Chapter 4

The influence of low energy Ar-sputtering on the electronic transport properties of InAs-based quantum well structures.

The influence of low energy (80 – 500 eV) Ar-ion milling cleaning techniques on InAs based quantum well structures is investigated. It is found that both etching with a Kaufmann source and sputter-etching with a rf-plasma enhances the electron density and reduces the mobility. An anneal at 180°C has little effect, and only recovers damage caused by low energy (80 eV) Kaufmann-etching.

4.1 Introduction.

InAs based quantum well (QW) structures are increasingly being used to study electronic transport in the ballistic regime [1, 2]. At the InAs surface the Fermi level is pinned in the conduction band. Due to this the metal-InAs contacts lacks a Schottky barrier, and it is not necessary to alloy the contact, as is needed, for
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instance, to make contact to the 2DEG in a GaAs/AlGaAs heterostructure. Therefore
the contact is free of a diffusive contact area usually present in alloyed contacts,
which is especially important when the electron transport should remain ballistic
up to the metal-contact, for instance in superconductor - 2DEG - superconductor
junctions [3, 4], or in the case of magnetic contacts, to investigate spin-polarized
transport [5, 6].

A standard procedure in making metal contacts to a semiconductor, for instance
InAs, is an in-situ cleaning of the semiconductor surface by low energy, 100 –
500 eV, Ar-ion milling prior to metal deposition, to remove the natural oxide present
at semiconductor surfaces. Millea et al. [7, 8] studied the effect of low energy Ar-
bombardment on the surface conductivity of p-InAs. They showed that low energy
Ar-bombardment enhances the electron density in the 2DEG present in the inversion
layer at the surface of p-InAs. The increase of the electron density can have two
causes, either the Fermi-level pinning at the surface is changed, or, as assumed by
Millea et al., donor states are introduced. In this chapter we will show that the latter
is more likely. The introduction of donor states upon low energy Ar-bombardment
is a result of the unique radiation damage properties of InAs [9]. When InAs is
irradiated, either by high energy electrons or low energy ions, point defects are
introduced. These point defects, that are located near the surface, consist of shallow
donors. Millea et al. [7] interpreted their results by assuming these point defects can
migrate into the bulk, up to a depth of 0.1 \( \mu \text{m} \). Because in the bulk-samples used
by Millea et al. the introduced donors are far below the surface, and thus spatially
separated from the surface 2DEG in the inversion layer, the mobility \( \mu_e \) increases
with increasing sheet electron density \( n_S \) as a result of enhanced electron screening
of scatterers.

So far, however, no systematic study has been made to investigate the influence
of the Ar-ion milling on the electronic transport properties of the 2DEG present
in an InAs QW. In this chapter we compare the influence on the 2DEG in an
InAs QW of two commonly used cleaning techniques, Kaufmann sputtering with low energy Ar-atoms, and rf sputter-etching with an Ar-plasma. We find that the electron density in the QW increases with the applied Ar-dose, as was observed by Millea et al. For the mobility we find that it decreases drastically with the Ar-dose, opposed to the result found by Millea et al. [8] for the 2DEG present in the inversion layer of p-InAs.

### 4.2 General results.

In a Kaufmann source Ar atoms are ionized by thermal electrons from a filament, and accelerated by means of a high voltage between a cathode and an acceleration grid. For focussing reasons the ion beam is neutralized by a second filament after acceleration. In our system the flux of Ar-atoms is determined by measuring the ion-beam current on a shutter, with a well defined area, prior to neutralizing the beam. In a rf-sputter etching machine a plasma of Ar-ions and electrons is created using a high voltage and a rf-signal. A self-bias comparable to the dc voltage-bias will occur in the plasma, which directs the Ar-ions towards the sample. For rf-sputtering the Ar flux is determined from the plasma current. In general the etch-rate using a Kaufmann source is much higher than for rf-sputter etching with the same Ar flux. This is because the working pressure used in rf-sputtering is much higher, resulting in scattering between ions, which reduces their forward energy towards the substrate.

