Electrical conductivity effects in ultra-thin Tungsten films.

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Abstract

In this work, an attempt is made to characterize the electrical conductivity of ultra-thin films of tungsten (W). This is done by measuring on wafers that have films of W with a thickness between 0.57 and 8.5 nm.

A method for characterizing ultra-thin films is by measuring their I-V relationship on circular transfer length method structures. This can give information about the film’s resistivity, contact resistance and transfer length. By looking at how non-linear the I-V relationship is, information can be obtained regarding the thickness at which the film goes from discontinuous to continuous. By measuring at various temperatures, the temperature coefficient of resistance (TCR) can be obtained.

It was found that the W layers on the tested wafers are highly non-homogeneous. As such, any characterizations from these tests are tentative. Nevertheless, some conclusions could still be drawn.

The thickness at which W films transition from semi-continuous to continuous is around 1.6 nm. W films with a thickness up to at least 0.9 nm have a highly non-linear I-V relationship, a negative TCR and a contact resistance that decreases with an increased temperature.

In order to better characterize ultra-thin W films, new wafers will have to be made. If these are made with W films with thicknesses around 1.6 nm, the thickness at which the transition to continuous occurs can be characterized more precisely.
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## List of acronyms

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<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>W</td>
<td>tungsten</td>
</tr>
<tr>
<td>CTLM</td>
<td>circular transfer length method</td>
</tr>
<tr>
<td>HWALD</td>
<td>hot-wire assisted atomic layer deposition</td>
</tr>
<tr>
<td>TCR</td>
<td>temperature coefficient of resistance</td>
</tr>
</tbody>
</table>
Chapter 1

Introduction

In integrated circuits ultra-thin conducting layers are used in a wide variety of applications. Such ultra-thin layers behave differently from thick layers. One difference is that when such layers fall under a certain thickness, the so-called percolation threshold, the film will become discontinuous. This has a large effect on the film’s conductivity. As such it is important to know at which thickness the film becomes discontinuous. Another difference is that thick layers of metal have an increase in resistance when the temperature increases, but in thin layers that may be reversed.

In this thesis measurements will be made on wafers on which ultra-thin layers of tungsten (W) have been grown by means of hot-wire assisted atomic layer deposition (HWALD). These wafers are of various thickness and have different test structures on them. Measurements will be made on circular transfer length method (CTLM) structures.

The goal of this thesis is to characterize electrical conductivity of ultra-thin W films. More specifically, the resistivity, voltage-dependent-resistance, contact resistance and temperature dependency of all those variables.

In chapter 2 the background for this assignment will be explained. That chapter will deal with conduction in ultra-thin films, a general explanation of CTLM structures, a more specific explanation of the structures used in this assignment and finally the measurement setup used. Chapter 3 deals with the measurement results. First up is the visual inspection, followed by sanity checks to find out if the measurements influence the W, followed by measurements at room temperature and finally measurements at temperatures ranging from 0 to 100 degrees centigrade. In chapter 4, conclusions are drawn and recommendations are made for further research.
Chapter 2

Background

2.1 Conductivity in ultra-thin metal films

If a metal film is extremely thin, it is discontinuous. In that case there are islands of the metal that are not interconnected. A current can still pass through the material, but the electrons will have to pass through a potential barrier. An externally applied voltage alters the height or shape of the barrier and as such the current does not scale linearly with the voltage.

If the metal film increases in thickness, the islands become larger and some of them become interconnected, resulting in a higher conductivity. If the metal layer keeps increasing in thickness, at a certain point all the metal will be connected and it has become continuous.

The thickness at which the transition between continuous and discontinuous occurs is called the percolation threshold. Since discontinuities greatly reduce the conductivity, it is important to know the value of this threshold for those applications in which the metal is used as a conductor. The $Rt^2$ method is one way to establish the value of the percolation threshold [1]. In that method, the resistance of the film times its thickness squared, is compared between the different film thicknesses. The threshold is at the thickness where this value is lowest.

Temperature influence  Temperature dependency on resistivity is called the temperature coefficient of resistance (TCR), indicated by parameter $\alpha [^\circ C^{-1}]$. The equation for TCR is as follows:

$$\rho(T) = \rho(0)(1 + \alpha T)$$  \hspace{1cm} (2.1)

Where $\alpha$ is the TCR, $T$ is the temperature in °C and $\rho(T)$ is the resistivity at a certain temperature. The equation can be rewritten as:

$$\alpha = \frac{\rho(T)}{\rho(0)} - \frac{1}{T}$$  \hspace{1cm} (2.2)
A positive TCR means that a higher temperature, will lead to a higher resistivity. Thick films of W have a positive TCR of 0.0045 [2]. Because electrons have more energy at higher temperatures, it is easier for them to pass through barriers. Therefore it is expect to see a negative TCR in ultra-thin films.

