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Structure of the InAlAs/InP interface by atomically resolved energy dispersive spectroscopy

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The structure of epitaxially grown InAlAs/InP interfaces was studied using atomically resolved x-ray energy dispersive spectroscopy in scanning transmission electron microscopy. As and P sublattices show sharp termination on the interface. The In sublattice is continuous across the interface. The study has shown the depletion of the Al concentration at the interface; at the last atomic columns of the InAlAs, In occupancy is close to 100%, while Al occupancy is almost zero. A monolayer of InAs at the interface is consistent with substitution of As for P at the surface preceding growth. © 2011 American Institute of Physics. [doi:10.1063/1.3645632]

InP-based III-V materials are technologically important in high-speed transistors and optoelectronics. Frequently, a buffer layer of lattice-matched $\text{In}_{0.52}\text{Al}_{0.48}\text{As}$ is used as the interface layer between the InP substrate and $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ materials. It has previously been shown that this interface enables high-quality epitaxial growth, and it has been posited previously that a thin layer of InAs can be found at this interface.¹ In this letter, the composition of the InAlAs/InP interface is studied at the atomic scale using atomically resolved energy dispersive spectroscopy (EDS) in scanning transmission electron microscopy (STEM).

It has been shown previously that it is possible to use atomically resolved EDS to obtain the direct elemental composition of atomic columns in crystalline materials.² However, the application of EDS to study the atomic structure of interfaces was blocked by the high sensitivity of interfaces to damage by the electron beam. The low collection angles of conventional, side-mounted, EDS detectors require relatively long dwell times which results in damage to the interfaces long before the atomic structure can be obtained. The increase of the probe current density by using Cs correctors makes the situation worse. Greatly increased EDS detection sensitivity with the introduction of the quadruple windowless detectors integrated into the column^{3,4} allows these limitations to be overcome. The increased sensitivity in combination with the use of very short pixel dwell times with iterative mapping allows maximum EDS signal collection while minimizing damage during acquisition by the high current of the probe corrected beam.⁵ By using a lower acceleration voltage, such as 80 kV, it is possible to harvest the full power of the probe corrector and high-brightness X-FEG source for the analysis of the atomic structure of interfaces, since damage is decreased while x-ray cross-section is increased at lower voltages. This current work, which shows the atomic structure of the InAlAs/InP, represents atomically resolved EDS results from an interface.

The sample being studied was grown by molecular beam epitaxy in a Varian Mod Gen II system. The oxide was desorbed by rapidly increasing the sample temperature to 570 °C (as measured using pyrometry) under a large As_2 overpressure obtained from a valved cracker effusion cell. The As_2 beam equivalent pressure was 10^{-5} Torr. Oxide desorption was confirmed by the presence of a 2×4 pattern in reflection high-energy electron diffraction (RHEED), and the substrate temperature was reduced to 490 °C. The InAlAs studied here was smooth as determined by RHEED.

The interface was studied using an FEI Titan G2 60-300 S/TEM equipped with a Cs probe corrector and ChemiSTEM technology (high-brightness X-FEG electron source in combination with the Super-X EDS detector, which consists of 4 windowless silicon drift detectors⁴). In order to minimize damage to the interface area, the maps were obtained at 80 kV with the beam size less than 0.14 nm and beam current of about 25 pA. Iterative maps of 128×128 pixels were recorded with a dwell time per pixel of 50 μs . The sample was prepared by conventional technique using tripod polishing followed by ion milling at 5 kV.

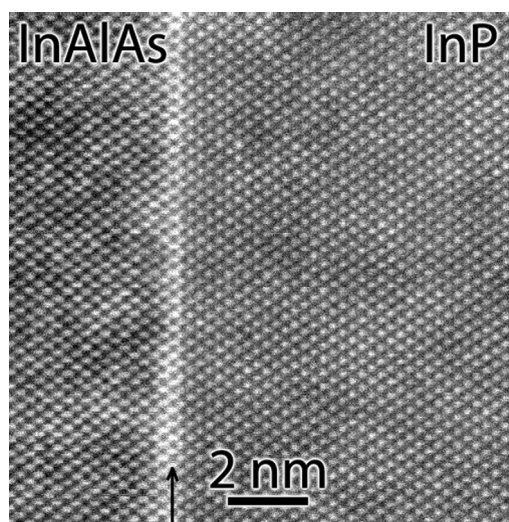


FIG. 1. HAADF-STEM image of the InAlAs/InP interface showing higher contrast at the interface (arrow).

