Fundamentals of Experimental Physics

Experiment B1,
Vacuum thermal evaporation deposition

Group 12

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1 Principle

1.1 Vacuum thermal evaporation deposition

Thermal evaporation involves heating a solid material inside a high vacuum chamber, which produces some vapor pressure. Inside the high vacuum, even a relatively low vapor pressure is sufficient to raise a vapor stream, which go through the mask and hits the substrate, sticking to it as a film.

To heat the source material, one method, often referred to as Filament Evaporation, is a simple electrical resistive heat element, tungsten boat in our experiment, which has high melting point. The filament source offers the safety of low voltage, although very high current is required, usually several hundred amps.

The mean free path of gas is sensitive to the vacuum degree, $\lambda = 5 \times 10^{-3}/P$, so if the vacuum degree is low, the mean free path of gas will be small, which will be scattered frequently by other molecules, then the quality of thin film would be low.

To know the thickness of the thin film, one use the quartz crystal monitoring system. When a mechanical force is applied to a quartz crystal, a electric field is generated in the corresponding direction in the quartz crystal, in turn, a quartz crystal will be mechanically deformed under a electric field. This is phenomenon called piezoeletrc effect. Now apply a alternating electric field to the quartz crystal, it will produce mechanical vibration, also an alternating electric field, due to the deformation of crystal, which is normally small. The amplitude will increase sharply only when the frequency of the applied alternating voltage is equal to a certain frequency.This phenomenon is called piezoelectric resonance and the specific frequency is called nature frequency of the quartz crystal, which on size and cutting angle of the crystal.

1.2 Van der PAUW four terminal measurement

To measure the resistivity of material with arbitrary shape, one can use van der PAUW four terminal measurement, once it satisfies:

1. the four contacts locate are located on the edge of device
2. the contact points are very small
3. the thickness of device is uniform
4. the surface of device is singly-connected

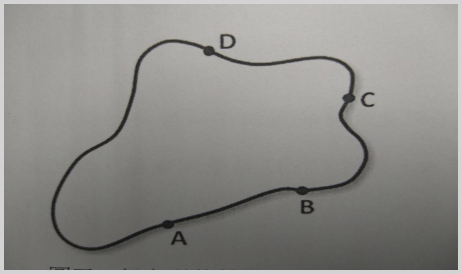

Figure 1: the device with arbitrary shape

Then according to Fig1, one can obtain the following relation

$$e^{-\pi R_{AB,CD} d/\rho} + e^{-\pi R_{BC,DA} d/\rho} = 1 \tag{1}$$

where ρ is the resistivity, d is the thickness of the device. $R_{AB,CD}$ is the resistance measured by making current in A,B while measuring voltage in C,D.

proof

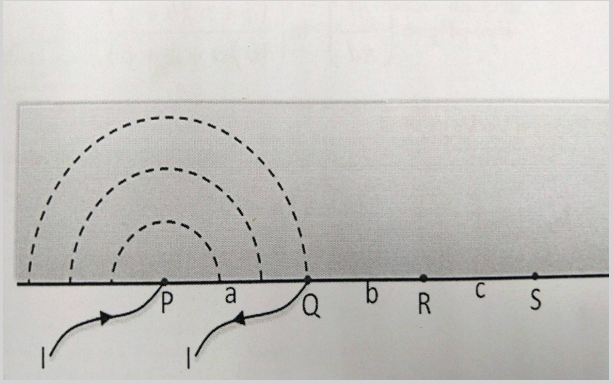

Figure 2: A infinite half plane, the dash line is the potential contour

Consider a infinite half plane, with four contact P,Q,R,S separated by a,b,c respectively. Assume the current flow from P to infinity, then flow back from infinity to Q uniformly. For any equipotential surface, the total current is I

$$J\pi r d = I \tag{2}$$

According to Ohm's law

$$E = \rho J = \frac{\rho I}{\pi r d} \tag{3}$$

So the potential difference of S and R due to the flowing out current is

$$\begin{aligned} V_{S,P} - V_{R,P} &= -\int E dr \\ &= -\frac{\rho I}{\pi d} \int \frac{1}{r} dr \\ &= \frac{\rho I}{\pi d} \ln\left(\frac{a+b+c}{a+b}\right) \end{aligned} \tag{4}$$