For cleaning purposes it is only needed to etch 10 to 20 Å, the typical thickness of the natural oxide on semiconductors. Etching more would unnecessarily roughen the surface. Furthermore, if the thickness of a QW is reduced, the quantized energy levels will shift up in energy, and as a result the electron density will go down. Samples consist of a standard Hall bar geometry in a GaSb/AlSb/InAs/AlSb heterostructure, with a 2DEG present in the 15 nm thick InAs layer, see Fig. 4.1.
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Figure 4.1: Schematic picture of the samples, a standard Hall-bar configuration. The dimensions are such that the measured longitudinal resistance equals $0.88 R_c$, the square resistance.

for a schematic sample layout and the measurement configuration. The GaSb/AlSb top-layer is removed chemically, using photoresist developer. This exposes the InAs layer, and in order to prevent ageing effects the Hall-bar is put in one of the two vacuum systems immediately, depending on whether it will be etched using a Kaufmann source, or a rf-plasma. We used two different wafers, G1425 and G1481, with comparable layer structure, but different transport parameters, see Table 4.1. To cover a wide variety of Ar-energies, from low to moderate, we investigated 4 different etching procedures; Kaufmann etching, (i) 500 eV, with a measured etch-rate per Ar-flux of $19.3 \pm 0.9 \text{ Å}/(\text{mC/cm}^2)$, (ii) 150 eV, etch-rate $4.5 \pm 1.0 \text{ Å}/(\text{mC/cm}^2)$, (iii) 80 eV, etch-rate negligible (not measurable), and rf-plasma sputtering, (iv) 400 eV, etch-rate $2.0 \pm 0.2 \text{ Å}/(\text{mC/cm}^2)$. In all measurements a maximum of 37 Å
Table 4.1: Electronic transport properties obtained from longitudinal- and Hall resistance measurements.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Energy (eV)</th>
<th>Dose (mC/cm²)</th>
<th>(n_s) ((10^{16}) m⁻²)</th>
<th>(\mu_e) (m²/Vs)</th>
<th>Etched (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>wafer G1425, bare sample, top-layer removed</td>
<td>–</td>
<td>–</td>
<td>1.1</td>
<td>2.2</td>
<td>–</td>
</tr>
<tr>
<td>wafer G1425, Kaufmann sputtering</td>
<td>500</td>
<td>1.06</td>
<td>20</td>
<td>0.02</td>
<td>20.0</td>
</tr>
<tr>
<td>B</td>
<td>100</td>
<td>1.05</td>
<td>5.4</td>
<td>0.4</td>
<td>2.2</td>
</tr>
<tr>
<td>C</td>
<td>100</td>
<td>1.08</td>
<td>4.3</td>
<td>0.5</td>
<td>2.2</td>
</tr>
<tr>
<td>wafer G1481, bare sample, top-layer removed</td>
<td>–</td>
<td>–</td>
<td>0.59</td>
<td>0.69</td>
<td>–</td>
</tr>
<tr>
<td>H2</td>
<td>–</td>
<td>–</td>
<td>0.54</td>
<td>0.87</td>
<td>–</td>
</tr>
<tr>
<td>H7</td>
<td>–</td>
<td>–</td>
<td>1.07</td>
<td>1.00</td>
<td>–</td>
</tr>
<tr>
<td>H7(^b)</td>
<td>–</td>
<td>–</td>
<td>0.56</td>
<td>1.23</td>
<td>–</td>
</tr>
<tr>
<td>H11</td>
<td>–</td>
<td>–</td>
<td>1.14</td>
<td>1.03</td>
<td>–</td>
</tr>
<tr>
<td>wafer G1481, Kaufmann sputtering</td>
<td>80</td>
<td>0.53</td>
<td>0.89</td>
<td>2.2</td>
<td>–</td>
</tr>
<tr>
<td>H3(^b)</td>
<td>–</td>
<td>–</td>
<td>1.3</td>
<td>1.2</td>
<td>–</td>
</tr>
<tr>
<td>H4</td>
<td>80</td>
<td>1.06</td>
<td>1.1</td>
<td>1.9</td>
<td>–</td>
</tr>
<tr>
<td>H4(^b)</td>
<td>–</td>
<td>–</td>
<td>1.3</td>
<td>1.3</td>
<td>–</td>
</tr>
<tr>
<td>H6</td>
<td>80</td>
<td>2.11</td>
<td>1.6</td>
<td>0.8</td>
<td>–</td>
</tr>
<tr>
<td>H6(^b)</td>
<td>–</td>
<td>–</td>
<td>1.2</td>
<td>1.3</td>
<td>–</td>
</tr>
<tr>
<td>H8</td>
<td>170</td>
<td>2.5</td>
<td>8.2</td>
<td>0.14</td>
<td>11.3</td>
</tr>
<tr>
<td>H8(^b)</td>
<td>–</td>
<td>–</td>
<td>7.5</td>
<td>0.14</td>
<td>–</td>
</tr>
<tr>
<td>H9</td>
<td>170</td>
<td>5.1</td>
<td>8.5</td>
<td>0.14</td>
<td>23.0</td>
</tr>
<tr>
<td>H10</td>
<td>155</td>
<td>6.7</td>
<td>8.4</td>
<td>0.17</td>
<td>30.1</td>
</tr>
<tr>
<td>wafer G1481, rf-sputtering</td>
<td>400</td>
<td>4.6</td>
<td>2.36</td>
<td>0.72</td>
<td>9.2</td>
</tr>
<tr>
<td>H12(^c)</td>
<td>–</td>
<td>–</td>
<td>11.4</td>
<td>0.076</td>
<td>–</td>
</tr>
<tr>
<td>H13</td>
<td>400</td>
<td>9.1</td>
<td>11.9</td>
<td>0.084</td>
<td>18.2</td>
</tr>
<tr>
<td>H13(^c)</td>
<td>–</td>
<td>–</td>
<td>11.4</td>
<td>0.082</td>
<td>–</td>
</tr>
<tr>
<td>H14</td>
<td>400</td>
<td>13.7</td>
<td>14.7</td>
<td>0.069</td>
<td>27.4</td>
</tr>
<tr>
<td>H15</td>
<td>400</td>
<td>18.3</td>
<td>15.6</td>
<td>0.072</td>
<td>36.6</td>
</tr>
</tbody>
</table>

