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Non-contact atomic force microscopy studies of (2×4) InP(001) surface

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Abstract

A sputter-cleaned indium-rich (2×4) InP(001) surface was investigated by non-contact scanning atomic force microscopy (NCAFM). Atomically-resolved images of the surface exhibit two different patterns. The patterns can be interpreted within the mixed dimer model of (2×4) reconstructed InP(001) surface. It is shown that due to contrast formation mechanism in NCAFM the features resolved are in close correspondence to scanning tunnelling microscopy (STM) data. Due to chemical interaction a P-terminated tip gives the image similar to an empty-state STM image, whereas an In-terminated tip gives the image resembling a filled-state STM one. Moreover, it is shown that due to dipole–dipole interaction, NCAFM can be sensitive to orientation of In-P dimers.

Keywords: Indium phosphide; InP; III–V semiconductors; Non-contact atomic force microscopy, NCAFM; Atomic surface structure; Surface reconstruction

1. Introduction

Structures of technologically important polar (001) surfaces of III–V compounds have been under debate for many years. In contrast to (110) cleavage faces, (001) surfaces exhibit a large variety of reconstructions depending on surface stoichiometry, which in turn depends on a preparation procedure [1–3]. Surface symmetries of reconstructed surfaces are well established on the basis of diffraction experiments. However, atomic composition of unit cells is often far from being understood. Scanning tunnelling microscopy (STM) was the most frequently used technique and several models of unit cells have been proposed based on analysis of STM images. However, tunnelling mechanisms responsible for contrast formation in STM are quite complex and STM data are often difficult to interpret unambiguously, even using sophisticated theoretical methods for their interpretation [4]. Therefore, there is no doubt that other techniques allowing for surface atomic structure investigation should be applied. Non-contact atomic force microscopy (NCAFM) has recently achieved the level of maturity necessary for using it as a routine tool in surface structure analysis. However, contrast formation mechanisms in the NCAFM are still under debate and atomically resolved images have to be analysed carefully. Recently, we have shown that for $c(8\times2)$ InSb(001) surface the NCAFM gives three different patterns which could be interpreted as images of the In surface sublattice, the Sb sublattice or both sublattices and were formed depending on the chemical composition of the tip apex [5]. Those results, together with earlier theoretical calculations [6,7] show that in the case of III–V semiconductors the NCAFM is in fact sensitive not only to geometrical positions of surface atoms but also to surface electronic structure. In the case of $c(8\times2)$ InSb(001) sublattice identification was

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possible since atomic positions were practically coincident with protrusions in the respective patterns. However, such a situation is not the rule and often protrusions in a NCAFM image cannot be directly ascribed to surface atoms [8]. That is the case for III–V (0 0 1) surfaces where surface dimers are present like in case of $(n \times 6)$GaAs(0 0 1) [9,10]. In the present study we are using NCAFM to image the (0 0 1) surface of another highly disputed III–V compound, on which surface dimers are present, (2×4) InP(0 0 1).

Due to lower partial pressure of group V elements, ion sputtering and annealing in UHV conditions lead to creation of surface structures enriched in group III atoms [1,3]. The most common surface symmetry obtained in such conditions is $(4 \times 2)/c(8 \times 2)$, which has been observed for compounds like GaAs, InSb and InAs [11,12]. Similarly for InP(0 0 1), (4×2) symmetry was also assumed for quite a long time for an ion-sputtered surface [13,14] and its structural models for were constructed based on analogous models of GaAs [2]. Later experiments, however, have shown that the surface symmetry is in fact (2×4) [15,16]. Several different models of the surface unit cell have been proposed, however, the most successful is the mixed dimer model proposed by Guo and co-workers [17] and Schmidt and co-workers [18,19] and all the data in the paper will by discussed with respect to that model.

