

Low-temperature STM on InAs(110) accumulation surfaces

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Abstract. The properties of InAs(110) surfaces have been investigated by means of low-temperature scanning tunneling microscopy and spectroscopy. A technique for ex-situ sulphur passivation has been developed to form an accumulation layer on such a surface. Tunneling spectroscopy at 4.2 K shows the presence of 2D subbands in the accumulation layer. Measurements in high magnetic field demonstrate Landau quantization of the energy spectrum, both in the 2D subbands and in the 3D bulk conduction band.

The properties of 2D electron gases (2DEG) subjected to high magnetic fields have generated a large amount of theoretical and experimental work. In particular, the study of edge channels and electron–electron interactions [1] is currently a field of great interest. Spatially resolved imaging of edge channels was performed by van Haren et al. [2], using a special technique to obtain macroscopically wide channels. For measurements with higher spatial resolution one would like to exploit the potential of STM techniques. However, this requires a semiconductor system having a 2DEG at the surface. InAs is a good candidate material for this, since it is known that it can have a surface 2DEG [3]. Wildöer et al. [4] have performed low-temperature scanning tunneling microscopy and spectroscopy on InAs(110) surfaces that were cleaved in situ at 4.2 K. A clean surface was obtained and Landau quantization was observed. In that study, however, no clear evidence was found for the formation of a surface 2DEG.

In this article we report on the development of a suitable system for STM studies on a 2DEG: the sulphur-passivated InAs(110) surface. We demonstrate the presence of 2D subbands and of Landau quantization in high magnetic fields.

From the work of Tsui [3], it is known that an oxide layer at the surface of n-type InAs pins the Fermi energy above the bottom of the conduction band, thereby forming a surface accumulation layer. We have found that oxidation of the InAs surface is not a practical ex-situ technique for an STM study, since the oxide grows too thick after just a few min of exposure to air. We have therefore studied different passivation

techniques for InAs, taking advantage of its chemical similarity with GaAs, for which a vast literature of passivation methods is available. Those techniques mainly use reactive S-containing solutions and/or gases. In practice, we have focused on two among the most practical and relatively less toxic wet techniques: one employing a solution of $(\text{NH}_4)_2\text{S}$ as in [5], the other one using a solution $\text{CH}_3\text{CSNH}_2/\text{NH}_4\text{OH}$ at 90°C as in [6].

We use InAs wafers with a doping concentration $n = 2.4 \times 10^{16} \text{ cm}^{-3}$, corresponding to a Fermi energy $E_F \approx 13 \text{ meV}$ above the conduction band. Passivated samples are prepared by cleaving them in solution after scribing a mark on the (100) surface. We study different samples with passivation times between 5 s and 20 min. After passivation, samples are rinsed in de-ionized water and blown dry with nitrogen gas. They are then quickly mounted in our STM [7], being thus exposed to air for about 10 min. The STM vacuum can is then pumped out for about 90 min (at room temperature) in order to sublimate the excess sulphur [8]. Helium exchange gas is allowed in the STM vacuum can just before it is cooled down to 4.2 K in a cryostat equipped with a 7 T superconducting magnet. The STM tips are cut in ambient from Pt(90%)Ir(10%) wire.

Figure 1 shows a typical topographic image of the S-passivated InAs surface at 4.2 K. The surface has a root-mean-square (rms) roughness of about 0.5 nm over an area of $400 \text{ nm} \times 400 \text{ nm}$. Topographic images for areas of $10 \text{ nm} \times 10 \text{ nm}$ can show an rms roughness of about 0.1 nm. Despite their flatness, those images do not show atomic resolution. Similar results were found on GaAs by Gwo et al. [5]. We find that the thickness of the sulphur passivation layer amounts to a few monolayers, unlike the case of oxygen passivation.

Different measurements for passivation times between 5 s and 10 min show that the flattest surfaces are obtained for passivation times between 30 s and 2 min. For longer passivation times the rms roughness increases up to a few nanometers. Concerning the rms roughness, we find similar results for both of the passivation methods. From Auger spectroscopy measurements we find that the content of oxygen at the surface is significantly lower for the samples passivated