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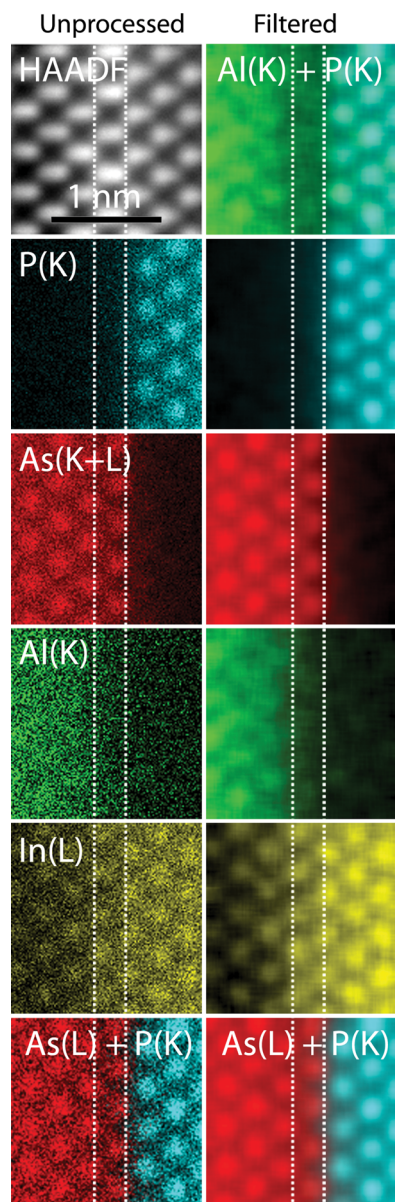


FIG. 2. (Color online) HAADF-STEM image and corresponding atomically resolved EDS maps of the InAlAs/InP interface. The elemental distribution is obtained with the integrated intensities of the corresponding energy. Left column shows acquired maps without any processing, except correction of the background in the final images. Right column shows the same elemental maps with applied averaging over 11 pixels in order to enhance signal of Al and In atomic positions. Dotted lines highlight interface region where Al is depleted.

Figure 1 shows a high angle annular dark field scanning transmission electron microscopy (HAADF-STEM) image of the InAlAs/InP interface. The dumbbells in the image are not well-resolved, partially due to an amorphous layer on the surface of the sample resulting from the sample preparation. The HAADF-STEM shows the increase of the contrast in the atomic columns next to the interface. In this case, the structure of the interface is difficult to resolve by the contrast interpretation in the images⁶ since some of the columns contain a mixture of atoms. The contrast could be attributed to an enrichment of indium. However, from the images it is not possible to identify where the Al atoms are and how crystals actually terminate on the interface. Consequently, atomically resolved EDS maps were acquired from different regions of the interface.

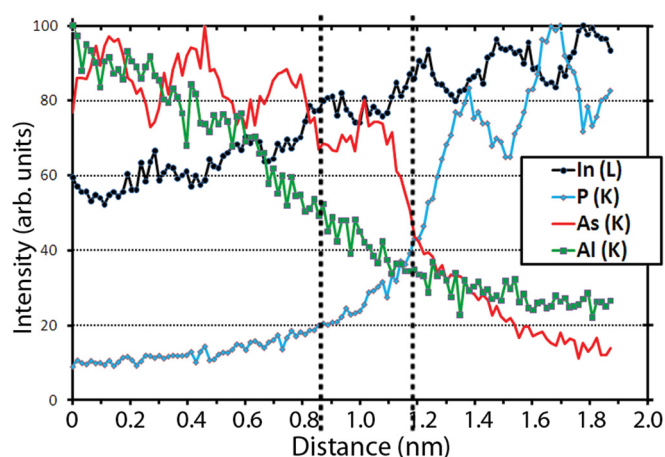


FIG. 3. (Color online) Line profile across the interface averaged over the whole height of the map. The Al concentration clearly decreases two atomic planes before the InP. On the other hand, the In concentration is as high in first atomic plane as inside the InP. The second plane shows slight decrease in the peak height gradually decreasing further into InAlAs.