### 2.2 CTLM structures

![CTLM structure diagram](image)

**Figure 2.1:** CTLM structure [3]

CTLM is a method to provide a lot of parameters regarding conductivity with a simple structure [4]. CTLM structures consist of a circular center electrode, around that a ring of the material to be measured and around that another electrode. An example is shown in figure 2.1. By measuring with various widths of the ring of the material to be measured, also known as the gap spacing, many parameters can be derived.
Figure 2.2: Example of a CTLM graph [4]

Figure 2.2 shows the parameters that can be derived through CTLM. A linear regression is drawn from the measured data. In this linear regression a resistance can be calculated for a gap spacing of 0. This resistance is twice the contact resistance ($R_c$), because the current passes from the electrode to the film and then from the film to the other electrode. The linear regression can be extended to the negative gap spacing where the resistance is 0. This is twice the transfer length ($L_T$), which is the length of the electrode to tested material contact where there is current flowing through both materials. The gradient of the linear regression is the sheet resistance ($R_{sh}$) over the circumference of the inner electrode ($W_c = 2\pi R_1$). By multiplying $R_{sh}$ with the thickness of the film, the resistivity ($\rho$) is obtained. By calculating the regression coefficient $R^2$, one can indicate how good the measurements are. In the ideal case $R^2$ would be one.
2.3 Fabrication of test structures

![Cross section of the wafers](image)

**Figure 2.3:** Cross section of the wafers [5]

The wafers on which the measurements took place were created by M. Yang. It was created by means of H WALD and is described in her paper [6]. The masks were designed by F.J. van der Velde and are described in his master thesis [5].

Figure 2.3 shows a section of the wafer. On the bottom in an aluminum contact. Above that is a highly doped silicon layer. These layers act as a gate that can be used in field effect testing. Above that is a silicon oxide layer, which acts as an isolator. Above that is a platinum layer, which is used for the electrodes. Above that is the tungsten layer. This is the material that is tested. The thickness of this layer is different on each wafer, namely 0.57, 0.9, 1.6, 2.85 and 8.5nm. Finally there is a layer of amorphous silicon, which is there to prevent the W from oxidizing [7].

In order to perform measurements, needles are applied to the electrodes. These needles need to be applied with some force to penetrate the two layers above it. This will damage the protective amorphous silicon layer and oxidation might occur faster at that position than at other positions. To prevent prior measurements from interfering thusly, measurements will only be made at locations that have not been measured before.
(a) Mask as used for all wafers. The numbers in black are for identification only and not part of the mask design.

(b) Matrix of CTLM structures with rows and columns identified

(c) CTLM structures

Figure 2.4: Wafer mask and groups of test structures [5]
All wafers have been made with the mask as shown in figure 2.4(a). In this thesis only the CTLM structure will be tested and they are shown in figures 2.4(b) and 2.4(c). The gap spacing between the inner and outer electrode varies between 2.5 and 300 µm. In the A row of the CTLM matrix, the inner electrode has a diameter of 125 µm. In the B and C rows that is 100 and 75 µm respectively.

CTLM structure coordinates on the wafer are determined first by the number of the matrix it is in and then by the row and column within that matrix. For example, the top right CTLM structures are labeled 4A3.

The wafers were manufactured in mid 2016 and measured in late 2017 and early 2018.

2.4 Measurement setup

Figure 2.5: Measurement setup. On the left is a Temptronic temperature regulator. In the center is a probe station with a microscope, probe needles and a chuck with a wafer on it. On the right is a Keithley 4200 semiconductor characterization system.

The measurement setup used for these experiments, is the Cascade setup at the University of Twente, which can be seen in figure 2.5. It consists of a microscope, four needles on which voltages and currents can be applied and measured and a temperature regulator.

In these tests two needles are used, one on the center electrode and one on the big outside electrode, as can be seen in figure 2.1. The voltage applied between those needles varies between -20 and +20 volt. To protect the wafers, the current is limited to 100mA. The tests where temperature related phenomena are not tested, are all performed at 25°C. When those phenomena are tested, the temperature is varied between 0 and 100°C.
Chapter 3

Measurements

3.1 Visual inspection

The first test I did was a visual inspection under the microscope. I noticed a lot of irregularities on the wafers, namely:

- Transparent circles of varying size that have rainbow colored edges.

- Gray circles of various size.

- Small black spots in various shapes.

- CTLM structures without a center electrode.

- CTLM structures where the center electrode was at least partially connected to the outer electrode.

