


Fundamentals of Micro and Nanofabrication
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Lecture - 24
Physical Vapor Deposition: Evaporation

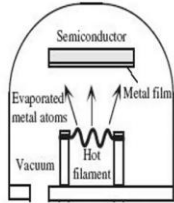
This module is on physical vapor deposition. In the previous lecture, we looked at some jargon and introduced the molecular flow regime. In this lecture, we will focus on one of the simplest and the first Physical Vapor Deposition techniques, Evaporation.

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Evaporation: Basics

- Idea:
 - Evaporate metal by melting it. Vapors then condense on substrate
- Procedure:
 - Convert metal from solid to vapor phase by melting + evaporation
 - Transport gaseous metal to substrate
 - Condense gaseous metal on substrate
- Done in high vacuum ($10^{-6} - 10^{-7}$ Torr)
 - To prevent chemical reactions
 - Oxygen/water may react with hot metal atoms
 - If needed, can force a reaction by flowing gas near the crucible
 - To get good material efficiency



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The basic idea of evaporation is that you have a filament in the vacuum chamber. It heats the material that you want to deposit to an extent where the material melts or sublimates. That creates vapors, and these evaporated atoms transport in line-of-sight, onto the substrate and condense. The same thing happens if you put a lid on a boiling water container. If you pick it up after 2 minutes, you see water sticking to the underside.

You would want to maintain a high vacuum, $\sim 10^{-7}$ - 10^{-6} torr during the evaporation to prevent chemical reactions because the evaporated (metal) atoms can often be very reactive. For example, aluminum vapors can very easily react with oxygen and deposit aluminum oxide instead of aluminum. To prevent that, you need to keep the background or the contaminant pressure low. You can also do reactive evaporation. To deposit aluminum oxide, you can leak in a little bit of oxygen during metal evaporation. Often,

the distance between the substrate and the filament is pretty small, for material efficiency. If you make it too short, however, you get non-uniformity.

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Evaporation: Need for High Vacuum

Blue particles: vapor, oxygen, nitrogen and carbon dioxide in the air
Red particles: materials of the film

- At low vacuum, source particles collide with trace gasses
 - Leading to scattering + chemical reaction
- Only at high vacuum can we get high deposition rate + purity


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Apart from purity, there is another need for high-vacuum, highlighted in this figure. If you don't care about contamination, oxidation, or are depositing a metal that does not oxidize, you may be okay with high pressure inside the evaporation chamber. At high pressures, however, the mean free path will be very tiny. When the metal atoms evaporate now, because of the interaction with the gas already present in the chamber, they will suffer collisions as the kinetic theory of gases predicts. The short mean free path means a lot of the evaporated atoms get scattered in various directions. You expect them to come out and go straight to the substrate, but a lot of them will go left and right, get scattered at weird angles, and you will waste a lot of material.

If you reduce the pressure and get high-vacuum, you get very long mean free paths. These red atoms will not interact with any of the blue atoms, and you get line-of-sight deposition with higher material efficiency. You do evaporation in high-vacuum not only to reduce contamination but to increase the material efficiency as well.

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Evaporation: Influence of Background Pressure



- Pressure sets the number of monolayers reaching substrate
- Chamber pressure due to
 - Melt: Vapor pressure
 - Residual gasses: Base pressure
- Arrival ratio: Number of source atoms reaching substrate for every impurity (gas) atom
 - Should be $\gg 1$

Pressure (mBar)	Z_A ($\text{cm}^{-2}\text{s}^{-1}$) Monolayer/s	Monolayer time
1	$\sim 10^5$	$\sim 10 \mu\text{s}$
10^{-3}	$\sim 10^2$	$\sim 10 \text{ms}$
10^{-6}	$\sim 10^{-1}$	$\sim 10 \text{s}$
10^{-9}	$\sim 10^{-4}$	$\sim 30 \text{h}$

Base Pressure (mBar)	Melt Vapor Pressure (mBar)	Arrival Ratio
10^{-3}	10^{-4}	10^{-1}
10^{-4}	10^{-4}	1
10^{-5}	10^{-4}	10^1
10^{-6}	10^{-4}	10^2
10^{-7}	10^{-4}	10^3
10^{-8}	10^{-4}	10^4