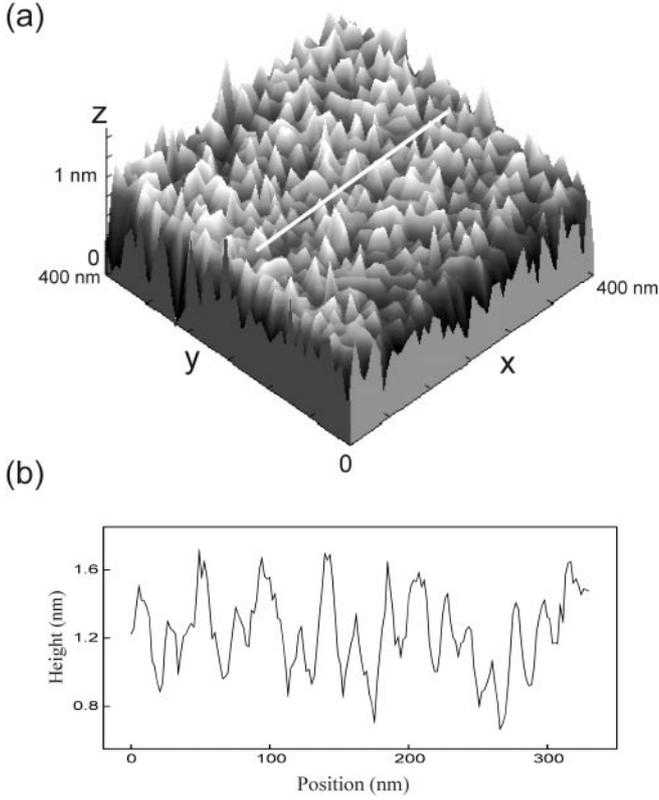


Fig. 1. **a** STM topographic image at 4.2 K of InAs(110) surface S-passivated with $\text{CH}_3\text{CSNH}_2/\text{NH}_4\text{OH}$ for 40 s. The image shows a rms roughness of 0.5 nm over an area of $400\text{ nm} \times 400\text{ nm}$. **b** Line profile along the line indicated in the topography

with $\text{CH}_3\text{CSNH}_2/\text{NH}_4\text{OH}$ than for the ones passivated with $(\text{NH}_4)_2\text{S}$.

Figure 2 shows the $I - V$ characteristic of an S-passivated InAs sample. The onsets of the conduction and valence band are found at about 0 and -400 mV , respectively, and are indicated by arrows in Fig. 2. These values are in good agreement with the value for the gap of bulk InAs at 4.2 K: 420 meV. Other authors measuring STM spectroscopy on InAs cleaved in situ [4, 9] have encountered tip-induced band bending. The actual tip-sample voltage was found to be less than the applied bias of about 200 mV, for samples with low doping level. We find that our samples do not show tip-induced band bending.

In Fig. 3a a typical spectroscopy measurement at 4.2 K is given. Three clear peaks are seen for negative bias positions: $V_0 = -131\text{ mV}$, $V_1 = -85\text{ mV}$, $V_2 = -37\text{ mV}$, and a fourth, smaller peak $V_3 = -17\text{ mV}$. We ascribe these peaks to tunneling from 2D electronic subbands in the accumulation layer at the InAs surface (see Fig. 4). The exact calculation of the confinement potential in the accumulation layer requires numerical self-consistent calculations [10]. We can use a simple expression for the energy values of the bottom of the 2D subbands when we approximate the confining potential by a triangular well [11]:

$$E_k = \left(\frac{\hbar^2}{2m^*} \right)^{1/3} \left(\frac{3\pi e F}{2} \left[k + \frac{3}{4} \right] \right)^{2/3}. \quad (1)$$

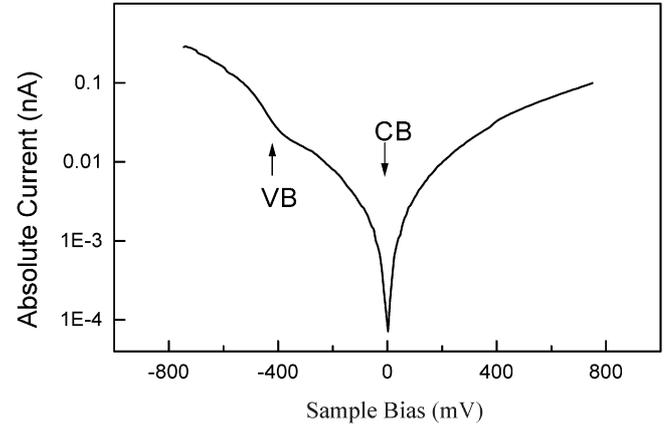


Fig. 2. STM spectroscopy at 4.2 K of the InAs(110) accumulation surface S-passivated with $\text{CH}_3\text{CSNH}_2/\text{NH}_4\text{OH}$ for 40 s. From the $I - V$ (and $dI/dV / I/V$) characteristic, the onset of the conduction band (CB) and valence band (VB) are found at about 0 and -400 mV , respectively. This is in good agreement with the value of the gap of InAs (420 meV)