Figure 3.1 shows two of those irregularities. The circles in that figure show some similarity in appearance to condensation, but heating up the wafer did not get rid of them.
3.2 Sanity Checks

3.2.1 Influence of the tests

In order to make sure that the measurements themselves do not influence the material, the same test is run several times in repeating order. This is performed on the thinnest W with the narrowest CTLM gap, 0.57nm and 2.5μm respectively. This is because the narrowest gap will cause both the highest current and electrical field and the thinnest W layer is the most susceptible to being changed or damaged during the test. During these tests the voltage applied between the electrodes is varied between -20 and +20 volt and then back to -20 volt, to check for hysteresis effects. This test is repeated 4 times.
3.2. Sanity Checks

Figure 3.2: Repeated I/V measurements of 2.5µm CTLM structure of 0.57nm thick W

Figure 3.2 shows that the difference between those 4 measurements is not huge, but on that scale it is not possible to see if there is a small scale effect. Therefore in figure 3.3(a) the difference between the run from -20 to +20 volt and the run from +20 to -20 volt is plotted. There is a relatively large difference at low voltages, probably related to how the measurements are performed when crossing 0 volt. Other than that a systematic but very small (in the order of 0.1% of the total current) difference can be seen.

Figure 3.3: Comparison between I/V measurements of 2.5µm CTLM structure of 0.57nm thick W

Finally, in figure 3.3(b) the difference between the first and subsequent runs is shown. The graph shows that the resistance has been slightly increased between the first and subsequent runs, but that again the difference is very small (again in
the order of 0.1% of the total current). This leads to the conclusion that testing does not alter the material in a significant manner.

### 3.2.2 Uniformity of the wafer

The results of the measurements should be independent of the position on the wafer. To test this, the same structure, in this case the CTLM structure with a 10 µm gap and 0.57 nm thickness, is tested at various locations on the wafer.

![Figure 3.4](image)

**Figure 3.4:** I/V characteristics of 0.57 nm thick W measured on CTLM structures with 10 µm gap spacing at various places on the wafer.

Figure 3.4 shows the results of this test. Clearly there is no uniformity. The difference between the measurements on location 4a1 and 14a1 is about a factor 30. Though those are the locations that provide the extreme results, neither of them appears to be an outlier.

At this point it is impossible to say whether this difference is due to non-uniform aging, or if it has always existed (the original report by van der Velde [5] does not mention testing at different locations), but either way it does not seem possible to provide accurate characteristics about W from these wafers.
3.3  I/V measurements at room temperature

While the sanity tests have shown that there is non-uniformity on the wafer, it is still possible that certain parameters, for instance transfer length, are the same regardless of position. To test this, every I/V measurement has been performed on different positions on each wafer. Figure 3.5 shows one such measurement for every wafer.

![Tungsten I/V Characteristics](image)

(a) 0.57nm at position 7A1
CHAPTER 3. MEASUREMENTS

Tungsten I/V Characteristics
width: 0.9nm

(b) 0.9nm at position 6A1

Tungsten I/V Characteristics
width: 1.6nm

(c) 1.6nm at position 1A2
3.3. I/V MEASUREMENTS AT ROOM TEMPERATURE

Tungsten I/V Characteristics
width: 2.85nm

(d) 2.85nm at position 3A1

Tungsten I/V Characteristics
width: 8.5nm

(e) 8.5nm at position 2A1

Figure 3.5: W I/V characteristics at various film thicknesses. Note that figures 3.5(a) and 3.5(b) have a different horizontal scale than the other figures.
The expected result would be that as the film thickness increases, the I/V relation would become more linear and the conductivity would increase. However, the 1.6nm graph (3.5(c)) shows more linearity and conductivity than the 2.85nm graph (3.5(d)). A possible cause for that result is that at the tested location, the film at the wafer that should have a W thickness of 1.6nm, is thicker than at the wafer that should have a thickness of 2.85nm.

After applying the correction factor for CTLM measurements, the results are plotted in figure 3.6.

(a) 0.57nm: $\rho$ values of 0.184 and 0.730 $\Omega$ m
(b) 0.9nm: $\rho$ values of 7.33 and 148 $\mu\Omega$ m
(c) 1.6nm: $\rho$ values of 1.05 and 4.74 $\mu\Omega$ m
(d) 2.85nm: $\rho$ values of 3.40 and 20.3 $\mu\Omega$ m
3.3. I/V Measurements at Room Temperature

(e) 8.5nm: $\rho$ values of 2.30 and 2.85 $\mu\Omega m$

**Figure 3.6:** Resistance plotted against CTML gap spacing for W of various thicknesses. The correction factor as a result of the gap spacing has already been applied.

In figure 3.6 the CTLM results of measurements on two positions of every film thickness are plotted. The squares are the measurement points and the solid lines are the linear regression of those measurements.