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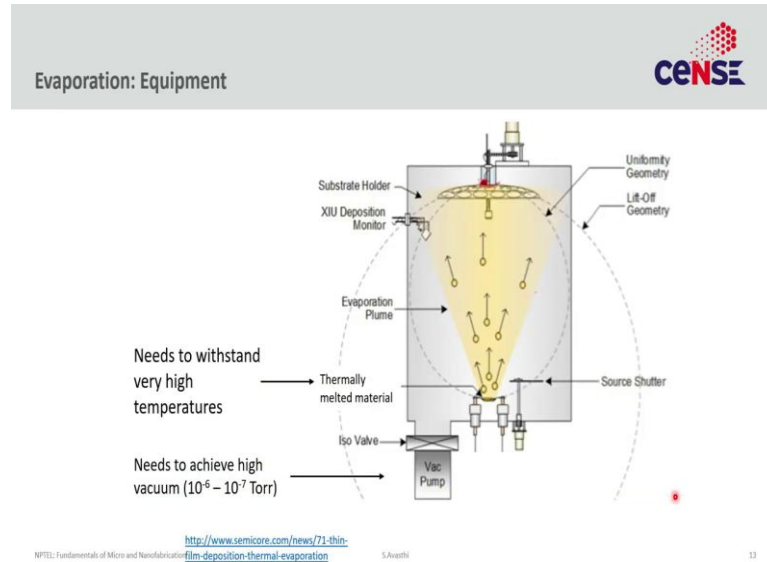
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While doing evaporation, you would like to be cognizant of time to a monolayer or impingement rate. At 10^{-6} mbar, the monolayer time is ~ 10 s. In 10 s, trace gases present in the chamber would form a monolayer if the sticking coefficient is 1. That is the worst-case scenario. During this time, you also deposit the desired material. If the deposition rate is 1 nm/s, you deposit 10 nm of the desired film in the time it takes the impurities to form a monolayer. In 10 nm, in the worst case, you get one monolayer of impurities. For purer films, either increase the growth rate or lower the pressure, which increases monolayer time. So, for the same thickness and the growth rate, you have fewer impurities. Arrival ratio is the number of source atoms arriving at the substrate for one impurity atoms. For pure films, this should be $\gg 1$. For a 99.99 % pure film, the arrival ratio should be 10000.

The table lists the arrival ratio for various base pressures, at a given melt vapor pressure. For a vapor pressure of 10^{-4} mbar, and base pressure 10^{-3} mbar, there will be ten times more impurities than source atoms, and arrival ratio - 0.1. You can't do evaporation under low-vacuum conditions. Only when you get to 10^{-6} mbar, you have an arrival ratio of around 100 or 1000, close to the 99.9 % type of purity. It also highlights why it is very challenging to get a device-quality film through evaporation. No matter how hard you try, you never get 6-7 N purity in the film, which CVD routinely gets. That is the limitation or the physical vapor process. The background pressure always introduces

impurities that are extremely hard to reduce unless you get an ultra-high vacuum, which is very hard and expensive.


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Typical evaporation equipment looks like this figure taken from this reference. You have a vacuum pump, an isolation valve, and a way of creating vapors. You can use electric current to resistively heat the material, which melts and vapors come out. The deposition is often far from the source. You mount multiple wafers because it takes a lot of time to get to the base pressures. You might as well coat them in one shot.

There are two circular regions here. One is for uniform geometry, and one is for lift-off geometry. We will not discuss it further right now. You must place this thermally melted material in a crucible that can hold it without itself melting. The vacuum system must be capable to get to 10^{-7} - 10^{-6} mbar.

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Evaporation: Evaporation Rate

- Impingement rate (Z_M) can also be used to describe loss of vapor from melt, i.e. mass evaporation rate (R_{ME} , unit: gm/cm²s)

$$Z_M = \frac{P_e}{\sqrt{2\pi m k_B T}} \Rightarrow R_{ME} = \sqrt{\frac{m}{2\pi k_B T}} (P_e - P_b)$$

— P_e is the vapor pressure of the melt
— P_b is the base pressure
— m is the gram-molecular mass
— T is the temperature (K)

Strongly depends on T

- Loss of mass (R_{ML} , unit: gm/s) from a melt of area A_M

$$R_{ML} = \sqrt{\frac{m}{2\pi k_B}} \int \frac{P_e - P_b}{\sqrt{T}} dA \approx \sqrt{\frac{m}{2\pi k_B}} \frac{P_e - P_b}{\sqrt{T}} A_M$$

