

Interface Roughness and Asymmetry in InAs/GaSb Superlattices Studied by Scanning Tunneling Microscopy

R. M. Feenstra

IBM Research Division, T. J. Watson Research Center, Yorktown Heights, New York 10598

D. A. Collins, D. Z.-Y. Ting, M. W. Wang, and T. C. McGill

T. J. Watson Sr. Laboratory of Applied Physics, California Institute of Technology, Pasadena, California 91125

(Received 15 December 1993)

InAs/GaSb superlattices are studied in cross section by scanning tunneling microscopy and spectroscopy. Electron subbands in 42 Å thick InAs layers are clearly resolved in the spectra. Roughness of the superlattice interfaces is quantitatively measured. Interfaces of InAs grown on GaSb are found to be rougher, with different electronic properties, than those of GaSb on InAs, indicating some intermixing in the former case.

PACS numbers: 68.35.Fx, 61.16.Ch, 68.65.+g, 73.20.Dx

The scanning tunneling microscope (STM) has been applied in recent years to the study of III-V semiconductor superlattices, viewed in cross section on a (110) cleavage face [1,2]. Such studies can determine structural and electronic properties of the superlattice interfaces, which have significant consequences for device performance. In this work we focus on two interface properties: first, roughness due to atomic steps at an interface, and second, grading or intermixing of the constituent materials between superlattice layers. We study superlattices composed of alternating layers of InAs and GaSb. This system forms a type II superlattice with electrons confined in the InAs and holes in the GaSb [3-5]. Since the identity of cation and anion both change across a heterointerface, the interfacial bonding can be InSb-like (As-In-Sb-Ga), GaAs-like (In-As-Ga-Sb), or mixed, depending on the growth conditions [3-6]. We observe, for the first time in STM, electron subbands resolved in the tunneling spectra of the quantum wells. Furthermore, we obtain from the images of spectrum of interface roughness, with detail surpassing that previously measured for any system. Finally, we find clear differences between the interface properties of InAs grown on GaSb compared with GaSb on InAs, which we attribute to intermixing at the InAs on GaSb interfaces.

The InAs/GaSb superlattices were grown on GaSb substrates using a Perkin-Elmer 430 molecular-beam epitaxy system, at a growth temperature of $\sim 380^\circ\text{C}$. The growth surface was soaked in either an Sb_2 or As_2 flux for 5 s at the termination of each InAs and GaSb layer, and similar STM results are obtained in both cases. The superlattices are Si doped at concentrations of $3 \times 10^{17} \text{ cm}^{-3}$ p type in GaSb and $3 \times 10^{17} \text{ cm}^{-3}$ n type in InAs. Three wafers have been studied, with superlattice periods typically of 42 Å InAs and 24 Å GaSb, and a total growth of 30-60 periods. Samples cut from the wafers were cleaved *in situ*, at 4×10^{-11} Torr. Single crystal (111) oriented tungsten probe tips were prepared by electrochemical etching and *in situ* electron beam heating, and were characterized *in situ* by field-emission micros-

copy. STM images were acquired with a constant current of 0.1 nA and at various voltages specified below. Details of the STM spectroscopic methods have been previously given [7]. Spectra from bulk materials were obtained using $2 \times 10^{17} \text{ cm}^{-3}$ p -type GaSb and $9 \times 10^{17} \text{ cm}^{-3}$ n -type InAs crystals.

In Fig. 1(a) we display an STM image obtained from the InAs/GaSb superlattice. For filled state images such as this, the GaSb layers are bright and InAs layers dark (as established by spectroscopy, below, or by imaging the adjoining GaSb substrate). Faint fringes, with spacing of 6.0 Å (two bilayers), arise from the atomic planes in the superlattice. The interface between InAs and GaSb layers can be more clearly defined by expanding the grey scale of the image and taking a derivative in the horizontal direction, as shown in Fig. 1(b). Interface roughness, with step heights of 3-6 Å, is clearly visible there. We see that the interfaces of GaSb grown on InAs have relatively long flat sections, whereas the InAs on GaSb interfaces have considerably more steps. From these interface profiles we compute the spectrum of interface roughness, as shown in Fig. 1(c) for the two types of interfaces. We fit the spectra to a Lorentzian form [8], yielding roughness amplitudes (Δ) and correlation lengths (Λ) as shown in Fig. 1(c). The roughness amplitudes for the two types of interfaces are comparable, but the correlations lengths for InAs grown on GaSb are consistently 2-3 times less than for GaSb on InAs, thus demonstrating that the former are rougher.