\(^a\) Sample C was etched using Xe instead of Ar.

\(^b\) After 20 min. anneal at 180°C.

\(^c\) After 10 min. anneal at 180°C.

of InAs was etched, see Table 4.1. After etching the samples are wire bonded as soon as possible, and measurement are done immediately. In an InAs layer exposed to air
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the transport properties of the 2DEG are likely to degrade. In order to prevent this as much as possible all steps, from chemically removing the toplayer to emerging the sample in L-He for measurements, are done within one day.

The longitudinal and Hall resistance are measured at 4.2 K, using a standard 4 point lock-in technique, as a function of magnetic field. At low magnetic field we may write for the longitudinal- and Hall-resistance:

\[
R_{\text{Hall}}(B) = \frac{B}{en_S} \quad (4.1)
\]
\[
R_S(B = 0) = \frac{1}{en_S \mu_e} \quad (4.2)
\]

From the measured resistances we extract, with the aid of Eq.(4.1) and (4.2), the carrier density and mobility of the 2DEG in the InAs QW, listed in Table 4.1. Fig. 4.2 shows the dependence of \( \mu_e \) and \( n_S \) on the dose of Ar-atoms, for 80 eV Kaufmann etching (a), and for 400 eV rf-sputtering (b). As can be seen, at low energy and low dose \( n_S \) increases linearly with the Ar-dose (Fig. 4.2a). The efficiency, the number of introduced donors per incoming Ar-atom, is very low, in the order of \( 10^{-4} \). From Table 4.1, and also from Fig. 4.2b, we can see that the efficiency increases when the energy is increased. At higher energy \( n_S \) saturates for very high Ar-dose (Fig. 4.2b).

The mobility \( \mu_e \) shows an initial increase at low Ar-energy and dose, as shown in Fig. 4.2a, but is rapidly reduced at higher energy and dose. At very high dose the mobility saturates, at the dose where also the electron density \( n_S \) starts to saturate, see Fig. 4.2b.

In a 2DEG we can write for the elastic mean free path \( \ell \)

\[
\ell = v_F \tau = \frac{\hbar k_F m^* \mu_e}{m^* e} = \frac{\hbar}{e} \sqrt{2\pi n_S \mu_e}. \quad (4.3)
\]

At the same time we can estimate that \( \ell \) will scale with the average distance between the defects, which in a 2D system can be expressed as

\[
\ell \propto \frac{1}{\sqrt{n_{\text{defects}}}}. \quad (4.4)
\]
Figure 4.2: Dependence of the electron density $n_S$ and mobility $\mu_e$ on the Ar-dose, for 80 eV Kaufmann etching (a), and for 400 eV rf-sputtering (b).