2. Experimental

Pieces of InP(0 0 1) epi-ready wafer, $1 \times 1\ \mathrm{cm}^2$, were clamped to a molybdenum sheet attached to a large copper sample holder without any prior chemical treatment. Then the samples were introduced into a ultra-high vacuum system with the base pressure below 5×10^{-11} Torr consisting of three interconnected chambers with magnetically coupled linear transfers allowing for sample preparation and examination in situ by various surface sensitive techniques. The wafers were outgassed overnight at the temperature of 550 K (as measured by a chromel–alumel thermocouple). Both sample holders and a sample stage mounted to the manipulator were massive copper blocks and a small amount of indium was put between the samples and the molybdenum sheets in order to ensure proper heat transfer. Since temperatures used were not high enough to lead to significant energy loss due to radiation we are quite sure that the temperatures measured by the thermocouple were close to the real temperature of the sample. After outgassing the samples were rapidly heated up to 750 K for approximately 2 min. Subsequently, the samples were cooled to 600 K and at that temperature ion sputtering was performed. Ar^+ ion beam of energy 300 eV and current of approximately 1 μA was rastered over the area of 1 cm^2. The samples were irradiated for 2×15 min with ion beam angle ±45° to the surface normal. Afterwards, the samples were rapidly heated for 5 min to the temperature between 700 K and 750 K. Sputtering–annealing procedure was repeated several times until a clear (2×4) LEED pattern was observed.

The sample exhibiting the clear (2×4) LEED pattern was than cooled to the room temperature and transferred to the microscope chamber. Sputtering–annealing cycles were repeated every few days in order to avoid gradual contamination of the surface due to adsorption of residual gases.

Park Scientific Instruments VP2 STM/AFM (scanning tunnelling microscopy/atomic force microscopy) microscope with NanoSurf easyPLL demodulator was used in the experiment to image the InP(0 0 1) surface in a non-contact mode (NCAFM) using the frequency modulation (FM) scheme [20]. Commercially available silicon piezoresistive cantilevers with resonant frequencies between 166 and 279 kHz oscillated typically with the amplitude of 20 nm were used as probes. In the FM–NCAFM, a cantilever oscillates with its resonant frequency and constant oscillation amplitude is maintained by a feedback loop. When the cantilever is close to a surface (i.e., the tip approaches less than several nanometres to the surface in the lower turning point) tip-surface interaction changes the cantilever frequency. The frequency change is demodulated by a phase-lock loop and the signal is then used as the feedback for topography imaging.

3. Results and discussion

The above described surface preparation procedure leads to the surface structure exhibiting the clear (2×4) LEED pattern, where (2×) periodicity is marked by disorder streaks, as seen in Fig. 1(a). A large-scale NCAFM image of the corresponding surface is shown in Fig. 1(b). The surface is covered by a system of atomically flat terraces, quite complex in shapes separated by steps of monolayer height oriented along [−1 1 0] and [1 1 0] directions. The terraces are typically few tens of nanometers wide and they are uniformly covered by reconstruction rows running along [−1 1 0] direction as shown in Fig. 1(c). Post-sputtering annealing to temperatures higher that 750 K leads to formation of In droplets on the surface having detrimental effect on stability of scanning, however not alternating the LEED pattern.

Atomically resolved NCAFM images of the (2×4) InP(0 0 1) surface are presented in Fig. 2. In the Fig. 2(a) the surface structure is imaged as series of bright protrusions with perfect ×4 periodicity along [1 1 0] direction, apart from quite common on the surface line defect where entire pattern is moved by the base surface lattice constant (i.e., 4.16 Å). Along [−1 1 0] direction, however, the periodicity is not perfectly maintained along the line of protrusions.

In Fig. 2(b) the same surface is depicted, however, as covered by wide bright stripes (with substructure) running along [−1 1 0] direction with periodicity corresponding to ×4 pattern. An atomic step is running through the middle of the image causing significant difference in brightness of the right and the left side of the image.

To interpret atomic patterns in NCAFM images it is necessary to discuss the contrast formation mechanisms