Similarly, consider the current flow from infinity to Q,

$$V_{S,Q} - V_{R,Q} = \frac{\rho I}{\pi d} \ln\left(\frac{b+c}{b}\right) \tag{5}$$

Adding eq(4) and eq(5), we get

$$V_S - V_R = \frac{\rho I}{\pi d} \ln\left(\frac{(a+b)(b+c)}{b(a+b+c)}\right) \tag{6}$$

then

$$R_{PQ,RS} = \frac{V_S - V_R}{I} = \frac{\rho}{\pi d} \ln\left(\frac{(a+b)(b+c)}{b(a+b+c)}\right) \tag{7}$$

By the same way

$$R_{QR,SP} = \frac{\rho}{\pi d} \ln\left(\frac{(a+b)(b+c)}{ca}\right) \tag{8}$$

Combine eq(4) and eq(5), one get eq(1). Then one can use conformal mapping to prove that eq(1) is true for any surface.

2 Method

To fulfill the principle talked above, we separate our experiment into four part, getting a clean glass, vacuuming, heating and measuring.

- Getting a clean glass
  It's important because Sn we want to coat on the glass is really thin. ANY impurities can cause huge error. The way we do is cut suitable size of glass and use water, cleaner, alcohol to clean it and dry it.
- Vacuuming and Heating
  This is the most important part of the experiment.
  And we use mechanical pump to lower the atmosphere and then use the diffusion pump and mechanical pump to finish the work together. So the steps are
  - Close all 3. And open bleed valve 1 and close
  - Open 1 to make mechanical pump work
  - Close 1 and open 2 to prepare for the diffusion pump
  - Open the heater to warming up
  - Open 1 to use mechanical pump help diffusion one
  - Close 1 and open 3 to use mechanical pump and diffusion one at the same time
- Heating and giving voltage and remove the shutter to coat
- Use van der PAUW and four-terminal measurement to check the properties of what we get

3 Result

3.1 Raw Data

In the experiment, we use four-point measurement for "Mask" and van der pauw method for others to measure the resistivity of S_n in different thickness. In each measurement, we measure five times and calculate the average value and uncertainty. (Assume the mean value distribution is Gaussian.)

$$\mu = \frac{1}{5}\sum_{n=1}^{5} x_n, \quad \sigma_\mu = \frac{s}{\sqrt{5}}$$

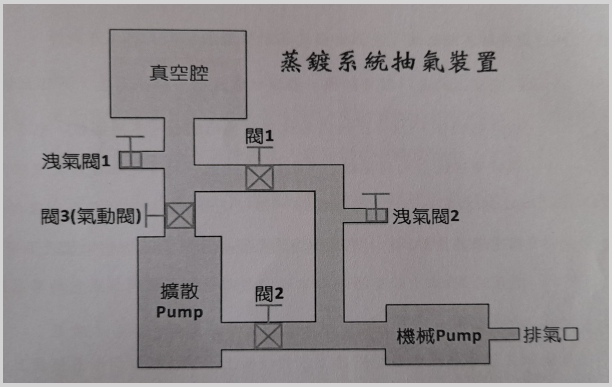

Figure 3: diagram of vacuuming pump

where x_n represents the measurement of each time and s is the sample standard deviation:

$$s^2 = \sum_{n=1}^{5} \frac{(x_n - \mu)^2}{5-1}$$

The following are the data:

- Week 1 (1000 $\mathring{A}$), circular sample:

Measurement (Ω)	1	2	3	4	5	Average
$R_{AB,CD}$	0.18143	0.18139	0.18141	0.18137	0.18139	0.18140 ± 0.00001
$R_{BC,DA}$	0.24665	0.24671	0.24672	0.24674	0.24676	0.24672 ± 0.00002

- Week 1 (1000 $\mathring{A}$), "Mask" sample:

