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Low energy ion beam-induced modification of InSb surface studied at nanometric scale

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Atomically flat InSb(001) surface has been prepared with cycles of sputter-cleaning and annealing. The surface structure has been characterized by low energy electron diffraction (LEED) and by atomic force microscopy (AFM). Then the surface has been bombarded with 4 keV Ar^+ ions incident 50° off normal, and the morphological changes have been studied with the AFM as a function of the ion dose. It was found that the surface was amorphized already for low ion doses ($\sim 2 \times 10^{15}/cm^2$). At higher ion doses (of $2 \times 10^{16}/cm^2$) the surface appeared to be covered with the system of parallel nanowires running along surface projection of the ion beam direction. Typical sizes of the nanowires were: 1.5–2 μm length, 50–70 nm width and height 5–7 nm.

1. Introduction

It has been reported recently that despite its stochastic nature, sputter-erosion of semiconductor and metal surfaces can lead to self-organization processes and as a result regular patterns of nanometer size structures can be formed on surfaces of the sputtered solids [1]–[3]. Characteristic features of those nanostructures, as well as their sizes depend on ion beam parameters such as current density, dose and incident angle. All those ion-beam parameters could be easily controlled and in principle, structures having desired shapes and sizes could be easily fabricated in a single technological step providing that underlying surface texturing mechanisms were well-understood. At the advent of nanotechnology, studies concerning formation processes of nanostructures deserve special attention.

On ion-bombarded $A_{III}B_V$ compound semiconductor surfaces two types of structures have been observed so far. On GaSb and InSb, FACSKO *et al.* [1] – [3] reported formation of nanodots of sizes 20 – 50 nm depending on ion dose (in the range $4\times10^{17} - 4\times10^{18}/cm^2$). The surface density of the dots is $\sim 4\times10^{10}/cm^2$ and it does not depend significantly on the ion dose. Formation of "granules" of the sizes of 30 – 100 nm has been also reported by CHINI *et al.* [4] on InP bombarded with 3 keV Ar^+ ions (dose 8×10^{18} ions/cm^2). Nanodots are formed for normal incidence angle of bombarding ions. For oblique ion incidence angles a different surface pattern is formed. DEMANET *et al.* [5] reported a system of ripples parallel to the surface projection of incident ion beam (5 keV Ar^+ 71°) with respect to the surface normal, 2×10^{18} ions/cm^2). Formation of ripples on ion bombarded solid surfaces has been also observed for materials other than compound semiconductors like Ag [6] and it is not material specific [7].

In the present paper we investigate structures formed by Ar^+ bombardment of InSb surface with an oblique ion beam and with ion doses in the range between $10^{14} - 10^{16}$ ions/cm^2. In contrast to previous studies in which the surface became textured as a whole due to sputter-etching, in our experiment nanowires are formed on the surface which remains essentially flat.

2. Experimental

An experimental system consisting of three UHV chambers (preparation, surface analysis, microscope) is used in the experiment. The base pressure in the system is 1×10^{-10} torr. The chambers are interconnected and samples can be moved with magnetically coupled linear motion transfers. The substrate is InSb epi-ready wafer purchased from Kelpin Crystals. A piece of the wafer (14 × 14 mm) is mounted on a tantalum sheet connected to a heated copper block. Sample holders can be heated up to 1000 K both in the preparation and in the analysis chambers. The sample temperature is measured on the copper block by a chromel-alumel thermocouple. Initially the substrate surface is cleaned with 0.7 keV Ar^+ ion beam, bombarding the surface at angles 60 and – 60 degrees off normal. The wafer is kept at $T =$ $= 700$ K during the ion beam sputtering. Cleaning cycles of approx. 1 hour duration are repeated until clean surface (as checked by Auger electron spectroscopy, AES) and clear c(8 × 2) LEED pattern is obtained (*cf.* Fig. 1). Ion bombardment is performed with a rastered 4 keV Ar^+ ion beam having average current density of 0.7 μA/cm^2. Ion incidence angle is 50° with reference to the surface normal and the bombarded crystal is kept at room temperature. The argon pressure during sputtering is in low 10^{-6} torr range. Scanning microscopy is performed with Park Scientific Instrument VP2 AFM/STM (atomic force microscopy/scanning tunnelling microscopy) device. All data are collected at room temperature few hours after ion-processing of the surface.