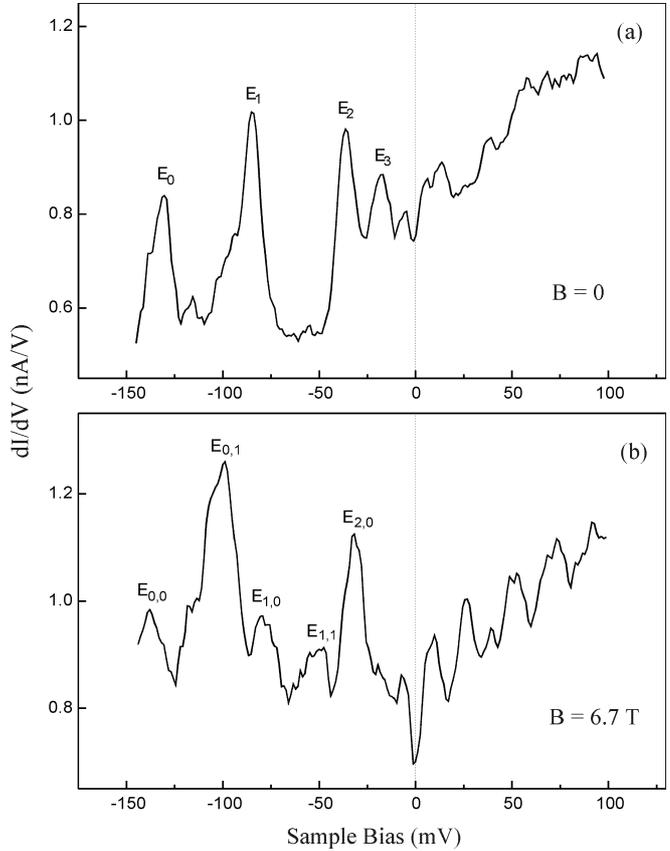


Fig. 3. **a** STM spectroscopy at 4.2 K of the InAs(110) accumulation surface S-passivated with $\text{CH}_3\text{CSNH}_2/\text{NH}_4\text{OH}$ for 40 s. Four peaks can be resolved for negative sample bias at the values: -131 , -85 , -37 , and -17 mV . This graph is calculated by numerical differentiation of the measured $I - V$ characteristic. **b** STM spectroscopy as in **a** but with a magnetic field $B = 6.7\text{ T}$ perpendicular to the surface. Peaks at negative sample bias values of: -138 , -99 , -79 , -49 , and -32 mV are observed indicating Landau splitting of the 2D subbands. Additional peaks for positive bias values: 10 , 26 , 50 , 73 , and 91 mV are due to Landau quantization in the conduction band of InAs. The tunnel resistance is about $1\text{ G}\Omega$. $I - V$ s are measured after turning the feedback control off. We average $I - V$ s over 25 curves each consisting of 201 points with acquisition time of 1.3 ms per point

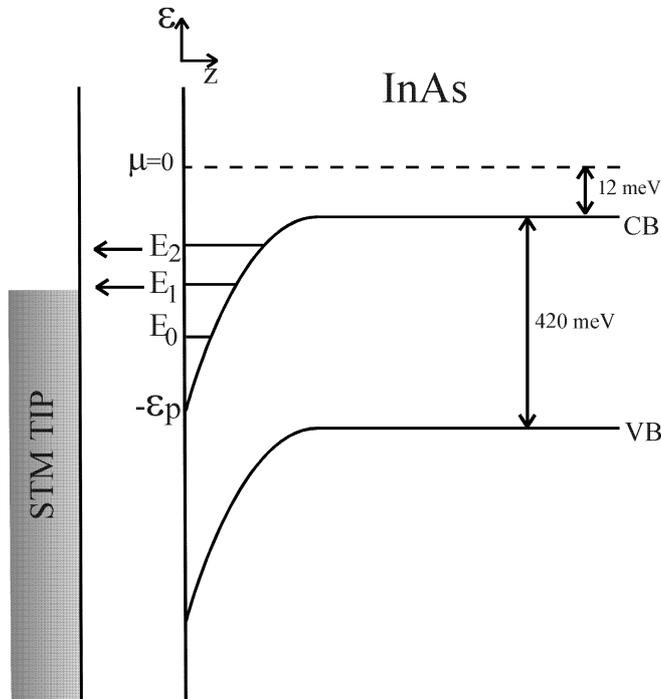


Fig. 4. Schematic band diagram of n-type InAs for the case of Fermi-energy pinning. The conduction band bottom at the surface is bent at the energy $-\varepsilon_p$ below the Fermi level. If the electric field in the accumulation layer is sufficiently strong, quantised 2D states are present at the surface (indicated with E_k). When the Fermi level of the tip is aligned with a 2D state, a peak in the dI/dV is expected