Measurement (Ω)	1	2	3	4	5	Average
R	5.04	5.35	5.32	5.01	5.01	5.15 ± 0.077

- Week 2 (500 $\mathring{A}$), circular sample:

Measurement (Ω)	1	2	3	4	5	Average
$R_{AB,CD}$	3.0153	3.0142	3.0130	3.0120	3.0110	3.0131 ± 0.0008
$R_{BC,DA}$	2.3463	2.3459	2.3457	2.3455	2.3450	2.3457 ± 0.0002

- Week 2 (500 $\mathring{A}$), "Mask" sample:

Measurement (Ω)	1	2	3	4	5	Average
R	17.0000	16.9800	16.9427	16.9418	16.9312	16.9591 ± 0.0131

- Week 2 ($0.1mm$), bulk material:

Measurement ($m\Omega$)	1	2	3	4	5	Average
$R_{AB,CD}$	0.30000	0.30869	0.30923	0.31062	0.31137	0.30798 ± 0.00205
$R_{BC,AD}$	0.21652	0.21976	0.22171	0.22227	0.22228	0.22050 ± 0.00110

3.2 Analysis

For four-point measurement method, we can use Ohm's Law $\rho = \frac{RA}{l}$ to calculate the resistivity. (For "Mask": $l = 11.5 \pm 0.02(mm)$, $A = d * 3 \pm 0.02(mm)$. And for van der paw method, we use $e^{-\pi R_{AB,CD} d/\rho} + e^{-\pi R_{BC,AD} d/\rho} = 1$ to get the resistivity. (where l is length of the "Mask", A is the cross-sectional area of the "Mask" and d is the thickness of the sample.) After analysis, we get:

Table	W1(circular)	W1(Mask)	W2(circular)	W2(Mask)	W2(bulk)
$d(nm)$	101.2	101.2	50.7	50.7	0.130 (mm)
$\rho\ (n\Omega \cdot m)$	97.38	136.	612.	224.	154.

3.3 Error Propagation

The error of ρ would be the function of d, $A, R, R_{AB,CD}, R_{BC,AD}, l$. More specifically, we have:

$$\sigma^2_{rho,\,four-point} = (\frac{\partial \rho}{\partial A})^2 \sigma_A^2 + (\frac{\partial \rho}{\partial l})^2 \sigma_l^2 + (\frac{\partial \rho}{\partial R})^2 \sigma_R^2.$$

and

$$\sigma^2_{rho,\,van} = (\frac{\partial \rho}{\partial R_{AB,CD}})^2 \sigma^2_{R_{AB,CD}} + (\frac{\partial \rho}{\partial R_{BC,AD}})^2 \sigma^2_{R_{BC,AD}} + (\frac{\partial \rho}{\partial d})^2 \sigma_d^2.$$

- For four-point measurement, we can calculate the uncertainty of resistivity by:

$$\sigma_\rho^2 = (\frac{A}{l})^2 \sigma_R^2 + (\frac{R}{l})^2 \sigma_A^2 + (\frac{RA}{l^2})^2 \sigma_l^2$$

  After calculation, we get $\sigma_\rho(W1) = 0.93\,(n\Omega \cdot m)$ and $\sigma_\rho(W2) = 3.0\,(n\Omega \cdot m)$.

- For van der pauw method, we have (let $R_{AB,CD} = R_1$, $R_{AC,BD} = R_2$):

$$\frac{\partial \rho}{\partial R_1} = \frac{e^{-\pi R_1 d/\rho}}{R_1 e^{-\pi R_1 d/\rho} + R_2 e^{-\pi R_2 d/\rho}} \quad \frac{\partial \rho}{\partial R_2} = \frac{e^{-\pi R_2 d/\rho}}{R_2 e^{-\pi R_1 d/\rho} + R_2 e^{-\pi R_2 d/\rho}} \quad \frac{\partial \rho}{\partial d} = \frac{\rho}{d}$$

  Thus, we get $\sigma_\rho(W1) = 0.069\,(n\Omega{\cdot}m)$, $\sigma_\rho(W2,\ "circular") = 0.60\,(n\Omega{\cdot}m)$ and $\sigma_\rho(W2,\ "Bulk") = 7.3\,(n\Omega \cdot m)$.

