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Nanometer-scale patterning of alkali halide surfaces by ion bombardment

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ABSTRACT

Structural properties of alkali halide surfaces (KBr, KI, and RbI) have been modified by low energy Ar^+ ion beam bombardment. The morphology developed on the irradiated surfaces was investigated by the atomic-force microscopy (contact mode) under UHV conditions. The results indicate that for the incidence angles between 30° and 40° a periodic ripple morphology is developed with the ripple vector parallel to the beam direction, whereas for the angles larger than 60° the vector is perpendicular to the beam. The average surface roughness and the wavelength of the ion-induced periodic structures were measured as a function of the ion fluence (10^{13}–10^{18} ions/cm^2), the beam energy (1–5 keV), and the sample temperature (from 300 K to 600 K). The results are explained in terms of a new atomic-scale model taking into account the fact that ion sputtering of alkali halides in the investigated energy range is dominated by electronic processes rather than ballistic collisions.

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1. Introduction

Surface patterning by ion bombardment has attracted considerable attention over the last years due to its possible applications in various fields of science and technology. In this context a low-energy ion beam has been considered as a convenient tool for manufacturing of large area patterned templates for deposition and assembling of large molecules, for achieving surfaces with tunable frictional properties, or for use in optoelectronic and quantum devices [1]. For example, KBr nanostructured by the electron stimulated desorption (ESD) process has been used successfully as a template for metal clusters [2], as well as for complex molecule deposition and immobilization [3,4]. Such structures, therefore, could be applied in future molecular electronics.

In all those applications it is highly desirable to create a surface pattern with controlled length scales by properly adjusting the ion beam parameters such as the beam energy, the angle of incidence, the ion current density, and the fluence. The structures most frequently found in metals and semiconductors are nanoscale dots at close to normal beam incidence [5] and ripples formed at more oblique angles [6,7]. Although there has been a general consensus about evolution of the ripple structure as being due to the competition between surface roughening introduced by ion sputtering and surface smoothing created by various diffusion related processes, details of such mechanisms are yet to be identified. Depending on particular experimental conditions, pure thermally assisted diffusion [8–10], viscous flow [11,12], ion-enhanced or ion-inhibited diffusion [13,14], and preferential sputtering without net mass transfer [15,16] were considered as important contributors to the effective surface smoothing. As a result, the exact mechanism for ripple formation remains controversial even after two decades since its first observation. Furthermore, validity of the continuum theory as applied to single

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crystalline metals has been questioned recently by van Dijken et al. [17] and Hansen et al. [18], who offered an alternate atomic-scale approach to surface morphology evolution under oblique ion bombardment.

Ion beam modification of ionic insulators, such as halides, was only scarcely investigated. The fabrication of periodic wire arrays by 4.5 keV Ar^+ bombardment of a CaF_2 (1 1 1) crystal at grazing incidence (4–6° with respect to the surface plane) at room temperature has been reported by Batzill et al. [19]. The morphology of the irradiated surfaces was investigated "ex situ" with an ambient AFM operating in contact mode. The results are indicating that the calcium fluoride surface is sputtered with preferential emission of fluorine, thus the excess of calcium at the surface is assembling into periodic wire-like stress domains. The postulated self-organization mechanism invokes minimization of the system free energy and the elongation of the wires along the beam direction due to angle dependent anisotropic sputtering of the wires. Such a mechanism is contrasted with the continuum theory of Bradley and Harper [8] and Makeev, Cuerno, and Barabasi (MCB) [7] which in that case is assumed not to play any substantial role.

Recently Mussi et al. [20] have investigated nanostructuring and optical activation of the randomly oriented lithium fluoride polycrystalline samples by 0.8 keV Ar ion sputtering at different ion fluences. The angle of incidence was fixed at 35° off-normal and the sample temperature was 80 °C. Although the AFM images were taken only for three different ion doses, the trend in ripple height and the mean periodicity seems to follow the predictions of the continuum theory and the scaling low predicted by Makeev, Cuerno, and Barabasi [7]. No oblique incidence ion bombardment was applied in that experiment.

