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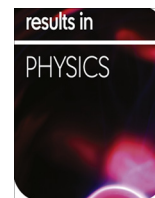
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Microarticle

The $c(4 \times 4)$ – $a(1 \times 3)$ surface reconstruction transition on InSb(001): Static versus dynamic conditions



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ABSTRACT

The transition between the $a(1 \times 3)$ and $c(4 \times 4)$ surface reconstructions of InSb(001) has been carefully monitored by reflection high energy electron diffraction as a function of temperature and Sb_2 flux, without incident In flux. Arrhenius-like behaviour is observed across the whole range of Sb_2 fluxes and temperatures, allowing accurate internal calibration of substrate temperature. This behaviour is in contrast to aggregated data obtained under dynamic molecular beam epitaxy conditions, which show two regimes rather than a single Arrhenius-like phase boundary. The results are explained qualitatively by the atomistic kinetics in static versus dynamic conditions.

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Introduction

The growth by molecular beam epitaxy (MBE) of semiconductor materials is frequently monitored by reflection high energy electron diffraction (RHEED). The III–V semiconductors show a variety of surface reconstructions, easily distinguishable by RHEED, as a function of substrate temperature T_{sub} and V:III incident atomic flux ratio J [1]. When both metal atoms and pnictogen molecules are incident on the surface, the MBE conditions are described as “dynamic” since material growth is normally occurring. However, surface reconstructions can also be altered by adjusting the substrate temperature and pnictogen flux, in static conditions where no growth occurs. Monitoring reconstruction transitions at fixed flux ratio or pnictogen flux can allow an accurate relative surface temperature calibration within a given MBE system [2]. The behaviour of the reconstruction boundaries, often Arrhenius-like, can be used to infer surface chemical behaviour [3,4]. Here we compare static and dynamic conditions for the InSb(001) transition between the $a(1 \times 3)$ and $c(4 \times 4)$ surface reconstructions. The $c(4 \times 4)$ is more Sb-rich [5] and occurs at lower T_{sub} for fixed J . The dynamic case has been studied by several groups and we compile their data sets. The static case provides a surface reconstruction phase boundary over a wide range of Sb fluxes but behaves differently to the dynamic reconstruction change.

Experimental details

Experiments under static conditions were performed in a GEN II MBE system, with a base pressure $< 2 \times 10^{-10}$ torr. The system is equipped with a valved cracker (Addon) supplying Sb_2 flux. A GaAs(001) wafer (AXT) was admitted to the chamber and the oxide thermally desorbed under a stabilising Sb_2 flux. A 500 nm buffer layer of GaSb was deposited, followed which the surface was planarised by the deposition of 500 nm of InSb. The substrate temperature was ramped at a rate of 2 K min^{-1} at different Sb_2 flux values in order to plot the boundary of the $a(1 \times 3)$ and $c(4 \times 4)$ surface reconstructions, as observed by RHEED. The temperature was determined by a thermocouple and the Sb_2 beam equivalent pressure (BEP), F , was measured by an ion gauge and normalised to $F_0 = 2.0 \times 10^{-7}$ torr. Transitions were reasonably abrupt even with the slow temperature ramp, giving an estimated uncertainty of $\pm 5 \text{ K}$.

Results and discussion

The dynamic results are shown in Fig. 1 with data from 5 groups; T_{sub} is converted to inverse absolute temperature $10^3/T_{\text{abs}}$. Absolute quantitative comparison of the data sets is not possible because: (1) offsets in the absolute value of T_{sub} are likely to occur between different MBE systems, and (2) the calibration of flux ratio J may differ amongst the groups. Oe et al. used atomic arrival rates $J = F_{\text{In}}/F_{\text{Sb}}$ calibrated by mass spectrometry and quartz microbalance [6]. Noreika et al. measured their atomic flux ratio by beam equivalent pressure adjusted for ionisation efficiency [7]. Liu