Hence, our result becomes:

Table	W1(circular)	W1(Mask)	W2(circular)	W2(Mask)	W2(bulk)
$\rho\ (n\Omega \cdot m)$	97.38 ± 0.069	136. ± 0.93	612. ± 0.60	224. ± 3.0	154. ± 7.3
Error (Percentage)	0.071 %	0.68 %	0.098 %	1.3 %	4.74 %

3.4 Discussion

For $1000\mathring{A}$ and $0.1mm$ thin tin film, the recognized value of resistivity is about $115(n\Omega \cdot m)$. For the result of W1(Mask) and W2(bulk), the resistivity is higher than the recognized value. We think this is caused by some other resistance such as the wire and the indium. Besides, the bulk material tin has been used for many times. There might be some high resistivity impurities inside it. As for the W1(circular), we measured a lower value. We think that this might be caused by our manipulation of van der pauw measurement. We might put the wires too far from the edge to cause this kind of phenomenon.

For $500\mathring{A}$, due to the quantum effect, the resistivity becomes larger as the layer becomes thinner. Our results for $500\mathring{A}$ thin film are indeed larger than the $1000\mathring{A}$ and $0.1mm$, which is compatible to the theory.

4 Question & Discussion

- Please simply explain other method and principle of vacuum pumping.
  - Ion pump[1]
    The elements of ion pump is that there are many cylindrical cells, which is anode, between cathode plate. While anode connected to the positive voltage and cathode connected to the ground. Also, there are parallel magnetic field.

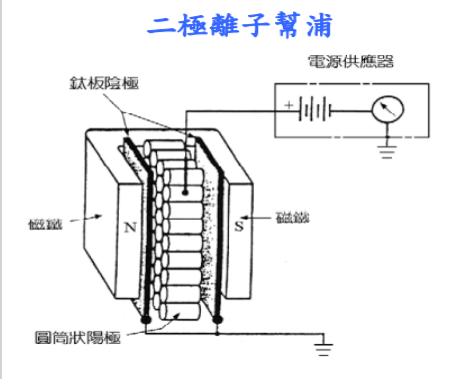

Figure 4: Diagram of ion pump

The principle of it is just like sputtering. Use the electron in the air to hit the gas molecular to ionize them. After the gas being ionized, they will be attracted by anode. So they fly and stick to the cylindrical cells. In this case the gas in that space will become less and less thus vacuum.

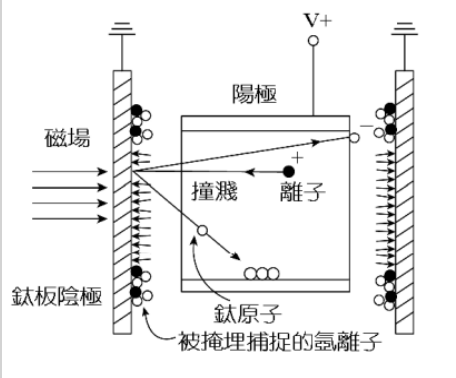

Figure 5: The principle of ion pump

- Getter pump
  The main concept of it is to create chemical reaction thus let gas becomes solid and stick to the material we put in. Common material is like Ta, Nb, Zr, Mo, W.

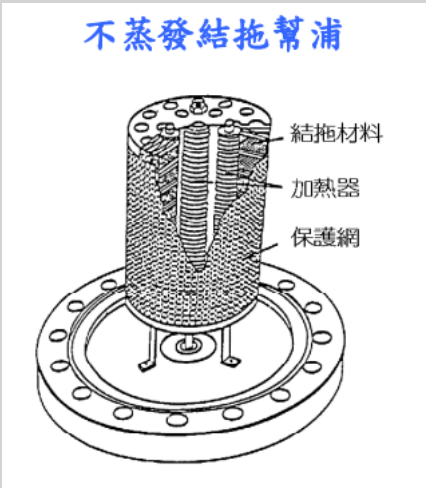

Figure 6: diagram of Getter pump

- Except for vacuum thermal evaporation deposition, what is the other way to coating.
  - There is still one way called sputtering[2]. The principle of it is like we mentioned above. The overall principle is that we accelerate the gas we have like Ar^+ to hit the target on cathode side. Once the target is hit, the ion of it will be attracted by anode and thus stick on it.
    According to the method of accelerating gas, we can classify them into DC magnetron of sputtering and AC magnetron of sputtering. And if the gas we use at the beginning is not just Ar^+ but also the target we want, this is so called reactive sputtering.