<table>
<thead>
<tr>
<th>Thickness [nm]</th>
<th>0.57</th>
<th>0.9</th>
<th>1.6</th>
<th>2.85</th>
<th>8.5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Location Test 1</td>
<td>7A1</td>
<td>6A1</td>
<td>1A2</td>
<td>3A1</td>
<td>2A1</td>
</tr>
<tr>
<td>Location Test 2</td>
<td>12A2</td>
<td>3A1</td>
<td>19A2</td>
<td>19A1</td>
<td>8A1</td>
</tr>
<tr>
<td>$R_c$ Test 1 [Ω]</td>
<td>$1.13 \times 10^6$</td>
<td>$1.95 \times 10^4$</td>
<td>14.1</td>
<td>46.8</td>
<td>10.3</td>
</tr>
<tr>
<td>$R_c$ Test 2 [Ω]</td>
<td>$4.01 \times 10^6$</td>
<td>$1.13 \times 10^6$</td>
<td>71.0</td>
<td>17.6</td>
<td>10.5</td>
</tr>
<tr>
<td>$L_T$ Test 1 [μm]</td>
<td>1.48</td>
<td>1.02</td>
<td>9.09</td>
<td>2.78</td>
<td>13.1</td>
</tr>
<tr>
<td>$L_T$ Test 2 [μm]</td>
<td>1.33</td>
<td>2.91</td>
<td>10.2</td>
<td>6.26</td>
<td>16.4</td>
</tr>
<tr>
<td>$R^2$ Test 1</td>
<td>0.987</td>
<td>0.994</td>
<td>0.983</td>
<td>0.990</td>
<td>0.995</td>
</tr>
<tr>
<td>$R^2$ Test 2</td>
<td>0.990</td>
<td>0.948</td>
<td>0.967</td>
<td>0.987</td>
<td>0.971</td>
</tr>
<tr>
<td>$R_{sh}$ Test 1 [Ω]</td>
<td>$3.24 \times 10^8$</td>
<td>$8.14 \times 10^6$</td>
<td>656</td>
<td>$7.13 \times 10^3$</td>
<td>335</td>
</tr>
<tr>
<td>$R_{sh}$ Test 2 [Ω]</td>
<td>$1.28 \times 10^7$</td>
<td>$1.65 \times 10^8$</td>
<td>$2.96 \times 10^3$</td>
<td>$1.19 \times 10^3$</td>
<td>271</td>
</tr>
<tr>
<td>$\rho$ Test 1 [Ωm]</td>
<td>0.184</td>
<td>$7.33 \times 10^{-3}$</td>
<td>$1.05 \times 10^{-6}$</td>
<td>$2.03 \times 10^{-5}$</td>
<td>$2.85 \times 10^{-6}$</td>
</tr>
<tr>
<td>$\rho$ Test 2 [Ωm]</td>
<td>0.730</td>
<td>0.148</td>
<td>$4.74 \times 10^{-6}$</td>
<td>$3.40 \times 10^{-6}$</td>
<td>$2.30 \times 10^{-6}$</td>
</tr>
</tbody>
</table>

**Table 3.1:** Parameters derived from the plots in figure 3.6

*R$^2$ is the regression coefficient

The parameters that can be derived from figure 3.6 are plotted in table 3.1. Figure 3.6(a) through 3.6(d) show a very large difference between the measurements on the two different locations. If the W was homogeneous, one would see an increased resistance when the gap spacing is increased. This is not always the case (for
instance on the 0.9nm thick measurements at location 3A1 with gap spacing 30 and 40 μm). This shows that there is a difference even between W layers that are positionally very close to each other.

As all the parameters depend on a linear regression of measurements where the only difference is the gap spacing and the previous paragraph shows that there are other differences, none of the parameters are reliable.

### 3.3.1 $Rt^2$ results

![Figure 3.7: $Rt^2$ results for different gap spacings](image)

Figure 3.7 shows the resistance times the film thickness squared, measured at different CTLM gap spacings. There are two lines in each plot to show both the high and low measured resistance. There is some difference between the plots, but in all the
3.4. Temperature effects

3.4.1 TCR Values

W α values
Thickness: 0.57nm, Gap spacing 10μm

(a) 0.57nm
W $\alpha$ values
Thickness: 0.9nm. Gap spacing 10$\mu$m

(b) 0.9nm

W $\alpha$ values
Thickness: 1.6nm. Gap spacing 10$\mu$m

(c) 1.6nm
3.4. TEMPERATURE EFFECTS

Figure 3.8: TCR values of W at various thicknesses and voltages
Figure 3.8 shows the TCR values of W at various thicknesses and voltages. All those measurements were taken at CTLM structures with a gap spacing of 10 \( \mu \)m. With the exception of the 8.5nm thick W films, all the TCR’s are negative, indicating that a higher temperature leads to a lower resistivity. There are two factors that play a role in the 8.5nm positive TCR. First, it is the thickest film being measured, so it will most closely resemble the thick-film behavior, which has a positive TCR. Second, that film has the lowest resistance (in the order of 20\( \Omega \)), meaning that the contact resistance of the probes to the electrodes plays a big part. This can change between the different tests, as the needles may not be applied with the same pressure. It can be shown that this plays a factor by the big difference in the TCR values between the different values.