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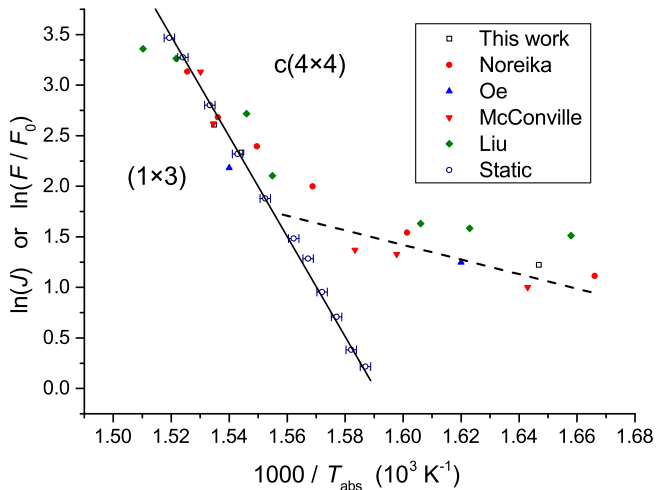


Fig. 1. Surface reconstruction transition temperature for $a(1 \times 3) \leftrightarrow c(4 \times 4)$ from several groups. Also shown is the same transition under static conditions (open circles with error bar). The flux ratio is J for dynamic conditions (coloured solid symbols and \square) or F/F_0 for the static case. Lines are guides to the eye. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

and Santos reported $J = F_{\text{In}}/F_{\text{Sb4}}$ inferred from BEP measurements [8], as did we. By scaling from Sb_4 to Sb atom ratio a reasonable comparison can be made. McConville et al. did not specify the flux calibration method [9], but their raw data using $J = F_{\text{In}}/F_{\text{Sb}}$ sit well in the aggregated plot.

Even bearing in mind potential temperature offsets and flux calibration differences, it seems clear that the aggregate data are better described by two regimes separated at about $T_{\text{sub}} = 365^\circ\text{C}$. A single fixed activation energy with Arrhenius behaviour is not consistent with the dynamic data. At high temperatures the slope is higher than in the low temperature regime. In contrast, the static data are linear over the whole range, giving an effective activation energy of 4.14 ± 0.06 eV. (The vertical offset of the static line depends on the normalisation factor F_0 of the Sb flux F .)

Chatillon et al. [3] made a direct comparison between static and dynamic conditions for an As-stable (1×3) to Ga-stable (4×2) transition on $\text{GaAs}(001)$, with a similarly slow temperature ramp to our experiment (0.3 K s^{-1}). Similarly to the present results, they saw close to straight-line behaviour for the static case but a strong reduction of gradient at lower T_{sub} for dynamic conditions. Rather than modelling the process by one or two activation energies, they solved thermodynamic and mass balance equations for GaAs and successfully explained the transition from an As-stable to Ga-stable surface in framework of GaAs bulk stability (with the Ga-stable surface occurring at the limit of liquid Ga droplet formation). Even though the present situation is different, careful observation of the reconstruction transitions can clearly reveal

behaviour more complicated than simple Arrhenius dependence of J with T_{sub} . The $\text{InSb}(001) a(1 \times 3) \leftrightarrow c(4 \times 4)$ transition involves a re-arrangement of Sb dimers on an Sb -terminated surface. Unlike the $\text{GaAs}(001) (2 \times 4) \leftrightarrow c(4 \times 4)$ transition, for example, there is no need for metal atoms to migrate [10]. Under dynamic conditions (epitaxial growth) incoming In atoms must be incorporated into the growth front as the reconstruction transition occurs. Conversely, in the static case the Sb rearrangement can take place without significant movement amongst the third layer In atoms. Hence we interpret the behaviour in the static case as a single free energy associated with the re-arrangement of Sb dimers on the Sb -terminated surface. In the dynamic case, at least two processes must occur: the incorporation of In through the $\text{Sb} + \text{Sb}$ dimer double layer and re-arrangement of the top-layer dimers. Hence the dynamic and static phase boundaries differ qualitatively.

Conclusions

In conclusion, we report a careful measurement of the $\text{InSb}(001) a(1 \times 3) \leftrightarrow c(4 \times 4)$ surface reconstruction transition by RHEED in static conditions (no In flux) which can allow accurate internal calibration of an MBE system in the temperature range $345\text{--}375^\circ\text{C}$. A simple Arrhenius-like transition boundary is found. Aggregate data on this transition under dynamic growth conditions reveal more complicated behaviour which can be ascribed to more than one atomistic process when In incorporation must be accounted for.

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