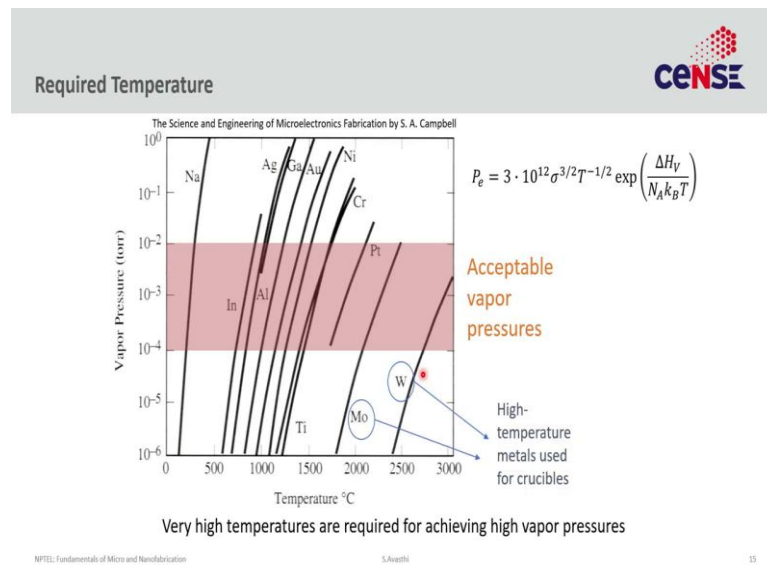
- The vapor pressure depends very strongly on the temperature
— In order to have a reasonable evaporation rate (0.01-1 μm/min), the vapor pressure must be about 10^{-4} - 10^{-2} Torr

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The evaporation rate is a function of the pressure, as well as the temperature of the material. The impingement rate here is not of the background gas or impurity. It is the rate at which the vapor leaves the melt. That depends on the pressure and inversely on the temperature. You can calculate a mass evaporation rate. P_e is the equilibrium vapor pressure of the melt. You also have a base pressure P_b in the chamber when you pump it down. The mass evaporation rate depends on the difference between P_e and P_b . The base pressure should be as small as possible for purity. The mass loss depends on the melt surface area. Larger crucibles have more mass loss and a higher deposition rate.

You don't need to remember any of these formulae, however, remember that the vapor pressure has an extreme temperature dependence. The temperature dependence of the evaporation rate is not weak polynomial as this formula on first sight suggests. Hidden inside this P_e is a very strong function of temperature.


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Here is a plot of the vapor pressure of various metals at different temperatures. With a very small change in temperature, P_e goes from 10^{-6} to 10^{-2} mbar. In high-vacuum, for the arrival ratios > 100 , you need the vapor pressure $> 10^{-4}$ mbar. It can be as high as 10^{-2} mbar, but above that, we run into the issue of too little mean free path and low material efficiency. You can see the acceptable range of vapor pressures highlighted in the figure. You need to heat the different elements at different temperatures for vapor pressure to be in that range. Sodium or indium would require a very lower temperature, while platinum would require a much higher temperature. Different metals require different amounts of power for evaporation.

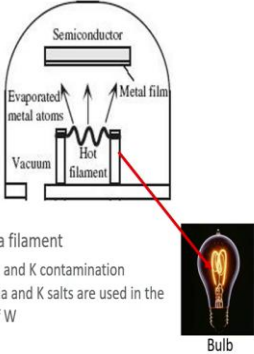
Notice that tungsten and molybdenum have very low vapor pressures, or require very high temperatures for appreciable vapor pressure. These are called refractory metals that can withstand very high temperatures. You often use them as crucibles to hold other metals while they evaporate. Everything has a vapor pressure. Put aluminum in a tungsten crucible and evaporate it at a temperature such that its vapor pressure is 10^{-3} mbar. At this temperature, what is the vapor pressure of tungsten? If at 1000°C , the aluminum has a vapor pressure 10^{-3} mbar, tungsten has a vapor pressure that is far below 10^{-6} , even 10^{-9} mbar, somewhere deep below this slide. The amount of tungsten coming out at that temperature is very small. Hence, the amount of tungsten impurity that you expect in the film is very small, as not enough tungsten evaporates at that temperature. Hence, you can use tungsten and molybdenum as boats.

