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Kinetics of atomic smoothing GaAs(0 0 1) surface in equilibrium conditions

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ABSTRACT

The technique for preparing step-terraced GaAs surfaces by annealing in the conditions close to equilibrium is further developed. The kinetics of GaAs(0 0 1) surface flattening is experimentally measured. The step-terraced surface morphology formation is characterized by the evolution of root mean square roughness, total length of monatomic steps, and by Fourier and correlation analyses. It is shown that the excess of monatomic step length over the value for the ideal vicinal surface is the most adequate characteristic of the surface smoothing because it decreases inversely proportional with annealing duration.

1. Introduction

Atomically smooth crystal surfaces are needed for fundamental surface science, reproducible fabrication of nanoscale structures, and future device applications. A regular technique for preparing flat surfaces of solids is chemo-mechanical polishing (CMP), which can provide very small root mean square (rms) roughness $R_q \sim 0.1$–0.2 nm, comparable with the interatomic distance. However, despite small values of R_q, after CMP the near-surface layer is disordered on the atomic scale due to mechanical action during polishing. This disorder can be effectively reduced by allowing surface migration at elevated temperatures. Thus, by means of annealing, a crystal surface can be brought to an ordered step-terraced morphology, which corresponds to a regular set of atomically flat terraces separated by monatomic steps, with the terrace width determined by the angle of misorientation from the singular crystal face. Almost perfect step-terraced surfaces of silicon are obtained by annealing in vacuum [1,2]. However, the application of this method to III–V semiconductors is hindered by high and different evaporation rates of the III and V components [3,4]. It is generally claimed that an effective method for preparing smooth crystal surfaces is epitaxial growth. However, growth is performed in non-equilibrium conditions, and, therefore, not always yields smooth step-terraced surfaces due to possible kinetic instabilities.

An opportunity to obtain atomically smooth surfaces of III–V semiconductors consists in annealing in the conditions close to equilibrium between the surface and the vapors of the III and V components, when neither growth, nor sublimation take place [5]. Ordered flat surfaces were obtained in Ref. [6] by *in situ* annealing GaAs(0 0 1) epitaxial films at sufficiently high As_4 vapor pressures and moderate temperatures in the growth chamber of a molecular beam epitaxy (MBE) set-up. Annealing GaAs(0 0 1) substrates in the mixture of arsine and hydrogen performed in metal organic chemical vapor deposition (MOCVD) setups may also yield step-terraced surfaces with monatomic steps [7] or step bunches [8]. In another technique of thermal smoothing the equilibrium between the GaAs surface and Ga and As vapors is provided by the presence of saturated Ga–As melt [9]. This technique yields step-terraced surfaces of GaAs(0 0 1) epi-ready substrates and proved to be more efficient and cost-effective as compared to the time- and resource-consuming experiments in the MBE and MOCVD growth chambers.

Although the opportunity to obtain step-terraced GaAs surface by annealing in equilibrium conditions was experimentally demonstrated, a number of important questions related to this method remained open. First, along with the isochronal anneals at various temperatures performed in [9], the kinetics of flattening in equilibrium conditions should be measured in order to open the opportunity of comparing the experimental results with a theory and determining microscopic parameters which govern the flattening process. Second, adequate methods should be developed for characterizing step-terraced surface morphology formation, because the use of the rms roughness is insufficient for the description of this process [3,9]. In the present paper the kinetics of GaAs(0 0 1) thermal smoothing in equilibrium