Atomic force images are obtained in a non-contact (NC-AFM) mode with the use of Nanosurf "easyPLL" demodulator or in a contact (C-AFM) mode. Commercially available silicon piezoresistive cantilevers are used as probes. The resonant

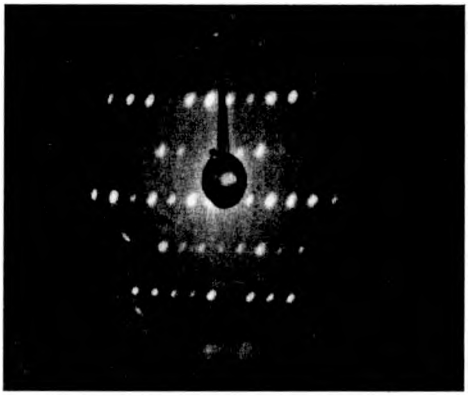

Fig. 1. LEED image of clean c(8 × 2) InSb (001) surface.

frequencies of non-contact cantilevers are typically about 200 kHz. The amplitudes of cantilevers' oscillations used during the measurements are of the order of 40 – 80 nm and detunings are different for different probes, typically between 10 and 50 Hz. Typical force applied in the contact AFM mode is about 70 nN. Scanning rate is 0.5 – 1 scanline per second.

3. Results and discussion

3.1. Initial surface morphology

The InSb (001) surface prepared according to the technique described above is composed of large, atomically flat terraces (of sizes 0.1 – 0.5 μm across) with edges oriented preferentially along ⟨110⟩ and ⟨1 – 10⟩ crystallographic directions (Fig. 2).

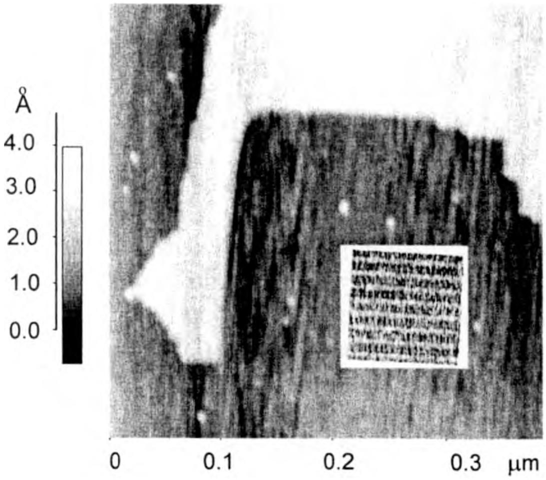

Fig. 2. NC-AFM image of clean (001) InSb surface. In the insert the NC-AFM image with near-atomic resolution reveals the arrangement of the surface atoms into rows of dimers.

LEED pattern characteristic for such a surface (shown in Fig. 1) reveals c(8 × 2) reconstruction. Fourfold reconstruction along the ⟨1 – 10⟩ direction is visible also in NC-AFM images of the surface (see insert in Fig. 2). These results are consistent with the recent model of InSb c(8 × 2) surface reconstruction by KUMPF *et al.* [8] where the surface is basically 4 × 1 and it is composed of atomic rows running along ⟨110⟩ direction differing slightly in height, and only ordering of subsurface dimers produces c(8 × 2) surface symmetry.

3.2. Low ion doses

The NC-AFM image of surface bombarded with an ion dose of $1.5 \times 10^{14}/\mathrm{cm}^2$ is shown in Fig. 3. The bombarded surface consists of terraces which are not atomically flat anymore, although the edges can still be recognized. No nucleation centers at the low doses can be found. As indicated by LEED pattern such a aurface is (almost) completely amorphized (*i.e.*, no diffraction spots are seen).

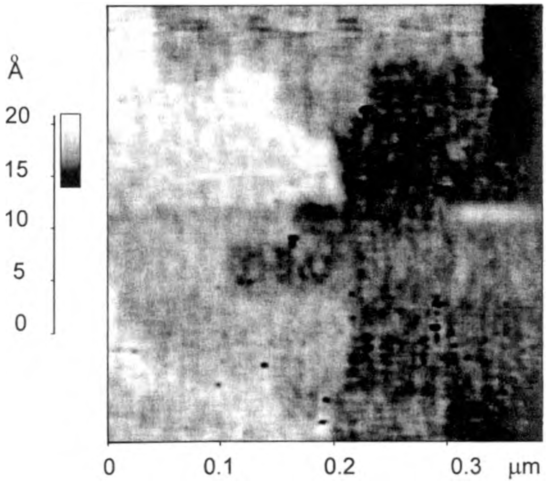

Fig. 3. NC-AFM image of the bombarded InSb(001) surface with Ar^+ dose of $1.5 \times 10^{14}/\mathrm{cm}^2$.

