. The SEE in the SEM image (Fig. 2a) gives rise to only a small surface topography contrast. The depression in the Ag island is only weakly visible. In the AFM image (Fig. 2b), on the contrary, this depression can very clearly be distinguished from the bumpy edges. The streaks visible on the island side walls are due to an imaging artefact. Figure 3 displays in (a) the image of a similar Ag island taken at $\Theta = 60°$. At this sample inclination the surface topography contrast is enhanced and height differences on the island can be recognized. The surface plot of an island observed by AFM is given in figure 3b. This 3D representation does not contain more information concerning the surface topography of the island but is used to visualise the side walls of the island. More details of the surface topography are given in figure 3c, where one corner of the island is scanned by AFM with increased magnification. A flatter region of differing height with two distinguishable depressions and terminated by elongated bumps at the island edges can be recognized. Figure 3d shows a line scan across this corner of the island. The height difference between the flat region and the top of the edge is 320 nm, the depth of one of the depressions with respect to the flat region amounts to 80 nm. The size of the whole island at its longest expansion is about 38 μm (see Fig. 3b). These inhomogeneities in the surface topography have only been observed on these very big islands. Supplementary experiments have been carried out on smaller islands (up to 5 μm). Smaller islands can be grown by changing either the amount of the material deposited or the substrate temperature. The latter influences the island density and as a consequence, for a given coverage, their size. In our case, the smaller islands were obtained by lowering the substrate temperature to about 300 °C while keeping the coverage constant. The shape of these smaller islands can be described as compact with a flat top face and very little height difference, as already mentioned in reference [3]. We therefore believe that the peculiar island shapes observed in the present study are due to changes in the growth behaviour related to the increase in island size. A similar behaviour has recently been reported for the Ag/Si(100) system [6].

4. Conclusion.

The images obtained with the two different microscopes contain the same information concerning the islands. To get all the details, SEM has to be employed in a complementary way by taking advantage of the dependance of the SEE on the tilt angle to get the full information of the island size and shape. At $\Theta = 0°$ the base of the islands and their size distribution can be analysed whereas at $\Theta = 60°$, information concerning the surface topography is accessible. AFM images, on the contrary, gather all these features in one image but care has to be taken when imaging the side walls of the island. If the angle between these sides and the surface is smaller than the angle of the imaging pyramid mounted on the cantilever, this pyramid is imaged onto the island side walls (see e.g. the island in Fig. 2b).

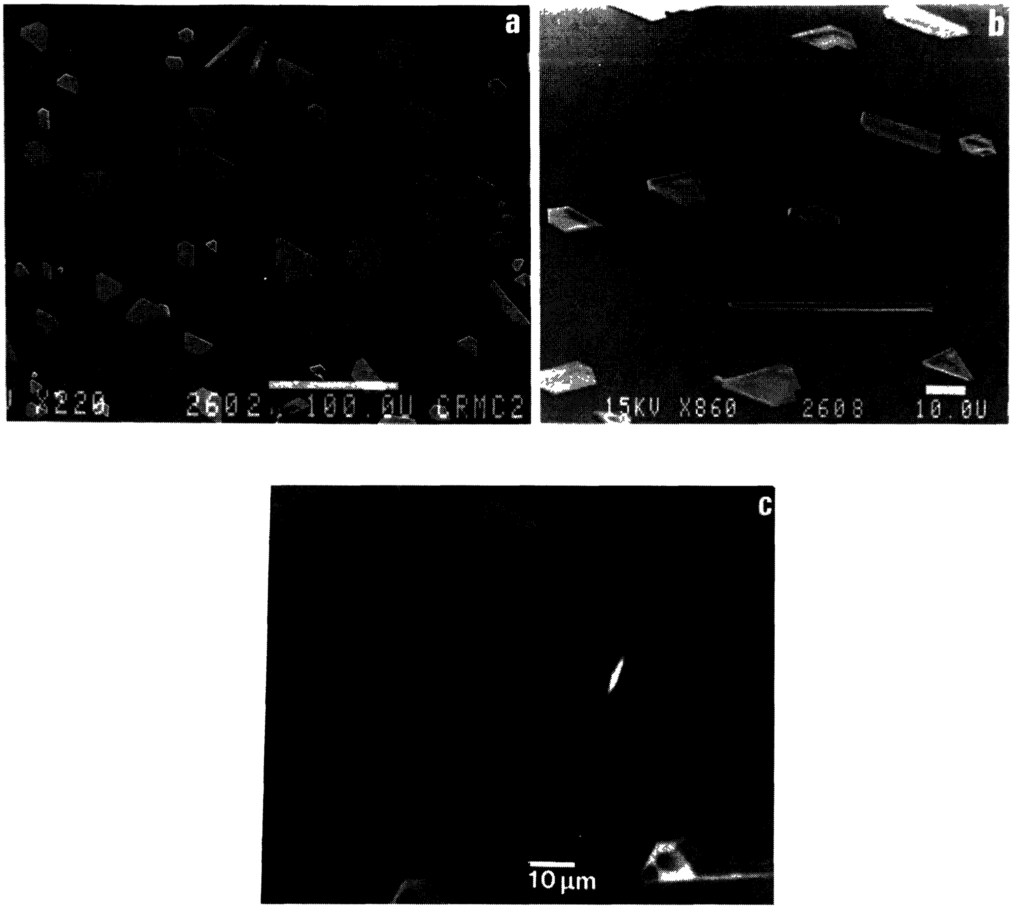

Fig. 1. — Overview images of Ag islands on the Si(111)$\sqrt{3} \times \sqrt{3}$-Ag interface. a) SEM image taken at Θ = 0°. b) SEM image taken at Θ = 60°. c) AFM top view image.