The voltage dependence of the STM images is shown in Fig. 2, where images (a)-(c) were acquired consecutively on the same surface region. The filled state images, Figs. 2(a) and 2(c), are similar to Fig. 1(a). The empty state images, Fig. 2(b), appears much different, with an enhanced signal (greater state density) at the InAs on GaSb interfaces compared with GaSb on InAs. (According to the spatially resolved spectroscopy below, the filled state images do indeed coincide with the physical superlattice structure.) The asymmetry seen between the two types of interfaces in Fig. 2 demonstrates that there is

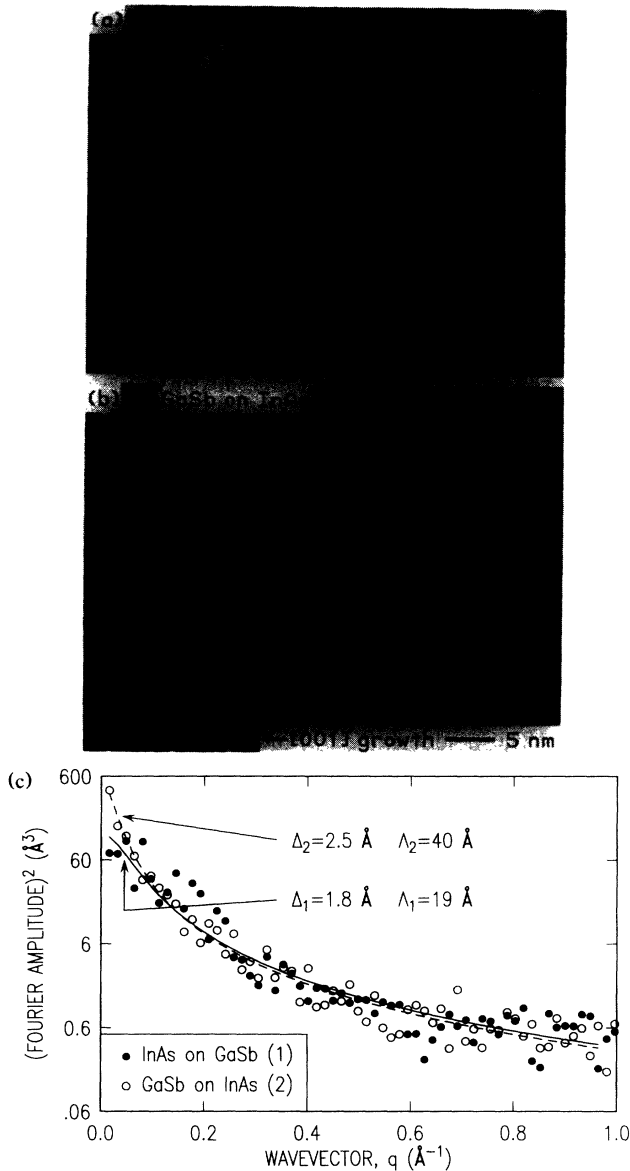


FIG. 1. (a) Constant-current STM image of InAs/GaSb superlattice, consisting of 42 Å thick InAs and 24 Å thick GaSb layers. Image was acquired at a sample voltage of -1.5 V. The [001] growth direction is indicated. Grey-scale range is 2.0 Å. (b) Derivative of (a). (c) Interface roughness spectra, computed on the 400 Å long interfaces of (b), using seven interfaces of each type and averaging the results. The data from each interface type are fit to a Lorentzian form, yielding results shown for the roughness amplitudes (Δ) and correlation lengths (Λ).

some significant structural difference between them, consistent with the extra roughness for InAs on GaSb seen above. To further explore this asymmetry, we perform detailed spectroscopic measurements.