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Two Methods of Heating Material

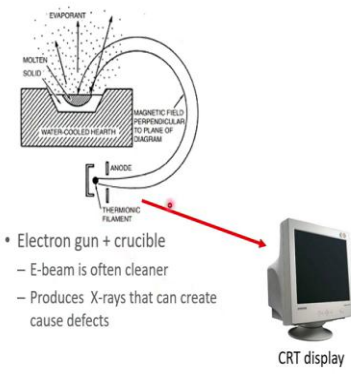
Resistive Filament



- W, Mo or Ta filament
- Causes Na and K contamination because Na and K salts are used in the refining of W

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Electron-Beam



- Electron gun + crucible
- E-beam is often cleaner
- Produces X-rays that can create cause defects

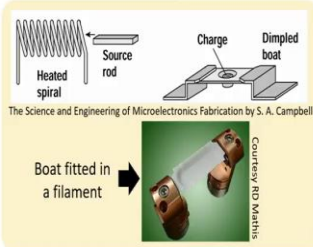
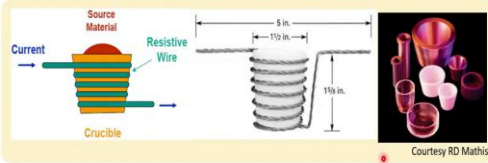
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Resistive heating is one of the simplest methods. The problem with it is these refractive elements often have some trace contaminants. While molybdenum or tungsten may not come out from this hot filament at typical temperatures, the impurities within them might. For example, tungsten has sodium impurities. Sodium has a very high vapor pressure. Any amount of sodium impurity in the tungsten will come out during evaporation, and that is a problem. That is why, people have moved away from resistive filament approach to an electron beam, which is incident on the top of the material. Only some volume at the top melts locally. You generate the electron beam in the vacuum and steer it on top of the crucible using magnetic fields.

The rest of the metal and the crucible is kept cool by a water-cooled hearth. The crucible stays at a much lower temperature than the metal. That further assures that any impurity in the crucible would not come out because it is at even lower temperature, and has lower vapor pressure. The disadvantage of this technique is the electron beams hitting the metal generate X-rays, which can damage sensitive devices. These things look esoteric, but you have probably seen them in the routine. When an incandescent bulb becomes defective, and you see the darkening of the glass, that is because the filament evaporates slowly and coats the glass. In the cathode ray tube displays, the cathode ray is nothing, but a beam of electrons. You focus the beam on top of the metal using the same technique that you use in the CRT.

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Evaporation: Resistive Heating



Courtesy RD Mathis

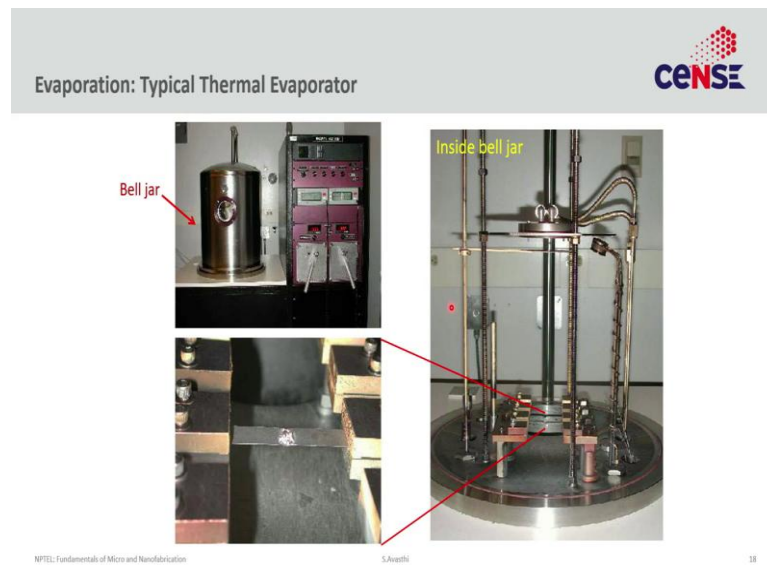
- Used for materials with high vapor pressure at $< 1600\text{ C}$
- Common evaporated materials:
 - Au, Ag, Al, Sn, Cr, Sb, Ge, In, Mg, Ga;
 - CdS, PbS, CdSe, NaCl, KCl, AgCl, MgF₂, CaF₂, PbCl₂.