The TCR values of 1.6nm at 75\(^\circ\)C and 2.85nm at 100\(^\circ\)C vary significantly from the other values at their respective thicknesses. This might also be due to different probe contact resistances.

The TCR value is voltage dependent, with the biggest voltage dependency shown at the 1.6nm thickness. In films up to 1.6nm in thickness, a lower voltage causes a higher absolute value of the TCR.

### 3.4.2 Comparisons at various gap spacings and temperatures

(a) 0.57nm at 1A2: \( \alpha \) between -5.74 and -8.97 

(b) 0.9nm at 2A1: \( \alpha \) between -5.23 and -8.51 m\(^2\)C\(^{-1}\)
3.4. TEMPERATURE EFFECTS

(c) 1.6nm at 19A3: $\alpha$ between 0.312 and 0.810 m$^\circ$C$^{-1}$

(d) 2.85nm at 1A2: $\alpha$ between -1.01 and -1.11 m$^\circ$C$^{-1}$

(e) 8.5nm at 19A3: $\alpha$ between -1.98 and +0.196 m$^\circ$C$^{-1}$

Figure 3.9: Comparisons of W resistance at various thicknesses and temperatures

In figure 3.9, as in figure 3.5, the squares show the measurements and the solid lines the linear regression of those measurements.

<table>
<thead>
<tr>
<th>Temperature[°C]</th>
<th>0</th>
<th>25</th>
<th>50</th>
<th>75</th>
<th>100</th>
</tr>
</thead>
<tbody>
<tr>
<td>$L_T$ [μm]</td>
<td>7.92</td>
<td>7.77</td>
<td>6.60</td>
<td>6.55</td>
<td>6.26</td>
</tr>
<tr>
<td>$R_c$[Ω]</td>
<td>$2.37 \times 10^7$</td>
<td>$1.80 \times 10^7$</td>
<td>$1.29 \times 10^7$</td>
<td>$1.02 \times 10^7$</td>
<td>$7.99 \times 10^6$</td>
</tr>
<tr>
<td>$R^{2\star}$</td>
<td>0.773</td>
<td>0.780</td>
<td>0.808</td>
<td>0.823</td>
<td>0.833</td>
</tr>
<tr>
<td>$R_{sh}$ [Ω]</td>
<td>$1.27 \times 10^9$</td>
<td>$9.84 \times 10^8$</td>
<td>$8.31 \times 10^8$</td>
<td>$6.60 \times 10^8$</td>
<td>$5.41 \times 10^8$</td>
</tr>
<tr>
<td>$\rho$ [Ωm]</td>
<td>0.724</td>
<td>0.561</td>
<td>0.474</td>
<td>0.376</td>
<td>0.309</td>
</tr>
<tr>
<td>$\alpha$ [°C$^{-1}$]</td>
<td>-</td>
<td>$-8.97 \times 10^{-3}$</td>
<td>$-6.90 \times 10^{-3}$</td>
<td>$-6.40 \times 10^{-3}$</td>
<td>$-5.74 \times 10^{-3}$</td>
</tr>
</tbody>
</table>

Table 3.2: Result from measurements of 0.57nm thick W at location 1a2 as plotted in 3.9(a)
## Chapter 3. Measurements

### Table 3.3: Result from measurements of 0.9nm thick W at location 2a1 as plotted in 3.9(b)

<table>
<thead>
<tr>
<th>Temperature[°C]</th>
<th>0</th>
<th>25</th>
<th>50</th>
<th>75</th>
<th>100</th>
</tr>
</thead>
<tbody>
<tr>
<td>$L_T [\mu m]$</td>
<td>13.6</td>
<td>13.4</td>
<td>12.4</td>
<td>11.7</td>
<td>11.0</td>
</tr>
<tr>
<td>$R_c [\Omega]$</td>
<td>$1.58 \times 10^7$</td>
<td>$1.22 \times 10^7$</td>
<td>$9.46 \times 10^6$</td>
<td>$7.56 \times 10^6$</td>
<td>$6.10 \times 10^6$</td>
</tr>
<tr>
<td>$R^2*$</td>
<td>0.671</td>
<td>0.670</td>
<td>0.719</td>
<td>0.733</td>
<td>0.752</td>
</tr>
<tr>
<td>$R_{sh} [\Omega]$</td>
<td>$4.93 \times 10^8$</td>
<td>$3.88 \times 10^8$</td>
<td>$3.24 \times 10^8$</td>
<td>$2.74 \times 10^8$</td>
<td>$2.35 \times 10^8$</td>
</tr>
<tr>
<td>$\rho [\Omega m]$</td>
<td>0.444</td>
<td>0.349</td>
<td>0.291</td>
<td>0.247</td>
<td>0.212</td>
</tr>
<tr>
<td>$\alpha [°C^{-1}]$</td>
<td>-</td>
<td>$-8.51 \times 10^{-3}$</td>
<td>$-6.88 \times 10^{-3}$</td>
<td>$-5.93 \times 10^{-3}$</td>
<td>$-5.23 \times 10^{-3}$</td>
</tr>
</tbody>
</table>

