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# Local monitoring of atomic steps on GaAs(001) surface under oxidation, wet removal of oxides and thermal smoothing

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ABSTRACT

The GaAs(001) step-terraced surface relief is studied under oxidation, wet oxide removal and thermal smoothing by *ex situ* atomic force microscopy with local monitoring of specific atomic steps using lithographic marks for surface area localization. Oxidation in the air and wet oxide removal lead to the formation of monatomic dips on terraces, while atomic steps keep their position and shape. Monitoring step mean position under thermal smoothing allows us to determine the deviation from equilibrium. The experimental smoothing kinetics is well described by Monte Carlo simulation.



## 1. Introduction

Smooth semiconductor surfaces are needed for fundamental surface science, device applications and reproducible fabrication of nanoscale structures [1]. Surfaces with regular arrays of smooth terraces separated by atomic steps can be obtained by annealing Si substrates in vacuum [2,3]. The application of this method to GaAs and other III-V semiconductors is impeded by surface roughening due to the preferential evaporation of a more volatile V component [4]. Epitaxial growth may yield smooth step-terraced or rough surfaces, depending on the growth mode and possible kinetic instabilities [5]. In principle, thermal smoothing should be made in the conditions close to the thermodynamic equilibrium, when neither sublimation, nor growth occurs. Ding et al. [6] proved that annealing MBE-grown GaAs(001) films at a sufficiently high $As_4$ vapor pressure and moderate temperature yielded flat surfaces, while, at lower $As_4$ pressures and higher temperatures, the preroughening and roughening transitions occurred. Step-terraced surfaces were also obtained by annealing GaAs(001) substrates in MOCVD setups in the mixture of arsine and hydrogen [7]. Another technique for preparing GaAs surfaces with atomically flat terraces separated by monatomic vicinal steps consists in oxide removal in the HCl solution in isopropyl alcohol (HCl-iPA) and subsequent annealing in a quasi-closed container, in which equilibrium between the GaAs surface and Ga and As vapors is provided by the presence of a saturated Ga-As melt [8]. This thermal smoothing technique has been proved to be efficient and cost-effective as compared to the time- and resource-consuming experiments in the MBE and MOCVD setups [7,6].

Although the opportunity to obtain a step-terraced GaAs surface by annealing in equilibrium conditions was experimentally demonstrated [8], the question remains open: how close are the annealing conditions to equilibrium? In the bulk phase approximation, the two-component (gallium and arsenic), three-phase (solid, liquid, and vapor) system should arrive to the equilibrium state for any given temperature [9]. However, the arsenic vapor departure from the container and a possible influence of surface phases (in particular, As-rich or Ga-rich reconstructions of the GaAs(001) surface [10]) may lead to deviations from the equilibrium towards sublimation or growth. The deviations can be revealed by the motion of vicinal steps towards lower- or higher-lying terraces in the case of step-flow growth or sublimation, respectively.

Direct tracking the movement and shape evolution of individual atomic steps became possible due to development of *in situ* reflection electron microscopy (REM) [11–14] and high-temperature scanning tunneling microscopy (STM) [15,16]). In particular, studying the Brownian motion of steps on Si(111) allowed Bartelt et al.

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