As an introduction to the spectroscopy, we first examine typical spectra acquired near the center of the superlattice layers compared with those obtained from bulk InAs and GaSb, as shown in Fig. 3. The bulk spectra are

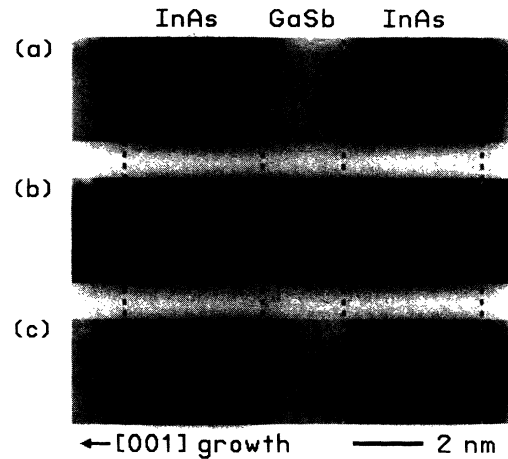


FIG. 2. Constant-current STM images, acquired at sample voltages of (a) -1.3 , (b) $+1.3$, and (c) -1.3 V. Grey-scale ranges are (a) 1.4, (b) 0.8, and (c) 1.4 Å. Dashed lines indicate the position of the interfaces between InAs and GaSb.

part of a larger study, including detailed spectroscopy of InSb, InP, and GaAs, which will be presented in its entirety elsewhere [9]. Spectral features are indicated in Fig. 3, where the energetic positions are determined using peak locations for surface states, and assuming linear onsets for the bulk bands, with a precision of ± 0.03 eV. Possible systematic errors due to tip-induced band bending could result in positions which are too large by 0.1–0.2 eV [9,10]. For bulk GaSb, Fig. 3(a), we find a band gap of 0.78 eV (compared with the known value of 0.72 eV) and surface state locations in good agreement with inverse photoemission results [11]. For bulk InAs, we find a band gap of 0.35 eV and the L -valley onset located 1.18 eV above the Γ -valley minimum (compared with known values of 0.36 and 1.08 eV, respectively). Turning now to the superlattice spectra, Figs. 3(c) and 3(d), we see some new features. First, significant conductance is observed within the apparent band gap regions. Based on the intensity and spatial dependence of this feature, we interpret it as arising from electron (hole) states tailing out from neighboring InAs (GaSb) layers. Second, we find that the apparent band gaps are slightly larger for the superlattice compared with the bulk, with observed gaps of 0.82 and 0.62 eV for the GaSb and InAs layers, respectively. We attribute the significantly larger gap for InAs to confinement effects in the quantum well (this effect is small for GaSb due to the much larger heavy hole mass); subtracting the known InAs gap yields an energy for the first electron subband of 0.26 eV. The third feature seen in the superlattice spectra is an additional onset at 0.51 V, which we attribute to the second electron subband. Relative to the observed InAs valence-band edge, it is located at 1.18 eV, and subtracting the InAs bulk gap yields an energy of 0.82 eV. These subband energies (0.26 and 0.82 eV) are in reasonable agreement with theoretical estimates based on an eight-

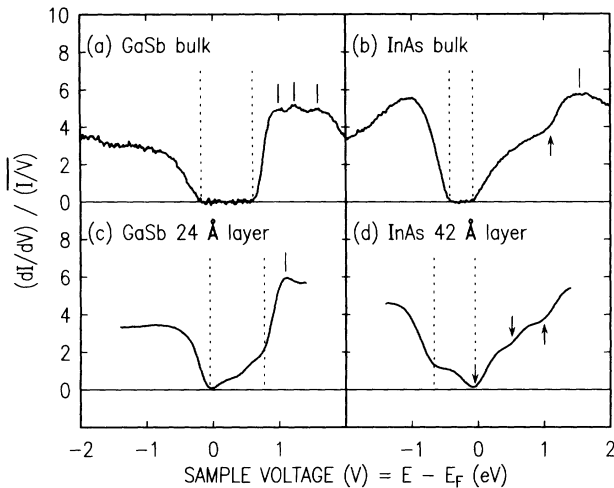


FIG. 3. Typical tunneling spectra, acquired from (a) bulk GaSb, (b) bulk InAs, (c) GaSb superlattice layer, and (d) InAs superlattice layer. Apparent band gaps are indicated by dashed lines, surface states by tic marks, and *L*-valley conduction band onsets by upward pointing arrows. Downward pointing arrows indicate subband onset energies in panel (d).

band tight binding model [12] of 0.21 and 0.77 eV, thus confirming the above identification.