### Table 3.4: Result from measurements of 1.6nm thick W at location 19a3 as plotted in 3.9(c)

<table>
<thead>
<tr>
<th>Temperature[°C]</th>
<th>0</th>
<th>25</th>
<th>50</th>
<th>75</th>
<th>100</th>
</tr>
</thead>
<tbody>
<tr>
<td>$L_T [\mu m]$</td>
<td>10.0</td>
<td>9.61</td>
<td>9.19</td>
<td>8.26</td>
<td>7.59</td>
</tr>
<tr>
<td>$R_c [\Omega]$</td>
<td>59.0</td>
<td>57.1</td>
<td>54.9</td>
<td>51.2</td>
<td>48.3</td>
</tr>
<tr>
<td>$R^2*$</td>
<td>0.983</td>
<td>0.984</td>
<td>0.988</td>
<td>0.992</td>
<td>0.993</td>
</tr>
<tr>
<td>$R_{sh} [\Omega]$</td>
<td>$2.50 \times 10^3$</td>
<td>$2.52 \times 10^3$</td>
<td>$2.54 \times 10^3$</td>
<td>$2.63 \times 10^3$</td>
<td>$2.70 \times 10^3$</td>
</tr>
<tr>
<td>$\rho [\Omega m]$</td>
<td>$3.99 \times 10^{-6}$</td>
<td>$4.03 \times 10^{-6}$</td>
<td>$4.06 \times 10^{-6}$</td>
<td>$4.21 \times 10^{-6}$</td>
<td>$4.32 \times 10^{-6}$</td>
</tr>
<tr>
<td>$\alpha [°C^{-1}]$</td>
<td>-</td>
<td>$3.72 \times 10^{-4}$</td>
<td>$3.12 \times 10^{-4}$</td>
<td>$7.09 \times 10^{-4}$</td>
<td>$8.10 \times 10^{-4}$</td>
</tr>
</tbody>
</table>

### Table 3.5: Result from measurements of 2.85nm thick W at location 1a2 as plotted in 3.9(d)

<table>
<thead>
<tr>
<th>Temperature[°C]</th>
<th>0</th>
<th>25</th>
<th>50</th>
<th>75</th>
<th>100</th>
</tr>
</thead>
<tbody>
<tr>
<td>$L_T [\mu m]$</td>
<td>5.43</td>
<td>5.37</td>
<td>5.23</td>
<td>5.22</td>
<td>5.50</td>
</tr>
<tr>
<td>$R_c [\Omega]$</td>
<td>115</td>
<td>110</td>
<td>105</td>
<td>102</td>
<td>104</td>
</tr>
<tr>
<td>$R^2*$</td>
<td>0.919</td>
<td>0.921</td>
<td>0.924</td>
<td>0.926</td>
<td>0.924</td>
</tr>
<tr>
<td>$R_{sh} [\Omega]$</td>
<td>$8.96 \times 10^3$</td>
<td>$8.71 \times 10^3$</td>
<td>$8.50 \times 10^3$</td>
<td>$8.27 \times 10^3$</td>
<td>$8.02 \times 10^3$</td>
</tr>
<tr>
<td>$\rho [\Omega m]$</td>
<td>$2.55 \times 10^{-5}$</td>
<td>$2.48 \times 10^{-5}$</td>
<td>$2.42 \times 10^{-5}$</td>
<td>$2.36 \times 10^{-5}$</td>
<td>$2.28 \times 10^{-5}$</td>
</tr>
<tr>
<td>$\alpha [°C^{-1}]$</td>
<td>-</td>
<td>$-1.11 \times 10^{-3}$</td>
<td>$-1.01 \times 10^{-3}$</td>
<td>$-1.02 \times 10^{-3}$</td>
<td>$-1.05 \times 10^{-3}$</td>
</tr>
</tbody>
</table>
3.4. Temperature Effects