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For resistive heating, you typically have refractory metals like tungsten, tantalum, molybdenum, etcetera. They come in these shapes that you can purchase from vendors. Sometimes you need a crucible to hold a small metal piece to melt and evaporate. To make them also, you can use refractory materials like alumina, boron nitride, or graphite. You can also make boats instead of a wire. You can have a tungsten boat with a dimple to keep some material, avoiding the need for a crucible.

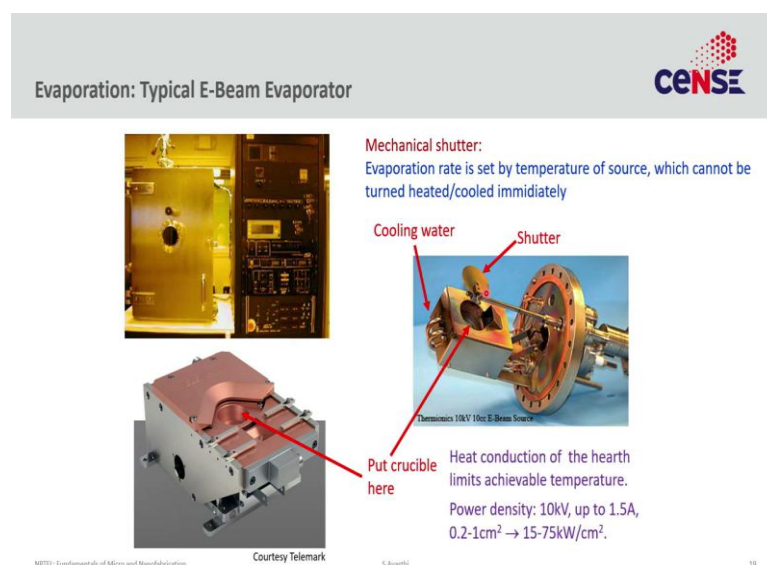
Out of the various ways, how do you select? Several vendors may give suggestions such as, to deposit gold, please use this type of crucible or this filament. You typically do not want alloying. For example, aluminum in tungsten forms an alloy. Tungsten is not the best boat for the aluminum evaporation. However, gold and silver do not alloy with tungsten. Tungsten is an excellent boat material for them. You need to be careful while selecting crucible materials, resistive elements, and the boat.

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Here is a typical thermal evaporator. You evacuate the steel or glass chamber. The two huge hunks of the copper inside the chamber are the electrical contacts. You put the boat in the middle, put some material, flow the current, and it heats up. There typically is a quartz monitor to measure the thickness of the film, so you have repeatability. You usually rotate the substrate to maintain uniformity.

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E - Beam evaporators are a little more complicated as they have water-cooled hearth inside the vacuum chamber. It is often made of copper to make it thermally conductive.

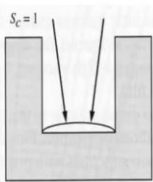
There are mechanical shutters to stop the deposition at will.

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Sticking Coefficient

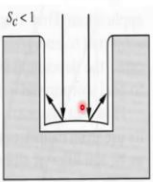


Sticking coefficient: $S_c = \frac{F_{stick}}{F_{incident}}$



$S_c = 1$

Sticking coefficient =1
All incident atoms stick to the surface perfectly



$S_c < 1$

Sticking coefficient << 1
Incident atoms do not stick to the surface. Get scattered.


Due to low temperature of substrate, for evaporation S_c is ~1

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When you have an influx of material, some of it stays on the surface, and some get backscattered. The amount that has stuck divided by the total incident amount is the sticking coefficient S_c . $S_c = 1$ means everything that comes, sticks, and for $S_c = 0$ nothing sticks. You melt the material in the crucible, so it is hot, but the substrate sits at room temperature and has negligible equilibrium vapor pressure as compared to the source. Any vapor incident on the substrate condenses. S_c during evaporation is often 1.