Here F is the electric field, $k = 0, 1, \dots$ is the subband index, and the zero for the energy is taken at the bottom of the well. From (1) we find $E_k/E_0 = (4k/3 + 1)^{2/3}$. The energy of the quantum states measured from the bottom of the conduction band is $E_k = \varepsilon_p + eV_k$, where V_k are the measured peaks positions, and ε_p is the value of Fermi-energy pinning (see Fig. 4). From the four observed peaks we obtain: $\varepsilon_p = 194 \pm 5$ meV. From (1) and the calculated value for ε_p we find for the accumulation electric field $F \approx 4 \times 10^6$ V/m. The accumulation length $L_a = \varepsilon_p/F$ is about 50 nm. From (1) and the given values of ε_p and F we can calculate the peak positions: $V_0 = -128$ mV, $V_1 = -78$ mV, $V_2 = -37$ mV, and $V_3 = -10$ mV, with an uncertainty of ± 5 mV; in good agreement with the measured values.

Peaks in the dI/dV vs. V characteristics for negative bias are observed in all our S-passivated samples. Peaks are absent in those areas where a tip crash has removed the surface layer. We find that the position of the peaks vary by the order of 10% (that amounts to ± 10 meV for the lowest level) for tip displacements of the order of 1 nm. After moving the tip around a few nanometers and coming back to the initial spot we find a different tip-sample distance and slightly different peak positions. What may happen is that the tip touches the S layer and eventually traps some sulphur atoms in an uncontrollable way. We conclude that the change in the peak voltages as a function of the lateral displacement of the tip does not provide direct information about the electronic localisation length. Instead it depends in a complicated and irreversible way on the configuration of the sulphur atoms around the STM tip and on the properties of the InAs accumulation layer.

In the simplest picture of the STM, the dI/dV characteristic is expected to measure the density of states of the probed system, this result is derived under the approximation of taking the tunneling matrix element between the tip and the sample as a constant. In the case of an ideal 2DEG a step-like characteristic is expected, with a new step arising for energies corresponding to the bottom of a 2D subband. Previous experiments [12, 13], with planar tunnel junctions made of Pb-oxide-InAs, have demonstrated the presence of a surface 2DEG. In those experiments the electric field was probably much more uniform than in ours, whereas the dI/dV characteristics can still be interpreted as in the case of STM [12]. In [12, 13] peaks were found in the dI/dV with a broadening between 25 and 40 meV. Peak positions were interpreted as the onsets of 2D subbands and the broadening due to electrical field inhomogeneity. Moreover the measured values for the bottom of the 2D subbands found in [12, 13] are in good agreement with our results. Therefore we may have reproduced those results in our STM spectroscopy characteristics. We interpret our peaks as arising from the discontinuity in the density of states of the surface 2DEG, which is expected and probed when the Fermi energy of the tip is aligned with the bottom of a 2D subband. The measured width of our peaks is about 15 meV.

Comparing dI/dV vs. V spectroscopy for the two passivation methods we find that samples passivated with $\text{CH}_3\text{CSNH}_2/\text{NH}_4\text{OH}$, for times between 30 s and 2 min, give spectroscopic characteristics with the best reproducibility and the highest resolution. In particular, on the sample that was passivated with $\text{CH}_3\text{CSNH}_2/\text{NH}_4\text{OH}$ for 40 s we found an area of about $10 \text{ nm} \times 10 \text{ nm}$ where the peak positions reproduced within 10 meV. Spectroscopy characteristics in that area are shown in Fig. 3.

Figure 3b shows the dI/dV vs. V characteristic with a magnetic field $B = 6.7$ T applied perpendicularly to the surface. This characteristic has been measured at the same point as that in Fig. 3a, but with the caveat that the magnetic field may cause a lateral displacement of the tip of the order of a few nanometers. We now discuss a simple model to interpret the oscillations present in high-magnetic-field characteristics. Landau quantization is expected for sufficiently strong magnetic field $\hbar\omega_c > \Gamma$, where $\hbar\omega_c = \hbar eB/m^*$ is the cyclotron energy, i.e. the energy separation between different Landau levels, and Γ is the broadening of the Landau levels due to disorder. Landau levels have energies $E_{k,n} = E_{k,0} + \hbar\omega_c(n + 1/2)$, where $k = 0, 1, \dots$ is the electric field quantization number as in (1), and $n = 0, 1, \dots$ is the Landau level index. The magnetic field also changes the value of the Fermi energy with respect to the bottom of the conduction band in the bulk InAs. Most probably, the value of the electric field in the accumulation layer also changes such that the bias position of the “electric peaks” ($E_{k,n=0}$) is expected to be shifted a few meV from the $B = 0$ peaks. We expect peaks in the dI/dV characteristic from the Landau levels at a distance of $\hbar\omega_c = 33$ meV (at 6.7 T) from the “electric peaks”.