Spatially resolved spectroscopy results are displayed in Fig. 4. The STM image shown there was acquired in two halves; the bottom part first, followed by a set of spectra measuring each 1.06 Å along the white line, followed by the top half of the image. Correspondence between the upper and lower halves of the image demonstrate the absence of STM drift during acquisition of the spectra. Six spectra are selected for display in Figs. 4(a)–4(f). The spectra acquired near the center of the superlattice layers, Figs. 4(a), 4(c), 4(d), and 4(f), appear similar to those in Figs. 3(c) and (d). At the interfaces, Figs. 4(b) and 4(e), we observe an enhanced intensity of the conductance near ± 0.5 V discussed above, consistent with its interpretation as arising from wave-function tails from adjoining layers. The apparent band gap is now marked as the minimum gap between empty and filled states (i.e., the superlattice band gap), observed to be 0.1–0.2 eV (compared with the theoretical value of 0.15 eV). Closely inspecting the interface spectra, Figs. 4(b) and 4(e), we find two differences between them: (1) For InAs on GaSb, the spectrum is smeared out, especially on the valence-band side, and (2) the normalized conductance is slightly higher at low positive voltage for InAs on GaSb. This latter feature leads to the enhanced empty state density seen for that interface in Fig. 2. Both of these features have been reproducibly seen in about twenty data sets acquired from two samples each studied with a different probe tip.

The above observation of spectral smearing at the InAs on GaSb interface is found to persist over a distance of ~ 6 Å (two bilayers). We thus propose that some inter-

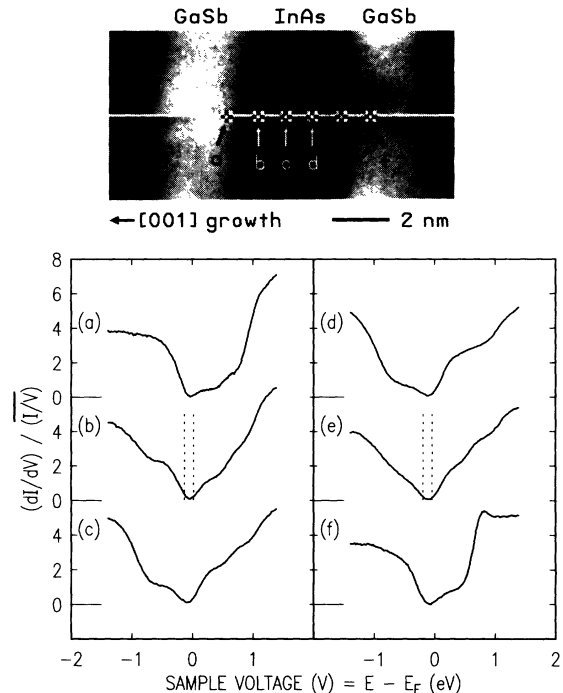


FIG. 4. Spatially resolved spectra were acquired every 1.06 Å along the white line shown in the middle of the STM image. Six spectra are selected for display, from the indicated spatial locations: (a) GaSb layer, (b) GaSb on InAs interface, (c), (d) InAs layer, (e) InAs on GaSb interface, and (f) GaSb layer. Superlattice band gap is indicated by dashed lines in spectra (b) and (e).

mixing occurs at this interface over this distance. This finding is consistent with the extra roughness seen at the InAs on GaSb interfaces in Fig. 1, and is also consistent with the interface structure deduced from x-ray photoemission and reflection high-energy electron-diffraction studies [4]. Those results depend somewhat on whether Sb_2 or As_2 soaks are used during growth, but in both cases the InAs on GaSb interface is found to have more intermixing compared to GaSb on InAs. This behavior is believed to arise from a lower surface free energy for Sb compared to As, producing exchange of Sb and As when InAs is grown on GaSb, but not for growth of GaSb on InAs.

In summary, we have used a new method to deduce interface roughness in InAs/GaSb superlattices, and have studied intermixing at the interfaces using tunneling spectroscopy. The method developed here for roughness determination should have general applicability to a wide class of III-V heterointerfaces, which can serve to quantify many of the prior discussions concerning roughness in these systems [13–15]. Asymmetry between interfaces in our InAs/GaSb superlattices is observed in both structural images and electronic spectroscopy, with the InAs on GaSb interfaces being rougher than GaSb on InAs. Further development in the interpretation of the tunneling spectroscopy results may lead to additional information

on the structure of these interfaces.

We greatly acknowledge H. Munekata for providing us with some InAs wafers. This work was supported in part by Advanced Research Projects Agency under Contract No. N00014-93-1-0881 and Air Force Office of Scientific Research under Contract No. F49620-93-1-0258.

