Atomic force microscopy of silicon stepped surface

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Atomic force microscopy (Solver P-47H, NT-MDT) has been applied to investigate stepped silicon surface morphology during various treatments performed in an ultrahigh vacuum chamber of a reflection electron microscope. Contamination free silicon surface was achieved by high temperature annealing at the ultrahigh vacuum conditions. Details of the sample cleaning have been given previously. AFM study of the sample was carried out at atmospheric conditions after removing from the UHV chamber. By comparing atomic force data and contrast of reflection electron microscopy images, atomic processes on a silicon surface were studied in detail during sublimation, epitaxial growth, oxidation, deformation and oxygen etching.

Detail inspection of the stepped silicon surface reveals number of structural features of monatomic steps and surface defects. Step distribution with a few remarks regarding the implications of step rearrangements may be characterized being mainly based on the Barton, Cabrera and Frank theory. Peculiarities of step bunching and debunching induced by an electromigration effect were revealed from experiments on sample heating at various temperatures. To get flat surface morphology, a “mesa”-structured silicon substrates were fabricated by lithography and chemical ion etching technology. After cleaning procedure and thermal annealing extremely wide terraces (larger 20 µm) on the silicon (111) surface were created.

The experimental results of structural transformations during epitaxial growth in this paper are coming from AFM and REM studies (Fig.1). Careful analysis of the sample by both these methods reveals the number of individualities of the initial stages of epitaxial growth in spite of large number of the publications in this field. The interaction of adsorbed atoms with other adatoms, reconstructed domains, monatomic steps or other surface defects was under consideration. The characteristic length of germanium adatom migration on the silicon (111) surface at the various temperatures and rate deposition was measured. These data drawn in the Arrhenius plots allow to deduce the activation energy (1.3eV) of the germanium diffusion on the silicon (111) surface. Obtained results give possibility to control the epitaxial mechanisms and to improve the quality of the growth film through manipulation of mass transport on the silicon surface.

Fig.1. Two typical AFM-images of the silicon (111) surface after silicon deposition at the conditions of two-dimensional nucleation.
To analyse role of vacancies on the surface, the interaction of molecular oxygen with the silicon surface was examined in the wide temperature and oxygen pressure ranges. At low temperatures and high pressures of oxygen, the silicon surface passivated by a thin film of dioxide. Wherever at high temperature and low oxygen pressure, the surface vacancies were generated. There were found two mechanisms of oxygen etching of the silicon surface (Fig.2).

Fig.2. Schematical representation of various atomic mechanisms of thermal etching of the silicon surface by molecular oxygen.

At high supersaturation of the vacancies on the surface the negative island nucleation (pits) and their enlargement occur during oxygen exposure. Fig.3 shows typical AFM images of the silicon surface with two-dimensional negative islands. Dark spots of triangular shape (a) and circular (b) correspond to negative islands nucleated on the terrace during thermal etching of the silicon surface. The shape of islands depends on the surface reconstruction. Direct measurements of the depth of negative islands provided by AFM coincide with the monatomic step height. It is notable the existence of a denuded zone free from islands was observed along atomic steps. All vacancies formed inside this zone disappeared through interaction with monatomic steps. Moreover, negative island nucleation was not observed at narrow terraces. Further exposure of the silicon surface to oxygen causes enlargement of negative islands and the step motion in the step-up direction. The interaction of the moving step with islands leads to drastic changing of step shape and disappearing of islands.

Fig.3. Typical AFM-images of the silicon (111) surface with negative islands after oxygen etching. (a) 7x7 reconstructed and (b) non-reconstructed surface.

Step movement during thermal etching of the silicon surface by oxygen was accompanied by roughening of step edges. That should occur according to the competition of two processes: free energy minimization and entropy increase. A straight monatomic step is unstable against step meandering. We carried out statistical analysis of step shape fluctuations and interstep distance measurements on the silicon (111) surface by both UHV-REM and AFM. At the same thermodynamical conditions the maximum amplitude of step fluctuation was registrated for monatomic step escaped from step bunch. Inside of step bunch step fluctuations have minimum amplitude due to restriction of step-step interaction. An increasing the amplitude of step fluctuations near bunch is result of the interaction between monatomic step and step bunch (Fig.4a). The same increasing of step fluctuation amplitude was found for atomic steps escaped from step bunch during homoepitaxial growth (Fig.4b). An amplitude analysis of these fluctuations allows to deduce number of parameters for the adatoms, monatomic steps and surface such as effective step edge stiffness, surface diffusion constants and step-step interaction.

Fig.4. AFM images (phase contrast) of the silicon (111) surface with step bunches after oxygen etching at 700°C (a) and silicon deposition (0.2ML) at 740°C (b).

The consecutive stages of nucleation of three-dimensional gold islands on the silicon (111) surfaces with various morphologies were visualized by AFM during gold adsorption. The shape and distribution of gold clusters were analysed over a wide temperature range of the substrate. It was found a drift of three-dimensional gold islands in the step-up direction during thermal annealing. This direction of the island drift occurred for both directions of an electric direct current heating the sample while the drift rate was higher with the heating current flowing in the step-down direction. An influence of step bunches and anti-bunches on the surface redistribution of the gold islands was observed.

Obtained results provide opportunities for better understanding elementary structural acts contributing self-assembled and self-ordered structural processes on semiconductor surfaces.
Preferable places for oxidation and epitaxial growth of silicon, germanium, gold and titanium were used for ordering nanostructures on the silicon surface.