Fundamentals of Micro and Nanofabrication

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Lecture - 19

Chemical Vapor Deposition: Nucleation and Growth

This is the third lecture in the continuing series of CVD in the module of additive processing and we are looking into the details of how chemical vapor deposition allows us to grow high-quality epitaxial films. In the previous lectures, we have looked at some basic definitions of conformality, step coverage, etc… and a brief introduction of the CVD mechanism, specifically the role that reactor design plays on the growth. We discussed hot walled CVD, cold walled CVD, atmospheric pressure CVD, and low-pressure CVD and different advantages and disadvantages of these techniques.

In this lecture, we will get into more details of the thermodynamics of the growth and look at the types of (surface) reactions and how they affect CVD growth. We will talk about nucleation and try to understand how grains form, films grow and what decides whether you get large grains or small grains which decides whether you get a 3D growth or a 2D growth.

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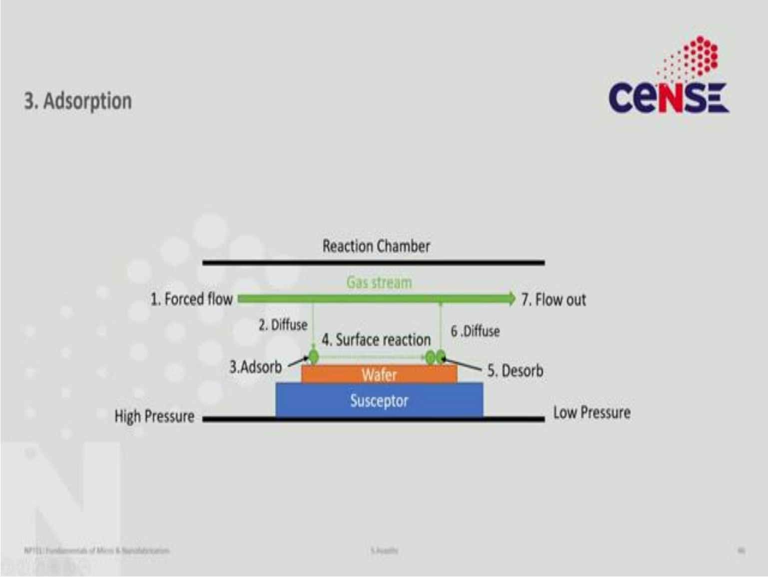

3. Adsorption

This is where we left in the previous lecture. We had finished the module on gas flow and discussed how diffusion through the gas allows you to get the precursors to the surface of the wafer where the reaction can occur. Next, we will talk about adsorption, as the precursor makes its way to the wafer, it must get adsorbed on the surface.

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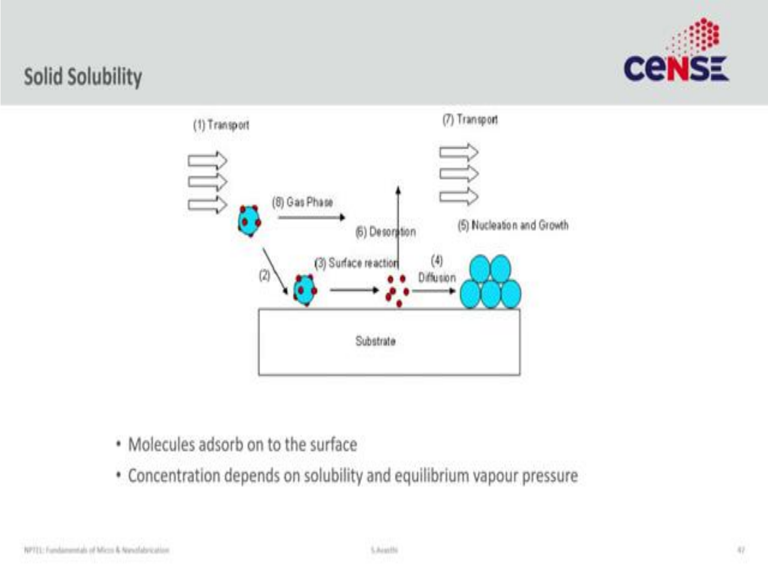

Solid Solubility

- Molecules adsorb on to the surface
- Concentration depends on solubility and equilibrium vapour pressure

Every precursor has a certain amount of solubility on the surface that decides the maximum concentration on the surface, which in turn decides the growth. All you have to do is pick up a handbook and figure out the solubility and equilibrium vapor pressure.