-
- [1] H. W. M. Salemink, O. Albrektsen, and P. Koenraad, *Phys. Rev. B* **45**, 6946 (1992).
- [2] S. Gwo, K.-J. Chao, C. K. Shih, K. Sadra, and B. G. Streetman, *Phys. Rev. Lett.* **71**, 1883 (1993).
- [3] R. H. Miles, J. N. Schulman, D. H. Chow, and T. C. McGill, *Semicond. Sci. Technol.* **8**, S102 (1993).
- [4] D. A. Collins, T. C. Fu, T. C. McGill, and D. H. Chow, *J. Vac. Sci. Technol. B* **10**, 1779 (1992); M. W. Wang, D. A. Collins, T. C. McGill, and R. W. Grant, *J. Vac. Sci. Technol. B* **11**, 1418 (1993).
- [5] B. R. Bennett, B. V. Shanabrook, R. J. Wagner, J. L. Davis, and J. R. Waterman, *Appl. Phys. Lett.* **63**, 949 (1993).
- [6] C. R. Bolognesi, I. Sela, J. Ibbetson, B. Brar, H. Kroemer, and J. H. English, *J. Vac. Sci. Technol. B* **11**, 868 (1993).
- [7] J. A. Stroscio and R. M. Feenstra, in *Methods of Experimental Physics*, edited by J. A. Stroscio and W. J. Kaiser, Scanning Tunneling Microscopy Vol. 27 (Academic, Boston, 1993), Chap. 4.
- [8] The fit function is $2\Lambda\Delta^2/(1+q^2\Lambda^2)$ where Δ is the roughness amplitude and Λ the correlation length. This form is appropriate for a random distribution of steps with average spacing Λ , and it provides a much better fit to the data than a conventional Gaussian form.
- [9] R. M. Feenstra (to be published).
- [10] R. M. Feenstra and J. A. Stroscio, *J. Vac. Sci. Technol. B* **5**, 923 (1987).
- [11] H. Cartensen, R. Manzke, I. Schäfer, and M. Skibowski, in *Proceedings of the 18th International Conference on the Physics of Semiconductors*, edited by O. Engström (World Scientific, Singapore, 1987), p. 125.
- [12] D. Z.-Y. Ting, E. T. Yu, and T. C. McGill, *Phys. Rev. B* **45**, 3583 (1992).
- [13] A. Ourmazd, D. W. Taylor, J. Cunningham, and C. W. Tu, *Phys. Rev. Lett.* **62**, 933 (1989).
- [14] C. A. Warwick and R. F. Kopf, *Appl. Phys. Lett.* **60**, 386 (1992).
- [15] D. S. Katzer, D. Gammon, and B. V. Shanabrook, *J. Vac. Sci. Technol. B* **10**, 800 (1992).