- Please explain the difference among two-points measurement, four-points measurement and Van der Pauw method.
  - Two-points measurement (Shown in the R.H.S of the following figure): In two-points measurement method, the source of electricity and the resistance measuring points share the same two wires connecting to the sample. Therefore, if the current is a little bit unstable, the value of ohmmeter would be fluctuating. Although there are some slightly disadvantage, two-points measurement is widely-used. In two-points measurement, Ohm's Law has been used.
  - Four-points measurement (Shown in the L.H.S of the following figure): In four-points measurement method, the current source is separated form the ohmmeter. It is used for measuring a more precisely resistance of samples, such as a thin film. Due to the disconnection of current source and ohmmeter, the value of the ohmmeter would be more stable. Same as four-points measurement, Ohm's Law has been used in the four-points measurement.
  - Van der Pauw method: Different form the above measuring technique, van der pauw method is used to deal with the samples which cross-sectional areas are not constant, such as circular shape. Van der Pauw method is a kind of four-points measurement method. One should first label arbitary four edge points as A, B, C, D. (ABCD should be continuous.) Next, one needs to calculate the resistance of CD with current flowing through AB and the resistance of DA with current flowing through BC. And finally, by using the equation $e^{-\pi R_{AB,CD}/R_{sample}} + e^{-\pi R_{BC,DA}/R_{sample}} = 1$ to calculate the resistivity or resistance (R_{sample}) of the sample.

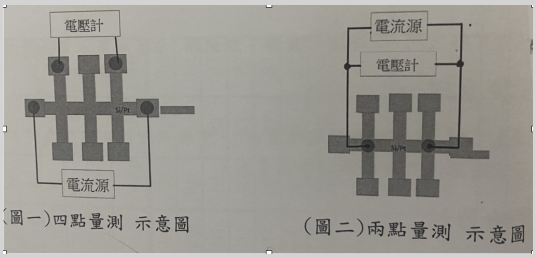

Figure 7: **L**:two-points measurement **R**:four-points measurement

5 Conclusion

In this experiment, we fabricated four tin samples, circular and masked with thickness 507$\mathring{A}$ and 1012$\mathring{A}$, by vacuum thermal evaporation deposition. Then we apply four terminal measurements for masked device and van der PAUW measurement for circular device to obtain the resistivity of tin. Finally we get the result: circular(1012$\mathring{A}$): $97.38 \pm 0.069(n\Omega \cdot m)$, Mask(1012$\mathring{A}$):136. $\pm$ 0.93, circular(507$\mathring{A}$): 612. $\pm$ 0.60, Mask(507$\mathring{A}$):224. $\pm$ 3.0, bulk(0.13mm):154. $\pm$ 7.3, with error 0.017%, 0.68%, 0.098%, 1.3%, 4.74% respectively. For 1000$\mathring{A}$ and 0.1nm tin thin film, the recognized value of resistivity is about $115(n\Omega \cdot m)$. Due to the quantum effect, the resistivity becomes larger as the layer become thinner, which is consistent with our results of 500$\mathring{A}$ samples. Some errors might be

caused by the impurity of our samples, and the imperfection of our manipulation of van der PAUW measurement.

6 Reference

References

[1] http://www.phys.nthu.edu.tw/~thschang/notes/VAC03.pdf

[2] Textbook of fundamental physics experiment, 111

[3] https://www.quora.com/Why-do-we-use-the-four-probe-method-instead-of-the-two-probe-method

[4] https://www.yxcxtal.com/news-detail/i-27