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Evaporation: Deposition Rate



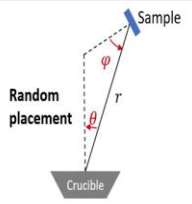
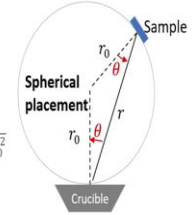
- Assume material travels in straight line to sample
 - Good assumption for long path lengths
- Also assume everything that makes to sample sticks
 - Sticking coefficient =1
- Mass flux arriving (gm/cm²s) at the sample

$$Z_S = \frac{\cos\theta \cos\varphi}{\pi r^2} R_{ML} = \frac{\cos\theta \cos\varphi}{\pi r^2} \sqrt{\frac{m}{2\pi k_B}} \frac{P_e - P_b}{\sqrt{T}} A_S$$

$$\Rightarrow \sqrt{\frac{m}{2\pi k_B}} \frac{P_e - P_b}{\sqrt{T}} \frac{A_S}{4\pi r_0^2}$$
- Deposition rate (cm/s) at the sample

$$R_S = \frac{\cos\theta \cos\varphi}{\pi r^2 \rho_S} \sqrt{\frac{m}{2\pi k_B}} \frac{P_e - P_b}{\sqrt{T}} A_S = \sqrt{\frac{m}{2\pi k_B}} \frac{P_e - P_b}{\sqrt{T}} \frac{A_S}{4\pi \rho_S r_0^2}$$

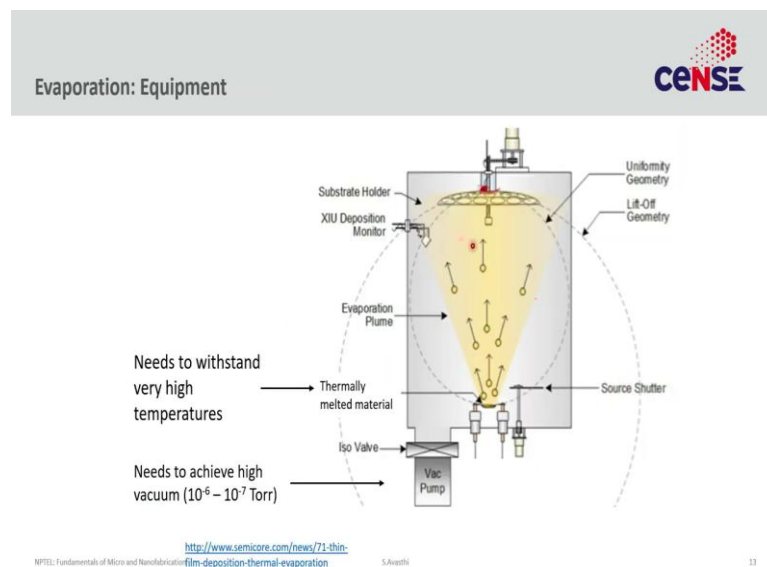
 - Deposition rate is the above expression divided by the density (ρ_S) of the material

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For practical purposes, assume that the crucible is a point source, and you have a sample mounted at distance r , at an angle ϕ and θ . The mass flux at the sample depends on r , ϕ , and θ . We will discuss a specific case where you place it on a sphere tangential to the crucible, called spherical placement. Both, the crucible, and the substrate are at a distance r_0 from the center, and $\phi = \theta$. In this case, the equation simplifies, and becomes independent of ϕ and θ , and depends only on r_0 . The sample placed anywhere on this imaginary sphere will get the same Z_s , independent of θ . To maintain uniformity across the substrates, place them on this spherical placement area.


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This is why the substrate holder is spherical, to make the deposition uniform on all the wafers.

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Evaporation: Non-uniformity



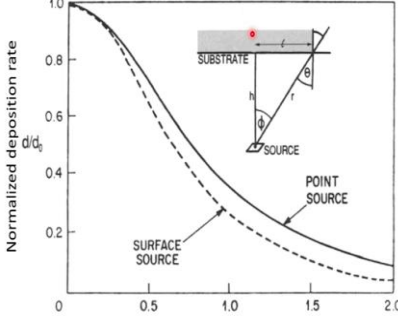
• Films are thicker at the center of the substrate

For point source

$$\frac{d}{d_0} = \frac{1}{\left\{1 + \left(\frac{l}{h}\right)^2\right\}^{3/2}}$$

For planer source

$$\frac{d}{d_0} = \frac{1}{\left\{1 + \left(\frac{l}{h}\right)^2\right\}}$$




Thin-Film Evaporation Processes by Milton Ohring

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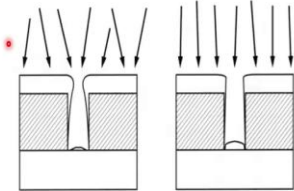
In the previous figure, we talked about wafer to wafer non-uniformity. Now, the wafer is not curved, but flat. So, the angle of the impingement of the incoming flux at the center is very different from the edge. There will be a θ dependence. You don't need to remember any of these formulas. The normalized deposition rate would be highest in the middle and fall as you go towards the edges. To solve this problem, make the h very large, so l/h becomes small. Keeping the substrate further away from the source, you make the thickness more uniform across the wafer, but the impingement rate falls. Given a substrate size, you decide the distance from uniformity versus deposition rate trade-off.