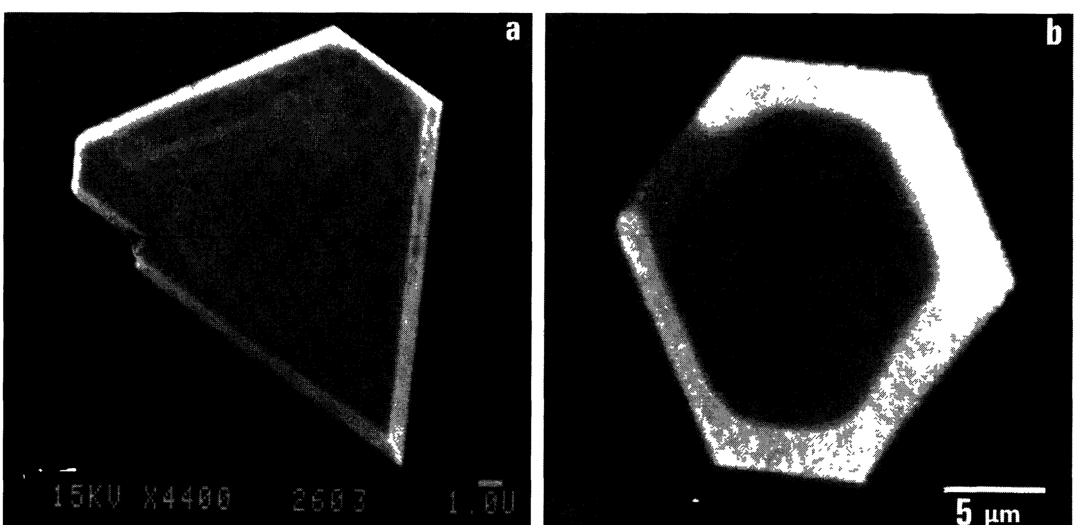

Fig. 2. — Top view images of two individual Ag islands. SEM image in a) and AFM image in b).

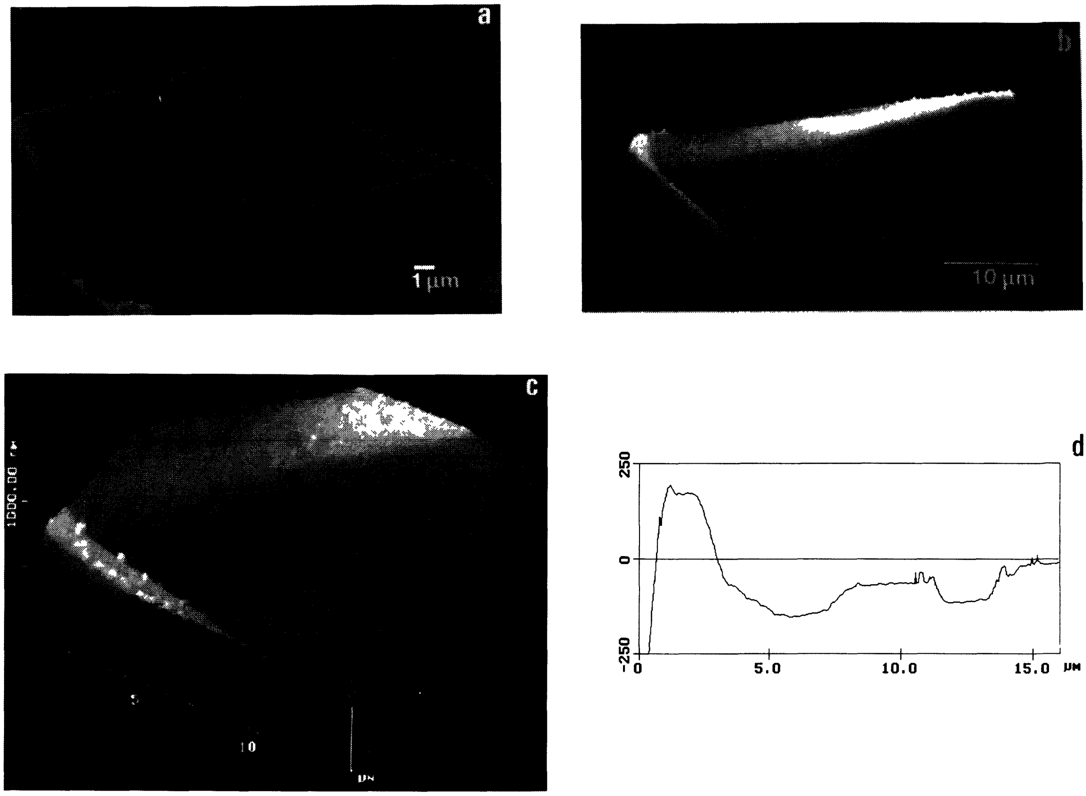

Fig. 3. — Visualization of the surface topography contrast of representative Ag islands on Si(111). a) SEM image at $\Theta = 60°$. b) AFM image reproducing a surface plot giving rise to a 3D representation of a similar island. c) Same island, same display as in b) but with higher magnification. d) Line scan across the corner shown in c).

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