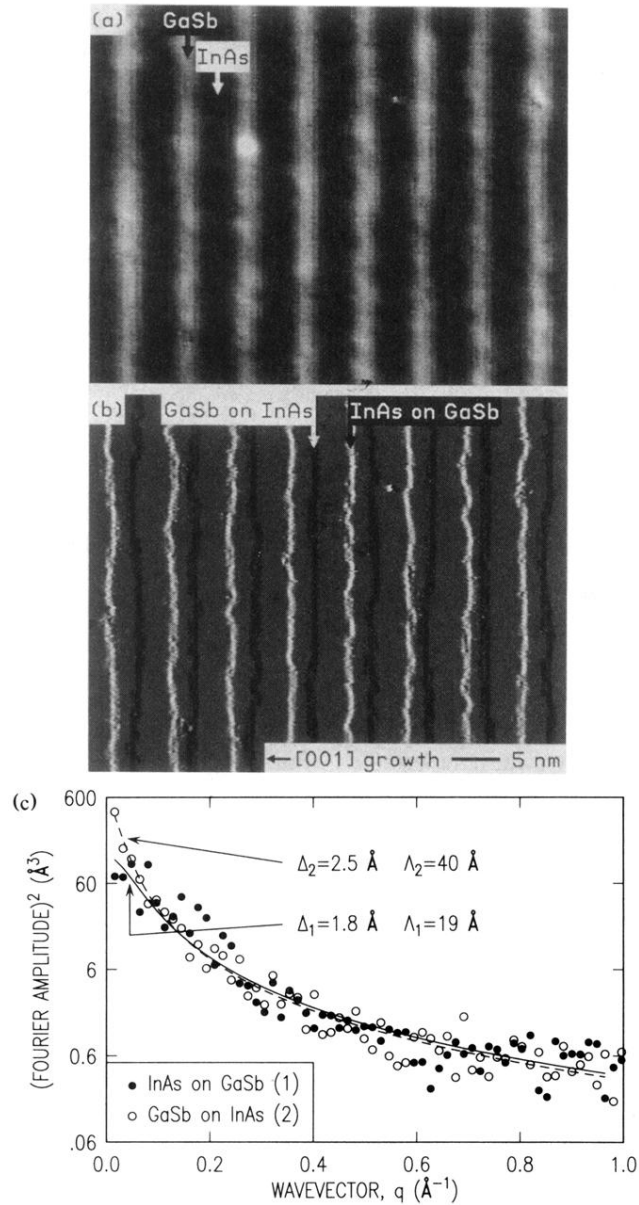


FIG. 1. (a) Constant-current STM image of InAs/GaSb superlattice, consisting of 42 Å thick InAs and 24 Å thick GaSb layers. Image was acquired at a sample voltage of -1.5 V . The [001] growth direction is indicated. Grey-scale range is 2.0 Å. (b) Derivative of (a). (c) Interface roughness spectra, computed on the 400 Å long interfaces of (b), using seven interfaces of each type and averaging the results. The data from each interface type are fit to a Lorentzian form, yielding results shown for the roughness amplitudes (Δ) and correlation lengths (Λ).

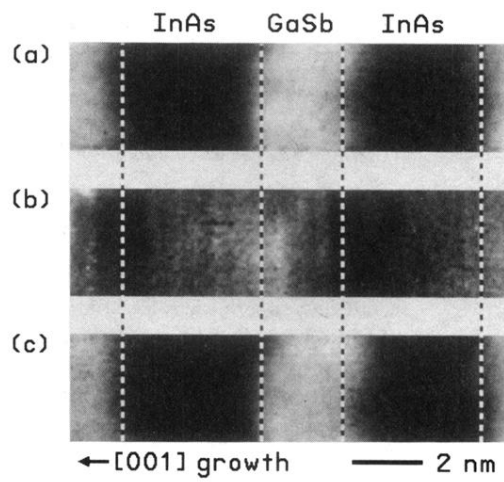


FIG. 2. Constant-current STM images, acquired at sample voltages of (a) -1.3 , (b) $+1.3$, and (c) -1.3 V. Grey-scale ranges are (a) 1.4 , (b) 0.8 , and (c) 1.4 Å. Dashed lines indicate the position of the interfaces between InAs and GaSb.

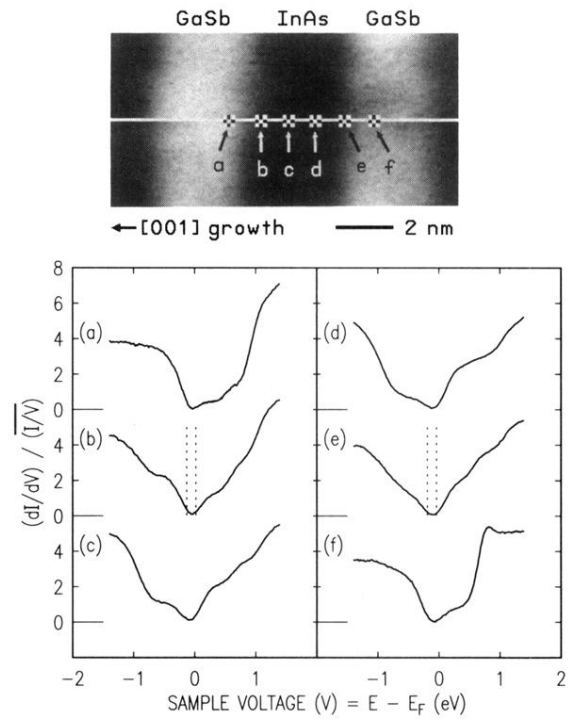


FIG. 4. Spatially resolved spectra were acquired every 1.06 Å along the white line shown in the middle of the STM image. Six spectra are selected for display, from the indicated spatial locations: (a) GaSb layer, (b) GaSb on InAs interface, (c),(d) InAs layer, (e) InAs on GaSb interface, and (f) GaSb layer. Superlattice band gap is indicated by dashed lines in spectra (b) and (e).