<table>
<thead>
<tr>
<th>Temperature[°C]</th>
<th>0</th>
<th>25</th>
<th>50</th>
<th>75</th>
<th>100</th>
</tr>
</thead>
<tbody>
<tr>
<td>$L_T$ [µm]</td>
<td>12.0</td>
<td>13.0</td>
<td>14.0</td>
<td>10.6</td>
<td>10.9</td>
</tr>
<tr>
<td>$R_c$ [Ω]</td>
<td>8.31</td>
<td>8.54</td>
<td>8.70</td>
<td>7.46</td>
<td>7.58</td>
</tr>
<tr>
<td>$R^2*$</td>
<td>0.983</td>
<td>0.995</td>
<td>0.993</td>
<td>0.988</td>
<td>9.84</td>
</tr>
<tr>
<td>$R_{sh}$ [Ω]</td>
<td>293</td>
<td>279</td>
<td>264</td>
<td>298</td>
<td>295</td>
</tr>
<tr>
<td>$ρ$ [Ωm]</td>
<td>$2.49 \times 10^{-6}$</td>
<td>$2.37 \times 10^{-6}$</td>
<td>$2.25 \times 10^{-6}$</td>
<td>$2.53 \times 10^{-6}$</td>
<td>$2.51 \times 10^{-6}$</td>
</tr>
<tr>
<td>$α$ [°C$^{-1}$]</td>
<td>-</td>
<td>-1.92 $\times 10^{-3}$</td>
<td>-1.98 $\times 10^{-3}$</td>
<td>1.96 $\times 10^{-4}$</td>
<td>5.59 $\times 10^{-5}$</td>
</tr>
</tbody>
</table>

Table 3.6: Result from measurements of 8.5nm thick W at location 19a3 as plotted in 3.9(e)

*R$^2$ is the regression coefficient

Figure 3.9 shows the resistance of W at various thicknesses and temperatures. The parameters from these measurements can be found in tables 3.2 through 3.6. For the two thinnest films, 0.57nm (figure 3.9(a)) and 0.9nm (figure 3.9(b)), the resistivity and contact resistance consistently reduce with an increased temperature. For the thicker films this is not consistently true.
Conclusions and recommendations

4.1 Conclusions

There is a lot of difference between the W at various positions on the wafers. It is unknown if this is due to differences in thickness and/or impurities that have persisted since they were manufactured, or if it is due to non-homogeneous aging. As such, the other conclusions are tentative.

- W films transfer from semi-continuous to continuous at a thickness of approximately 1.6nm.
- W films of a thickness up to at least 0.9nm, have a highly non-linear I/V relationship.
- W films of a thickness up to at least 0.9nm, have a negative TCR.
- W films of a thickness up to at least 0.9nm, have a voltage dependent TCR, with lower voltages providing a higher absolute value of TCR.
- W films of a thickness up to at least 0.9nm, have a contact resistance that decreases with an increased temperature.

4.2 Recommendations

To evaluate if HWALD produces a homogeneous W film, new wafers should be made. Shortly after production, measurements should take place on different locations and both the results and locations should be documented.

Those wafers should be made at various thicknesses around 1.6nm, so that the percolation threshold can be determined with greater accuracy.

To evaluate the aging process of W films, the measurements should be repeated at set intervals. If previous measurements found that the W films were not homogeneous, the measurements should be repeated at the same locations. If they are homogeneous, it is preferable to measure at different locations, as the measurement
probes pierce the protective amorphous silicon layer. The damage to that layer could cause an accelerated rate of oxidation.
Chapter 5

Acknowledgments

I would like to thank F.J. van der Velden and Mengdi Yang for designing and making the wafers respectively.

I would like to thank Satadal Duta for introducing me to the probe station and reserving time at it for me.

I would like to thank Alexey Kovalgin for supervising me during the thesis.


Appendix A

Matlab scripts

A.1 I/V characteristics

Matlab script that was used to generate figure 3.6 and parameters in table 3.1. In this case the values of the 0.57nm tests have been entered.

clear all
clf
x=[50,40,30,20,10,5,2.5]; %gap spacings in um
r = 0.57; %thickness in nm
rad=62.5; %Radius of inner electrode in um
cor=rad./x .* log((rad+x)/rad); %correction factor for CTLM
%resistances
y1=[2.87E+07,2.58E+07,2.28E+07,1.68E+07,9.31E+06,5.32E+06,2.49E+06];
z1=[1.15E+08,9.94E+07,8.86E+07,6.49E+07,3.75E+07,1.91E+07,1.02E+07];
y2=y1./cor;
z2=z1./cor;
hold on;
p=polyfit(x,y2,1); %generates straight line
q=polyfit(x,z2,1);
x2=[-max([p(2)/p(1) q(2)/q(1)]),50];
y3=polyval(p,x2);
z3=polyval(q,x2);
plot(x2,y3,'red');
plot(x2,z3,'blue');
plot(x,y2,'s', 'MarkerEdgeColor', 'red');
plot(x,z2,'s', 'MarkerEdgeColor', 'blue');
ax = gca;
ax.YAxisLocation = 'origin';
A.2 Temperature dependencies

Matlab script that was used to generate figure 3.9 and parameters in tables 3.2 through 3.6. In this case the values of the 0.57nm tests have been entered.