In this paper, we report on the comprehensive studies of nanostructure formation processes in single crystal alkali halides (KI (0 0 1), RbI (0 0 1), and KBr (0 0 1)) induced by an Ar ion bombardment. In contrast to the previous work on halides, the surface morphology investigations were done in a UHV environment at every stage of the experiment, i.e., without exposing the samples to an ambient environment for AFM imaging. Since alkali halide surfaces are very sensitive to moisture, they could easily be modified by water adsorption at improper vacuum conditions. This is especially important for irradiated samples with a large surface density of structural and electronic defects [21]. We have found that appearance and orientation of the nanostructures are dramatically dependent on the ion beam angle of incidence, whereas the energy dependent studies revealed that the ripple wavelength and the RMS surface roughness increase with increasing ion energy. Also the ion fluence and the sample temperature dependences have been determined for various alkali halides and the results are compared with both the continuum theory predictions [7,8] as well as with the atomic-scale model proposed by Hansen et al. [18].

2. Experimental

The experiment has been performed in a multi-chamber ultrahigh vacuum system which consists of a preparation chamber equipped with the low-energy (1–5 keV) ion gun, a scanning probe STM/AFM microscope chamber (VT STM/AFM manufactured by Omicron Nanotechnology GmbH, Germany), an electron spectroscopy chamber (VG ESCA MK-II system), and a UHV mechanical sample transfer system (manufactured by PREVAC, Poland). The system enabled to maintain very good vacuum conditions (the base pressure in the range of 10^{-10} mbar) in all stages of the experiment including SPM characterization which is of crucial importance for studying alkali halide surfaces. Alkali halide crystals purchased from TBL-Kelpin (Neuhausen, Germany) have been cleaved in air parallel to the (0 0 1) cleavage plane and mounted on the molybdenum holder with the integrated resistive heater. After mounting, the samples have been immediately transferred into the vacuum system for proper cleaning by annealing in UHV at 450 K for several hours. The annealing process is essential in order to remove the adsorbed impurities and to reduce the stress and charge accumulation. The samples have been exposed to an Ar ion beam rastered over the entire surface area. The average ion current density at 3 keV was 16–18 $\mu A/cm^2$. The crystal holder was kept at the room temperature for all measurements, except the experiment monitoring the surface morphology dependence on the sample temperature. In this last case, the sample temperature was measured with a thermocouple attached to it, and an ample time was allowed for temperature stabilization prior to each bombardment cycle. The angle of incidence of the ion beam was varied in the range 5°–75° with respect to the surface normal and most often the beam projection on the surface plane coincided with the low index directions of the crystal. The data presented in Fig. 4, however, were taken with the crystal deliberately rotated changing the polar angle by about 30°. The beam energy was varied in the range 1–5 keV. The resulting surface topography has been analyzed by the atomic-force microscopy in contact mode (CM-AFM) under UHV conditions using the silicon cantilevers with a nominal tip radius of 20 nm. The average ripple wavelength is measured directly from AFM topography images, as well as from the corresponding 2D height–height autocorrelation functions. For better statistics, the AFM measurements have been done at several different areas of the irradiated sample surface. A supplementary measurement of translational velocity distributions of atoms sputtered from the selected alkali halides was performed by means of the mass-selected time-of-flight (TOF) spectroscopy. Details of the time-of-flight spectrometer are given elsewhere [22].

3. Results

A KBr (0 0 1) single crystal subjected to an ion bombardment develops a rather complex surface morphology strongly dependent on the angle of beam incidence. The AFM images shown in Fig. 1 represent typical examples of the surface patterns obtained after crystal exposure to 4 keV Ar ions with the total fluence of 4.0×10^{17} ions/cm^2. For the beam directions close to the surface normal the image corrugations are in the form of cavities and dots with the RMS roughness of a few nanometers (not shown). For somewhat more oblique angles (see the example for 15° in Fig. 1a), dots seem to accumulate into a network of nanorods with dimensions of 50–90 nm in width and 5–8 nm in height. The ripple-like structures are seen already at 30° off-normal incidence (Fig. 1b) but the ripple wave vector for this geometry is parallel to the surface projection of the incident beam. With further increase of the incidence angle the ripples are becoming more blurry and at 45° no clear signs of periodicity are seen. For the incidence angles larger than 60°, however, regular ripple structure is seen again but the wave vector orientation changes towards the one perpendicular to the incident beam. The example shown at 75° (Fig. 1c) represents a very fine example of a regular pattern of parallel ripples which could only be obtained at the oblique incidence. The 2D self-correlation function calculated for the image (shown in the inset) determines a well-defined value of 48 nm as the ripple wavelength. The overall roughness of the bombarded surface is not much sensitive to the incidence angle variation until the incidence angle of 50°–55° is reached where the amplitude of the sample buckling is rapidly doubling.

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