Comparing the two characteristics in Figs. 3a and 3b, for $B = 0$ and 6.7 T, respectively, we see that the “electric peaks” present in Fig. 3a are reproduced also in Fig. 3b with a shift of (± 7 meV). Two extra peaks are present in Fig. 3b that can be interpreted as being due to tunneling from the levels $E_{0,1}$ and $E_{1,1}$ with measured cyclotron energies of 39 meV and

30 meV, respectively. We note that for lower magnetic field and/or for other samples the spectra show magnetic field dependence but no clear peak splitting was found. From this fact we find an upper boundary for the Landau level broadening of $\Gamma \approx 35$ meV.

In Fig. 3b, peaks in the dI/dV vs. V characteristic are also present for positive sample bias. We interpret these peaks as being due to Landau quantization in the conduction band of InAs. The energy spectrum of electrons in the conduction band in the presence of a magnetic field directed along the z axis is $E_{k_z, n} = \hbar^2 k_z^2 / 2m + \hbar\omega_c(n + 1/2)$, where k_z is the wave vector in the z direction. The spectrum is continuous, but the density of states contains singularities at the energies $\hbar\omega_c(n + 1/2)$. These singularities are also expected to give rise to peaks in dI/dV characteristics. The Fermi energy in bulk InAs, for the given electron density and $B = 6.7$ T, is located at about 4 meV above the first Landau level. Peaks in the dI/dV , for positive bias, are then expected to be due to tunneling of electrons into the empty Landau levels with numbers $n = 1, 2, \dots$

Figure 5 shows the dI/dV characteristic in a magnetic field $B = 6.7$ T for a sample passivated with $\text{CH}_3\text{CSNH}_2/\text{NH}_4\text{OH}$ for 30 s. Six peaks can be clearly distinguished, due to tunneling into the Landau levels $n = 1$ to 6. The level spacing gives a measure of the cyclotron energy

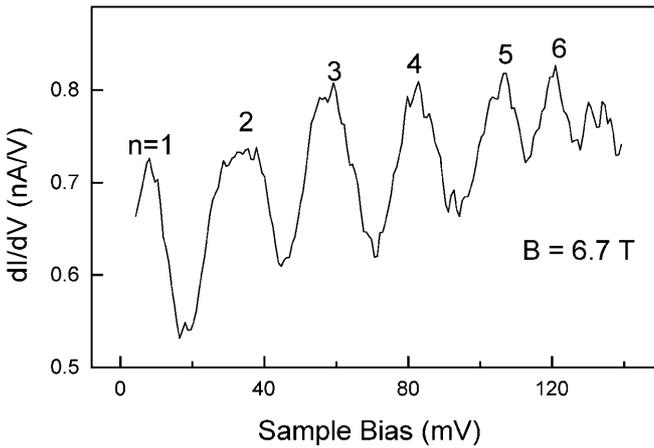


Fig. 5. STM spectroscopy at 4.2 K of InAs(110) accumulation surface S-passivated with $\text{CH}_3\text{CSNH}_2/\text{NH}_4\text{OH}$ for 30 s. Six peaks can be resolved for positive sample bias: 8, 35, 59, 83, 106, 121 mV, indicated with $n = 1, \dots, 6$. They are due to tunneling of electrons into empty Landau levels in the conduction band of InAs

$\hbar\omega_c = 23 \pm 4$ meV. From this value we can calculate the effective mass: $m^*/m_0 = 0.033 \pm 0.006$, which is higher than the generally accepted value of the mass at the bottom of the conduction band: $m^*/m_0 = 0.023$. We attribute this discrepancy to mass non-parabolicity in the conduction band of InAs [12, 13]. Peaks due to Landau quantization in the conduction band have been observed for different samples in magnetic fields above 6 T.

In conclusion, we have developed a technique for ex-situ near-monolayer passivation of InAs. We have demonstrated, using STM spectroscopy, the presence of 2D subbands at the passivated (110) surface of InAs, and shown the presence of Landau quantization in high magnetic field. These are the first STM measurements on a 2D electron gas at the surface of a semiconductor. We have shown that our system avoids the problems of tip-induced band banding, and we have studied Landau quantization in the conduction band of InAs, where up to six Landau levels have been clearly resolved.

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