clear all
clf
x=[50,40,30,20,10,5,2.5]; %gap spacicings in um
t = 0.57; %thickness in nm
rad=62.5; %Radius of inner electrode in um
cor=rad./x .* log((rad+x)/rad); %correction factor for CTLM
%resistances
a1=[5.55E+07,4.47E+07,5.81E+07,4.60E+07,3.01E+07,1.72E+07,8.77E+06];
a2=[6.72E+07,5.61E+07,7.16E+07,5.85E+07,3.72E+07,2.14E+07,1.09E+07];
a3=[8.50E+07,6.92E+07,9.34E+07,7.36E+07,4.68E+07,2.71E+07,1.37E+07];
a4=[1.01E+08,8.98E+07,1.12E+08,9.82E+07,6.25E+07,3.50E+07,1.77E+07];
a5=[1.35E+08,1.10E+08,1.49E+08,1.22E+08,8.53E+07,4.64E+07,2.36E+07];
b1=a1./cor;
b2=a2./cor;
b3=a3./cor;
A.2. TEMPERATURE DEPENDENCIES

\[b_4 = a_4 / \text{cor};\]
\[b_5 = a_5 / \text{cor};\]
hold on;
plot(x, b_5,'s', 'MarkerEdgeColor', 'red');
plot(x, b_4,'s', 'MarkerEdgeColor', 'green');
plot(x, b_3,'s', 'MarkerEdgeColor', 'blue');
plot(x, b_2,'s', 'MarkerEdgeColor', 'cyan');
plot(x, b_1,'s', 'MarkerEdgeColor', 'magenta');
c_1 = \text{polyfit}(x, b_1, 1); % generates straight line
c_2 = \text{polyfit}(x, b_2, 1);
c_3 = \text{polyfit}(x, b_3, 1);
c_4 = \text{polyfit}(x, b_4, 1);
c_5 = \text{polyfit}(x, b_5, 1);
d = [-\text{max}([c_1(2)/c_1(1) c_2(2)/c_2(1) c_3(2)/c_3(1) c_4(2)/c_4(1) c_5(2)/c_5(1)]), 50];
e_1 = \text{polyval}(c_1, d);
e_2 = \text{polyval}(c_2, d);
e_3 = \text{polyval}(c_3, d);
e_4 = \text{polyval}(c_4, d);
e_5 = \text{polyval}(c_5, d);
plot(d, e_5,'red');
plot(d, e_4,'green');
plot(d, e_3,'blue');
plot(d, e_2,'cyan');
plot(d, e_1,'magenta');
ax = gca;
ax.YAxisLocation = 'origin';
xlabel('gap spacing [\mu m]');
ylabel('Resistance [\Omega]');
legend('0C','25C','50C','75C','100C','location','northwest');
title([\text{num2str}(t), 'nm']);
ylim([0 \text{ max}([e_1(2) e_2(2) e_3(2) e_4(2) e_5(2)])]);
xlim([d(1) inf]);
f_1 = \text{polyval}(c_1, x);
f_2 = \text{polyval}(c_2, x);
f_3 = \text{polyval}(c_3, x);
f_4 = \text{polyval}(c_4, x);
f_5 = \text{polyval}(c_5, x);
Rsq1 = 1 - \text{sum}((b_1 - f_1).^2) / \text{sum}((b_1 - \text{mean}(b_1)).^2); % regression coefficient
Rsq2 = 1 - \text{sum}((b_2 - f_2).^2) / \text{sum}((b_2 - \text{mean}(b_2)).^2);
Rsq3 = 1 - sum((b3 - f3).^2)/sum((b3 - mean(b3)).^2);
Rsq4 = 1 - sum((b4 - f4).^2)/sum((b4 - mean(b4)).^2);
Rsq5 = 1 - sum((b5 - f5).^2)/sum((b5 - mean(b5)).^2);
Rsh1 = c1(1)*(2*pi*rad); % sheet resistance
Rsh2 = c2(1)*(2*pi*rad);
Rsh3 = c3(1)*(2*pi*rad);
Rsh4 = c4(1)*(2*pi*rad);
Rsh5 = c5(1)*(2*pi*rad);
rho1 = Rsh1 * t * 10^-9; % resistivity
rho2 = Rsh2 * t * 10^-9;
rho3 = Rsh3 * t * 10^-9;
rho4 = Rsh4 * t * 10^-9;
rho5 = Rsh5 * t * 10^-9;
alpha4 = (rho4/rho5-1)/25;
alpha3 = (rho3/rho5-1)/50;
alpha2 = (rho2/rho5-1)/75;
alpha1 = (rho1/rho5-1)/100;
Rc1 = c1(2)/2;
Rc2 = c2(2)/2;
Rc3 = c3(2)/2;
Rc4 = c4(2)/2;
Rc5 = c5(2)/2;
Lt1 = c1(2)/c1(1)/2;
Lt2 = c2(2)/c2(1)/2;
Lt3 = c3(2)/c3(1)/2;
Lt4 = c4(2)/c4(1)/2;
Lt5 = c5(2)/c5(1)/2;