3.3. High ion doses

The 3-dimensional C-AFM image of the surface bombarded with an ion dose of $2.5 \times 10^{16}/\mathrm{cm}^2$ is shown in Fig. 4. The surface is covered predominantly with the system of parallel nanowires. Among the long wires a few small structures like dots are also visible. The nanowires are running along surface projection of the ion beam. The nanowires with typical diameter of 50 – 70 nm are long up to 3 µm. The wires are rather flat as their typical height is about 5 – 7 nm. They are incorporated into a flat (with an average roughness of a few Å) amorphous substrate.

Several aspects of the sputtering process may influence the formation and composition of investigated features, these are:

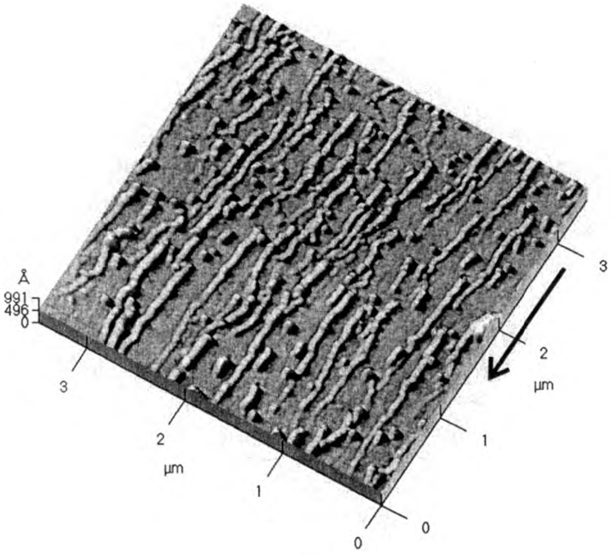

Fig. 4. The 3-dimensional C-AFM image of system of nanowires created on the InSb(001) surface after Ar^+ bombardment with the dose of $2.5 \times 10^{16}/cm^2$. The arrow indicates the projection of the ion beam on the surface.

– *Preferential sputtering.* According to SIGMUND [9] the partial sputtering yields depend on atomic masses and surface binding energies of the surface elements. The theory predicts especially large nonstoichiometry for sputtering of InP (mainly because of large difference in mass for In and P atoms) and ion bombarded InP surface is enriched with indium consistently with the theoretical predictions [10]. For compounds with similar masses of constituents like GaAs or InSb, the theory does not give reliable estimation of partial sputtering yield ratios and, for example, for GaAs Ga enrichment was observed experimentally [11] in contrast to theoretical predictions. Enrichment with metallic element (In droplets) has been reported also for ion-bombarded and annealed (100) InSb surface [12], however it was not sure which one of the two treatments caused the enrichment.

– *Bombardment induced segregation.* As a result of ion bombardment the defects like vacancies and interstitials are generated in large numbers and these are often mobile even at room temperature. Minimization of the surface free energy may drive one of the components to the surface.

– *Beam enhanced diffusion.* Binary collision cascade produces large numbers of adatoms or admolecules on the surface. These particles may become mobile either thermally, or their mobility may be induced by atomic collisions within the cascade, or by primary knock-on process. Fractional order (reconstruction) spots disappear at low ion doses ($< 1 \times 10^{15}/cm^2$) indicating that the strict surface layer is essentially amorphized before the nanowires start to form. Also, at low doses (see Fig. 3), where the surface could retain some of its initial anisotropy, no nucleation centers can be seen. Thus the formation process of the wires must be related to the primary knock-on recoils and direction of the wires is determined by the anisotropy of momentum transferred in knock-on collision of primary ions with surface atoms. In case of developed linear collision cascade the "memory" of the primary ion momentum is lost in multiple collisions and the energy supplied to the surface by "late" collisions atoms cannot be accounted for the formation of anisotropic patterns on amorphous surface. Having in mind the complexity of the sputtering process discussed above we must state that we have not collected sufficient information to determine the composition of the ion-beam formed nanostructures on InSb. They are relatively thin (< 10 nm) and this value is comparable with the penetration length of ions. Therefore the composition of the structure could be altered by sputtering. There are some indications that the wires could have metallic character [12]. Further analyses of the modified samples with high surface chemical sensitivity and at the same time nanometric scale stoichiometric resolution are in progress.

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