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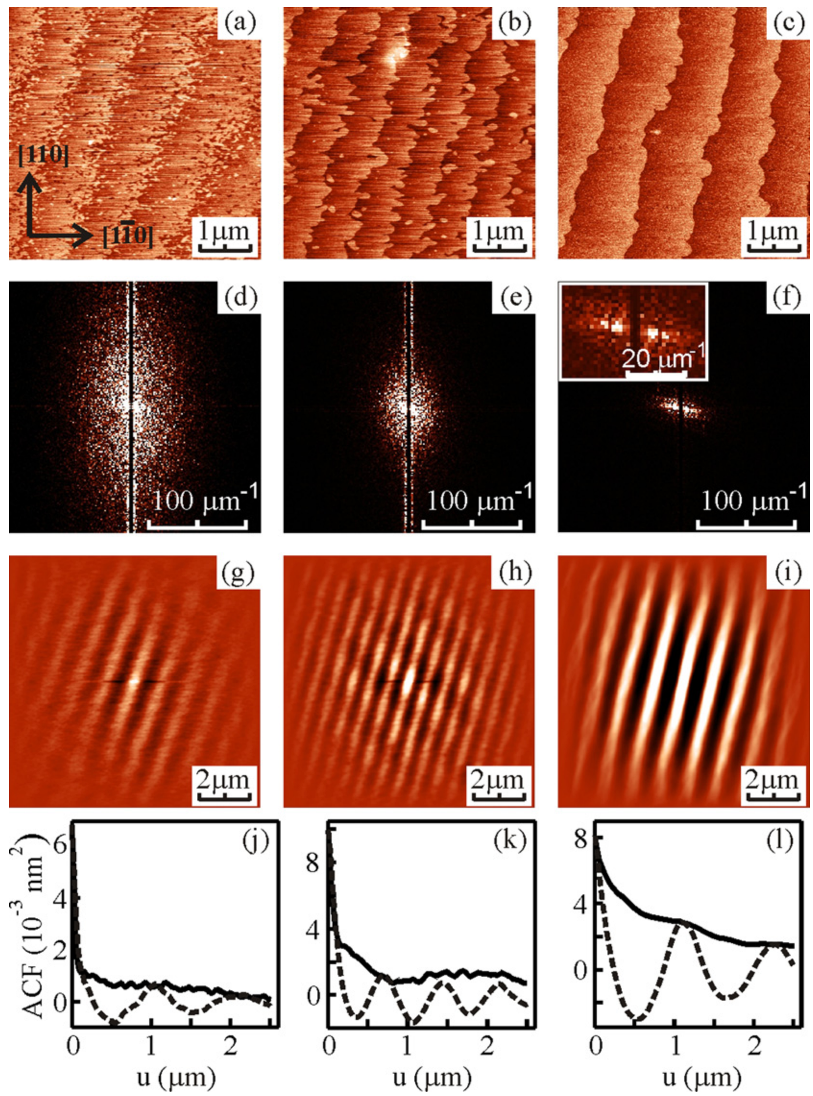

Fig. 1. AFM surface relief images (top row, a–c), their Fourier transforms (second row, d–f), autocorrelation functions (ACFs, third row, g–i), and cross-sections of the ACFs in the directions perpendicular (dashed lines) and parallel (solid lines) to the steps (bottom row, j–l) obtained after anneals at 625 °C for 15 min (a, d, g, j), 40 min (b, e, h, k), and 240 min (c, f, i, l). The inset in (f) shows zoomed image of the FT with two peaks which arise due to the periodicity of the surface steps (color online).

conditions is experimentally studied, and the evolution of the resulting step-terraced morphology of the surface is analyzed by means of two-dimensional Fourier transforms (FTs) and autocorrelation functions (ACFs), along with the rms roughness and length of monatomic steps.

2. Experimental

The samples were cut from “epi-ready” GaAs(0 0 1) substrates with misorientation angles $\theta \leq 0.1^{\circ}$. Prior to annealing, the surface oxides were removed in the solution of HCl in isopropyl alcohol [10]. The anneals were performed at temperatures in the range of T = 400–800 °C in a hermetic quartz tube, under the flow of molecular hydrogen purified by palladium filters. The conditions close to equilibrium between the GaAs surface and arsenic and gallium vapors were provided by the presence of the saturated Ga–As melt in a quasi-closed graphite container. The morphology of the initial and annealed GaAs surfaces was studied *ex situ* by an atomic force microscope (AFM) Solver P-47H (NT-MDT), provided that the oxide layer formed in air replicates the surface relief with submonolayer accuracy. The details of anneals and AFM measurements were described earlier [9,11].

3. Results and discussion

Thermal smoothing and the formation of step-terraced surface morphology at the GaAs(0 0 1) substrates were studied in [9] by means of isochronal anneals in equilibrium condition. It was found that on the initially disordered surface of an “epi-ready” substrate, after 2 h anneal at T = 575 °C, distinct terraces with jagged steps were formed, with a substantial concentration of islands and pits on terraces. Increase of the annealing temperature up to 650 °C led to step smoothing and to decrease of the concentration of islands. Thus, the surface morphology approached that of an “ideal” vicinal surface with a regular set of atomically flat terraces separated by monatomic steps of ≈0.3 nm in height [9].

To understand further the formation of the step-terraced morphology, we studied the kinetics of surface smoothing at a fixed temperature T = 625 °C and various annealing durations in the range from 15 min to 8 h. The top row in Fig. 1 shows AFM images of an epi-ready GaAs(0 0 1) substrate obtained after some of these anneals. It is seen that under increasing duration of annealing the step-terraced morphology is formed with atomically flat terraces of ∼1 μm in width, indicating a misorientation angle of ∼0.02°. The height of steps in Fig. 1b and c is equal to ≈0.3 nm, which coincides