Figure 2 shows examples of the atomically resolved EDS map from the interface region. It is important to note that both sublattices of the InP are resolved, unlike in the HAADF-STEM image, where phosphorous does not produce strong scattering, and as a result its contrast is very weak compared to indium, making it difficult to recognize unambiguously. In the EDS map, however, the P positions are clearly identified, allowing direct observation of the crystal polarity. Both As and P positions are clearly resolved. The In positions also can be identified directly from the integrated intensities map. The Al signal is the weakest, but it is still possible to identify atomic positions, particularly if signal averaging is used (right column in Figure 2). Consequently, the atomic structure of the interface can be directly established from the EDS map.

As expected, the In sublattice is continuous across the interface. The As and P sublattices reveal sharp terminations. The Al sublattice is not as well resolved as the As and P. This is caused by the lower concentration of the Al in the [110] columns. However, it is clearly visible that the Al concentration is very low in the first atomic plane of the InAlAs. The Al contrast in the In/Al sites is very low in the first two planes from the interface compared to those further into the film. In order to distinguish the Al distribution better, the signal was averaged over 11 pixels (Figure 2, right column). This is possible because of the high spatial resolution of the initial map (about 0.015 nm per pixel). The processed maps reveal the positions of the Al and confirm its lower concentration at the interface. Accordingly, the In shows higher concentration on the interface plane than in the InAlAs film. The maps show that In should occupy columns completely, similar to those in the InP. This is also confirmed by the line profile extracted from the maps and integrated across the whole map height (Figure 3).

The quantification of the Al concentration in the corresponding atomic columns is complicated due to channeling.^{7–10} However, the line profile in Figure 3 shows that a small amount of Al might still be present in the last atomic columns at the interface. Spectra integrated from single atomic columns at the

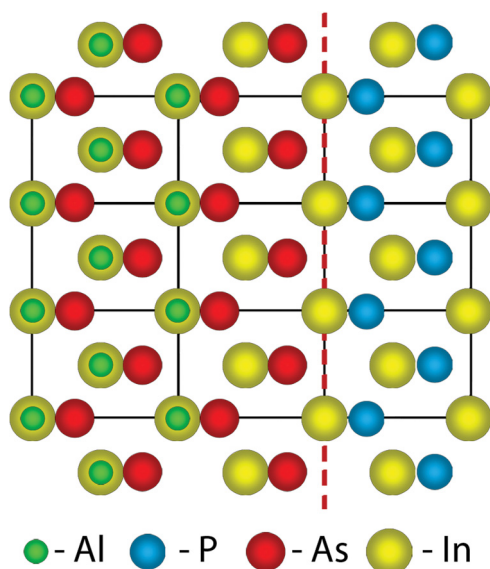


FIG. 4. (Color online) Corresponding schematic of the structure of the InAlAs/InP interface.

interface show no Al signal above background which is evidence of the depletion of Al at the interface to below detection limits. Based on the EDS results and HAADF-STEM image, the final structure of the interface is shown in Figure 4.

The presence of a monolayer of InAs at the InAlAs/InP interface can be explained by considering the kinetics of the growth process. Immediately following oxide desorption, a P-terminated InP surface is exposed to a large arsenic overpressure. Because the bond enthalpies of InP and InAs are nearly identical (197.9 ± 8.4 kJ/mol and 201 kJ/mol, respectively),¹¹ the relative abundance of the two species at the interface will cause the substitution of the more-abundant arsenic for the less-abundant phosphorous, resulting in the

InAs monolayer which is consistent with the experimental observations reported here.

In summary, a high sensitivity, multi-detector EDS system has been applied as a technique for directly determining the chemistry of the epitaxial InAlAs/InP interface with atomic resolution. A monolayer of InAs was found at the interface, which can be readily explained by considering the kinetics of the growth process. Additionally, this work demonstrates that the InAlAs/InP interface is atomically smooth and well-controlled, which enables the growth of high quality (opto)electronic devices on InP.

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