Cross-sectional scanning tunneling microscopy study of GaAs/AIAs short period superlattices: The influence of growth interrupt on the interfacial structure

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We report studies of GaAs/AlAs short period superlattices using cross-sectional scanning tunneling microscopy. In particular, we investigate the role of growth interrupt time on the resulting interfacial structure. Superlattices with repeated periods of four layers of GaAs and two layers of AlAs are resolved atom by atom. Superlattices grown using a 30 s growth interrupt time are observed while those grown with a 5 s growth interrupt time are not. We also discuss residual effects of the growth interrupt process on layers grown on top of the short-period superlattice. © *1995 American Institute of Physics*.

Short period superlattices are a new class of electronic devices with highly unusual properties, making them potentially useful for future electronic devices. Due to the extremely small barrier thicknesses within the superlattice, the electronic state amplitudes within the individual quantum wells have considerable overlap, resulting in novel electronic properties.^{1,2} It should be obvious that for such atomic-scaleengineered devices, controllability of the atomic structure at the heterojunction interfaces will play the most critical role. Much time and effort has been spent in the attempt to characterize the layer composition and interface quality. While cross-sectional transmission electron microscopy (XTEM) has in the past provided very useful interfacial structural information by averaging over the sample thickness (a columnar average), cross-sectional scanning tunneling microscopy (XSTM) has recently made it possible to obtain atomically resolved structural information by probing only a single atomic layer.^{3–12} In this letter, we utilize the tool of XSTM to investigate the role of growth interrupt time on the resulting structural quality of heterointerfaces, particularly in the case of short period superlattices.

We perform our experiments in a vacuum chamber with a base pressure of less than 4×10^{-11} Torr. Polycrystalline W tips are electrochemically etched and loaded into the vacuum chamber. They are then cleaned in situ using a field emission technique on separate clean substrates. Samples studied are MBE grown p type at 10^{19} cm⁻³ [Be] on p-type GaAs(001) substrates at 580 °C. Two different kinds of short period superlattices were grown, namely (a) two unit cell lengths of GaAs (11.3 Å) followed by one unit cell length of AlAs (5.66 Å), repeated ten times for a total length of 171 Å, and (b) four unit cell lengths of GaAs (22.6 Å) followed by two unit cell lengths of AlAs (11.3 Å), repeated ten times for a total length of 363 Å (note that each unit cell length contains two bilayers). Each of these superlattices was grown twice, once using a 5 s growth interrupt time and once using a 30 s growth interrupt time. To be specific, the growth interrupt is

imposed at each interface of the superlattice, that is, when changing from GaAs to AlAs or vice versa. Thus, altogether, four unique superlattices were grown, separated from each other by 500 Å of $Al_{0.3}Ga_{0.7}As$ for a total thickness of 2568 Å. Finally, a 500 Å $Al_{0.3}Ga_{0.7}As$ layer was grown with a 2000 Å GaAs layer on top. All of these layers, including the short period superlattices were grown on top of a 6 μ m thick region of alternating 150 Å GaAs/150 Å $Al_{0.3}Ga_{0.7}As$ heterostructures. These heterostructures were grown *without* the growth interrupt method. The overall device structure looked like that shown in Fig. 1(a).

Before we discuss the results on the superlattice layers, we first show a typical interface structure for one of the 150 Å AlGaAs layers which was grown without the use of growth interrupt. Figure 1(b) is an atomic resolution image of a 150 Å region of AlGaAs sandwiched on both sides by GaAs. The total image size is 203 Å×269 Å and was acquired with a sample bias of -2.13 V and a tunneling current of 0.29 nA. In this image, the AlGaAs region shows a mixture of light and dark atomic features, and the dark ones appear to preferentially order themselves along diagonal directions in the image, similar to that observed by Johnson.⁴ This may be attributed to alloy ordering, the details of which will be discussed elsewhere.¹² Shown below in part (c) of the same figure is a cross line cut from the point A to the point A' marked in the image. The atomic corrugation stands out quite clearly with an amplitude of about 0.2 Å. The depth of the dark features in the AlGaAs region varies quite a lot, reflecting local variations in AlAs content.

It is important to note the amount of interfacial roughness shown in this 150 Å region both for the normal (AlGaAs grown on top of GaAs) interface and also for the inverted (GaAs grown on top of AlGaAs) interface. Typical of images we regularly obtain on these regions, the normal interface is sharper than the inverted interface by about two lattice constants. The extent of the roughness agrees with what we have reported earlier^{9,10} for both the UHV-cleaved surface and also for the sulfide-passivated surface (deduced from the tunneling spectroscopy), in this case about 4-5

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FIG. 1. (a) Planned device structure for this investigation. (b) 203 Å \times 269 Å atomic resolution image of Al_{0.3}Ga_{0.7}As region with GaAs regions on either side. The sample bias was -2.13 V, and the tunneling current was 0.29 nA. The interface on the right appears sharper than the one on the left by one to two atomic rows. (I) indicates "inverted" interface, (N) indicates "noninverted" interface. (c) cross line cut taken between the points labeled A and A' in the image of part (b). Atomic corrugation is about 0.2 Å while the AlGaAs region shows dark features up to 1.3 Å in apparent depth, reflecting local variations in AlAs content.

atomic layers for the inverted interface and 2-3 atomic layers for the normal interface. Obviously, such a large amount of interfacial roughness at the atomic scale would make it extremely difficult to successfully grow a short period superlattice where the periodicity is on the same length scale as the amount of roughness. This directly implies the need for the growth interrupt technique which is intended to result in smoother growth surfaces and hence, sharper interfaces.