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Step Coverage at high λ & Large Aspect Ratio



- Angle of arrival affects step coverage
 - Wide arrival angle distribution leads to poor side/bottom coverage
 - Narrow arrival angle distribution leads to better side bottom coverage.
- Higher aspect ratio features need the narrow arrival-angle distribution for adequate coverage.



Wide arrival angle
distribution

Narrow arrival
angle distribution


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In reaction rate limited CVD, you often get very good step coverage if the temperature is uniform. But here, it doesn't matter, as the substrate is at room temperature. Here, the direction of the influx matters. If the influx is at arbitrary angles, you might not get coverage at the bottom of a high aspect-ratio feature, like sunlight in New York; Because of the tall buildings, while at the top, you get a lot of sunshine, down on the street level, not much reaches. Most of the incoming material gets stuck on the top, and very little reaches the bottom. The deposition at the bottom is very different from the top. You can solve this problem a little by making the influx very vertical. Even when it is vertical, very small changes in the angle, and you would not get deposition at the bottom.

Even when you get some deposition at the bottom, you get very little on the sides, because of the directional influx. If you tilt it to the left, you get some thickness on the left, but nothing on the right. It is very hard in evaporation to get a conformal deposition, which is very easy in ALD.

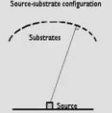
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Evaporation: Step Coverage

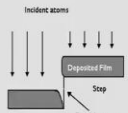


- Evaporation is very directional due to large λ
 - Conformality is poor
 - Poor conformality is sometime desirable, e.g. shadow masking and lift-off,
- Rotation of the substrate can improve step coverage.
- Heating substrate promotes surface diffusion
 - Improves step coverage and adhesion.

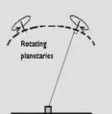
Source-substrate configuration



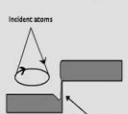
Incident atoms




Rotating planetaries



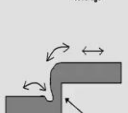
Incident atoms



Rotating planetaries
Substrate heaters



Incident atoms



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<http://cleanroom.byu.edu/metal>
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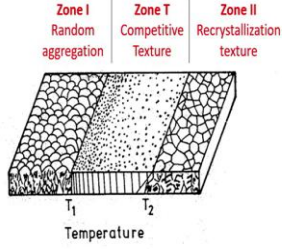
If you have a uniform flux, which is what you design a lot of these systems for, you would not get step coverage, but non-conformal deposition. It is suitable for lift-off, though. We will talk about it during etching. For conformal deposition, evaporation is not the best method. You can improve it a little by mounting the substrate at an angle and rotating it. It is called the planetary motion. Planetary motion keeps changing the angle of arrival, which gives you some side- deposition, but not perfectly conformal. Heating

the substrate can increase surface diffusion that enhances step coverage, and improves conformality.

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Nucleation and Growth: Structure Zone Model (SZM)

- Surface reorganization needs energy
 - Diffusion is temperature dependant
 - Important parameter is $\frac{T_{deposition}}{T_{melting}}$
- Near room temperature ($< 0.3T_m$), almost no reorganization happens
 - Get polycrystalline + porous
- At high temperature ($> 0.45T_m$), films crystallize similar to CVD
 - Get large-grained oriented films



	Zone I	Zone T	Zone 2
Metals	$< 0.3 T_m$	$0.3 - 0.45 T_m$	$> 0.45 T_m$
Oxides	$< 0.26 T_m$	$0.26 - 0.45 T_m$	$> 0.45 T_m$


B.A. Movchan, A.V. Demchishin, Fiz. Met. Metalloved 28 (1969) 83

The structure zone model from this paper describes the growth at different homologous temperatures in evaporation. Homologous temperature T_{sub}/T_m is the temperature of the material at the growth substrate divided by its melting point. Suppose you evaporate aluminum and hold the substrate at aluminum's melting point ($T_{sub}/T_m = 1$). The incoming aluminum will diffuse a lot on the surface, reorganize, find the lattice positions, and grow epitaxially. As you reduce T_{sub} , the energy that the substrate can provide to the incoming aluminum to orient itself reduces, and so does the crystallinity. You get progressively poor crystallinity until you get to polycrystalline film.