Combining Eq. (4.3) and (4.4), under the assumption that all introduced defects act as shallow donors, thus $n_S \approx n_{\text{defects}}$, we may write:

$$\ell = \frac{\hbar}{e} \sqrt{2 \pi n_S \mu_e} \propto \frac{1}{\sqrt{n_{\text{defects}}}} \approx \frac{1}{\sqrt{n_S}}$$

$$\Rightarrow$$

$$\mu_e \propto \frac{1}{n_S}.$$  \hspace{1cm} (4.5)
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Figure 4.3: Mobility $\mu_e$ plotted versus the electron density $n_S$, on a log-log scale. For both wafers a straight line is shown: wafer G1425 (dashed line) slope $-1.08 \pm 0.01$, wafer G1481 (dotted line) slope $-1.21 \pm 0.04$. Individual points: G1425 bare sample (+), 100 eV Kaufmann etched (△), 500 eV Kaufmann etched (▽, ignored in linear fit), G1481 bare sample (×, ignored in linear fit), 80 eV Kaufmann etched (○), 155–170 eV Kaufmann etched (□), and 400 eV rf-plasma sputtered (◇).

The mobility $\mu_e$ is thus inversely proportional to the electron density $n_S$. Strictly speaking Eq.(4.3) is only valid for a 2DEG with only the lowest subband filled. In the case of two or more occupied subbands the system in principle consists of several 2D electron layers in parallel, we therefore believe that Eq.(4.5) is still a valid approximation. In Fig. 4.3 $\mu_e$ is plotted versus $n_S$ on a log-log scale, showing a linear dependence. For wafer G1425 we obtain a slope, by applying a linear fit to the data, of $-1.08 \pm 0.01$, and for wafer G1481 we get $-1.21 \pm 0.04$, both reasonably close to the expected slope of $-1$.

The initial rise of the mobility can not be explained within this model. It can be
understood qualitatively by realizing that defects are introduced first at the surface and later also in the InAs layer. Due to the increased electron density $n_S$ the conduction band in the InAs QW will bend in such a way that electrons on average will move away from the surface, and thus suffer less scattering from the surface defects, hence the initial increase of the mobility $\mu_e$. When the electron density is further increased defects in the InAs layer are also introduced, and the mobility will decrease.

The sample that has been processed using 500 eV Kaufmann-etching does not fit onto the straight line shown in Fig. 4.3. In Eq.(4.3) and (4.4) we assume that the electron gas is still 2-dimensional, or at least that the relevant scattering is only in the plane of the 2DEG. From $n_S$ and $\mu_e$, given in Table. 4.1, we can calculate for sample A $\ell \approx 15$ nm, which is comparable to the thickness of the InAs layer. Therefore in this sample (A) scattering along the perpendicular direction also becomes important, and Eq.(4.4) does no longer apply.

We also studied the effect of annealing at 180°C. The samples were, with chip carrier, placed in a photoresist oven for 10 to 20 min., after which they were immediately measured again. In all samples where the Ar-etch removed some of the InAs the anneal had very little effect, meaning the mobility remained low and the electron density stayed high. Only for the samples that were exposed to Ar-atoms with an energy of 80 eV, with a negligible etch-rate, the damage was recovered by annealing.

4.3 Conclusions.

The general trend observed is that the electron density $n_S$ increases and the mobility $\mu_e$ decreases as a result of Ar-sputtering. Especially for higher energies, that are of interest for cleaning purposes, the effect is quite dramatic. The linear dependence of $\mu_e$ on $n_S^{-1}$ indicate that this is the result of introducing defects that act as donors.
Using Xe instead of Ar does not make a significant difference (compare sample B and C), as could be expected. Annealing at 180°C does not recover the damage introduced by the ion milling, except for the case of 80 eV Kaufmann etching, where the etch-rate is negligible, and probably only some minor surface defects are created. Low energy Kaufmann sputtering introduces only small, recoverable, damage, but it does not remove the surface oxide, and is therefore not useful in making good contacts. When higher energy is used the metal-InAs contact is better for two reasons; first the oxide is removed, and second the electron density directly under the metal contact is enhanced. When it is, however, essential that $n_S$ and $\mu_e$ remain constant underneath a metal contact on top of an InAs QW, Ar-sputtering can not be used for cleaning purposes.

References