Shown in Fig. 2(a) is a 450 Å \times 290 Å atomicresolution STM image of the 22.6 Å GaAs/11.3 Å AlAs (4/2) superlattice acquired at a sample bias of -2.25 V and a tunneling current of 0.2 nA. This superlattice region was grown with the 30 s growth-interrupt time. The four layers of GaAs are clearly resolved in this filled state image as lightershaded atomic rows running along the vertical direction. The two AlAs layers appear as the two darker rows. Mixed bonding of GaAs and AlAs within a single atomic row is also evident. On the left, one can see the beginning of the 500 Å Al_{0.3}Ga_{0.7}As region. Shown in Fig. 2(b) is a cross line cut through the image shown in part (a) from point B to point B'. The GaAs regions show an atomic corrugation of about



FIG. 2. (a) 450 Å \times 290 Å constant-current STM image of 23 Å GaAs/11 Å AlAs superlattice region acquired at a sample bias of -2.25 V and a tunneling current of 0.2 nA. In this image, the GaAs regions show up as light in comparison with the AlAs regions with a total gray scale of about 1.5 Å. On the left, following the last 23 Å GaAs region, is a region of Al_{0.3}Ga_{0.7}As. (b) Line cut across the image in (a) from point B to point B'. As seen, the height difference between GaAs and AlAs is typically about 1 Å with atomic corrugation of about 0.15 Å. (c) 450 Å \times 150 Å constant-current mosaic STM image of superlattice region grown with 5 s growth interrupt. No short periods are observed.

0.15 Å, and the 11.3 Å AlAs regions are lower than the GaAs regions by about 1 Å.

We have not observed the 22.6 Å GaAs/11.3 Å AlAs (4/2) superlattice which was grown with the 5 s growth interrupt time. This is seen clearly in Fig. 2(c) which is an atomically resolved mosaic STM image of the region where we should see this short period superlattice. However, it appears virtually indistinguishable from an AlGaAs alloy region. This result implies that such a short amount of interrupt time may be insufficient in order to obtain a sharp enough interface to observe at the atomic level.

We have also not observed the 11.3 Å GaAs/5.66 Å AlAs (2/1) superlattice in our studies for either amount of growth interrupt time. Based on the amount of interfacial roughness evident in the image of Fig. 2(a) for the larger period (4/2) superlattice, this may come as no surprise since the roughness of the interfaces may wipe out the resolution of such short periods at the atomic scale. However, this does not imply that these layers cannot be delineated with XTEM which performs a columnar average over the sample thickness and may therefore be able to average over atomic scale fluctuations. Conversely, even when one observes a sharp contrast between layers using XTEM, it does not necessarily imply that the interfaces are atomically sharp. Furthermore,



316 Å x 316 Å

FIG. 3. 316 Å \times 316 Å constant-current STM image of the heterojunction between the last 500 Å Al_{0.3}Ga_{0.7}As region and the 2000 Å GaAs region. Tunneling parameters were typical: sample bias -2.14 V, tunneling current 0.27 nA. The dark spots in the GaAs region appear to be vacancies. The interfacial sharpness of this inverted interface is quite high, having a variation of only about 1–2 atomic layers.

the lack of an atomically sharp interface will very likely have an important influence on the electronic properties of these short period superlattices.¹

We have found that the growth interrupt performed on the short period superlattices appears to have a residual effect on layers grown up to 500 Å after the end of the last growth interrupt. Figure 3 is an atomic resolution image of the interfacial region where the 2000 Å layer of GaAs is grown on top of the final 500 Å layer of AlGaAs, as indicated in Fig. 1(a). Note that the AlGaAs layer was not grown using growth interrupt, nor was the 2000 Å GaAs layer. What appears somewhat unexpected is the sharpness of this inverted GaAs/AlGaAs interface. As we have mentioned previously, the inverted interface for GaAs/AlGaAs usually has 4-5 atomic layers of roughness. In this case, the interface has at most two atomic layers of roughness. We suspect that the influence of the growth interrupt may be responsible for the improvement in interface quality. This implies that the sharpness of the interface depends on how flat the surface is on which the layers are grown. Since growth interrupt produces a very flat surface as indicated by our observation of the short period superlattice, layers grown on top of them will also be relatively flat.

In conclusion, we have studied the influence of growth interrupt on the resulting interfacial structure of GaAs/AlAs short period superlattices grown with molecular beam epitaxy. We observe that an increased amount of growth interrupt time does indeed make a difference in the interfacial sharpness. In particular, for 5 s of growth interrupt, we do not observe the short period (4/2) superlattice while for 30 s of growth interrupt, we do observe it. In addition, we have not observed the even shorter period (2/1) superlattices in any of our investigations. We have also found that the GaAs/AlGaAs inverted interface grown on top of the superlattice is sharper than typical GaAs/AlGaAs inverted interfaces by about two atomic layers. We attribute this to the residual effect of the growth interrupt. More detailed work is necessary to firmly establish this fact.

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