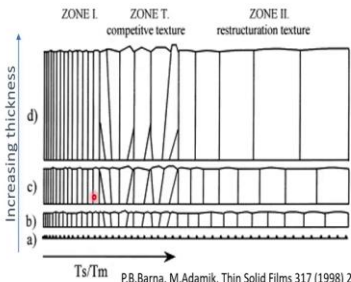
This nomenclature of zones - zone 1, zone T, and zone 2 is relatively modern. Why we call it 1, T, and 2, will become clear as we discuss sputtering. In zone 1, you have random agglomeration or a polycrystalline film. It happens if you deposit at $T_{sub}/T_m < 0.3$, or 30 % of the melting point. It is often the case. As we deposit metals at room temperature, we get polycrystalline films. In the $0.3 < T_{sub}/T_m < 0.45$ range, you start getting some texture, oriented grains, or columnar growth. Only above $T_{sub}/T_m = 0.45$, you start seeing large grain, near epitaxial films. In evaporation, if you want orientation and crystallinity, heat the substrate. Typically, at room temperature, you always get a polycrystalline, randomly oriented film.

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Competitive Texturing: Zone T



- In competitive texture, two orientation compete for growth
- Those with faster growth rate tower over the other orientations
 - Sunlight does not reach smaller building in New York
- Grains growing faster grow even faster
 - Positive feedback
- Ultimately (for thick enough films) one orientation dominates




Ts/Tm P.B.Barna, M.Adamik, Thin Solid Films 317 (1998) 27

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This figure shows the film's cross-section. In zone T, you get a columnar structure, but it changes with depth. At small thicknesses, you have different orientations. Some orientations grow better at the expense of others by shadowing. Suppose the growth rate of one orientation is 20 % higher than another. Even though they start at the same point, the one with a higher growth rate would become a little taller. Because it is a little taller, it would get more flux just like the taller building in New York see more sun. So, it will grow even taller. Faster growing orientation gets more flux and grows even faster. Competitive growth makes it win. Here, the kinetics decides the growth rate, not thermodynamics. In thick layers, the faster growth orientation wins out.

So, you get some texture, but in the cross-section, you see different types of orientations at the bottom. As you go further up, some of these orientations lose and got extinct. The one that grows the fastest keeps growing.

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Evaporation: Salient Features

<p>Advantages</p> <ul style="list-style-type: none">• With filaments, very little damage to Si• Deposited films are usually very pure• Limited step coverage• Easy	<p>Disadvantages</p> <ul style="list-style-type: none">• Some class of materials are difficult to evaporate<ul style="list-style-type: none">– Refractory metals (W, Mo)– High temperature dielectrics (Al₂O₃)– Alloys (Nicrome)– Compounds (SiO₂)• Limited step coverage• With e-beam, X-rays damage gate-oxides• With filament, Na/K contamination
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Let us end the lecture with the salient features of evaporation. The advantage is simplicity. It is one of the oldest PVD techniques. The films can be very pure, not as much as CVD, but say 99.99 % pure. It has limited step coverage, which can be advantageous or disadvantageous. The arrival energy of atoms is very low, so it is a very gentle process. You may think that since you are melting the source, the ejected atoms would cause some damage, but that is not the case. You can do it on very delicate films.

The disadvantage is, you can't evaporate refractory elements, high-temperature dielectrics, or alloys. Since the components of the alloy have different evaporation rates, the ratio of evaporation rates decides the film composition, not the source. If you melt nichrome, nickel will selectively evaporate more or chromium will. The stoichiometry of the film is decided not by their alloy composition but the ratio of their vapor pressures. Even compounds like silicon dioxide are tough unless you do reactive evaporation, which is a little tricky. If you use e-beam, you might cause X-ray damage. With filaments, you might get sodium or potassium contamination.

In the next lecture, we shall look at sputtering, which is the more modern form of physical vapor deposition and is becoming a little more common.