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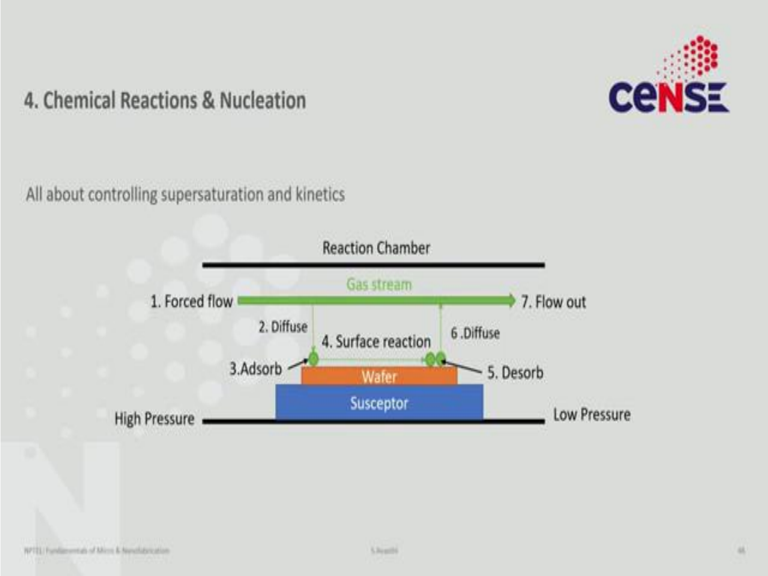

4. Chemical Reactions & Nucleation

All about controlling supersaturation and kinetics

Once the precursor has adsorbed on the surface, it must undergo a chemical reaction that would eventually form the material of the desired film. The material that forms is in molecular form. Those molecules have to adjust on the surface to form a crystal given that we are depositing solid films. This diffusion or reorganization of the molecules on the surface can be fairly non-trivial. We will look at some of those details under the concepts of surface reaction and nucleation.

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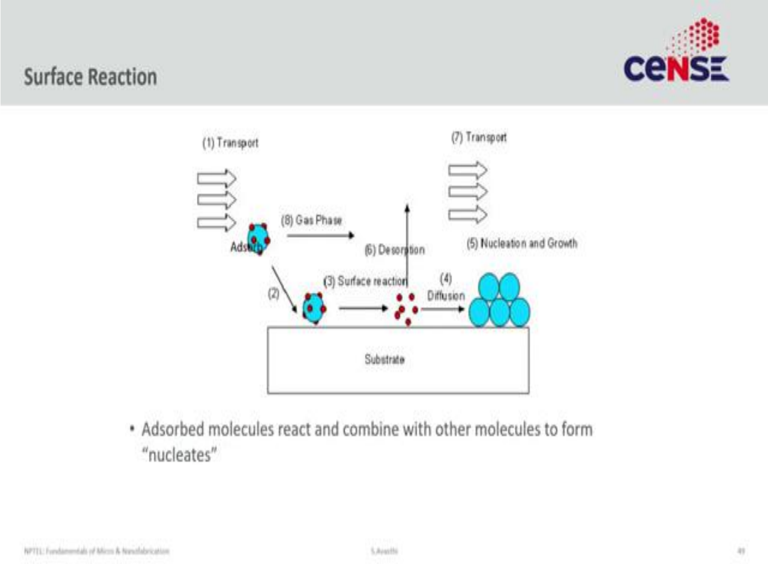

Surface Reaction

- Adsorbed molecules react and combine with other molecules to form "nucleates"

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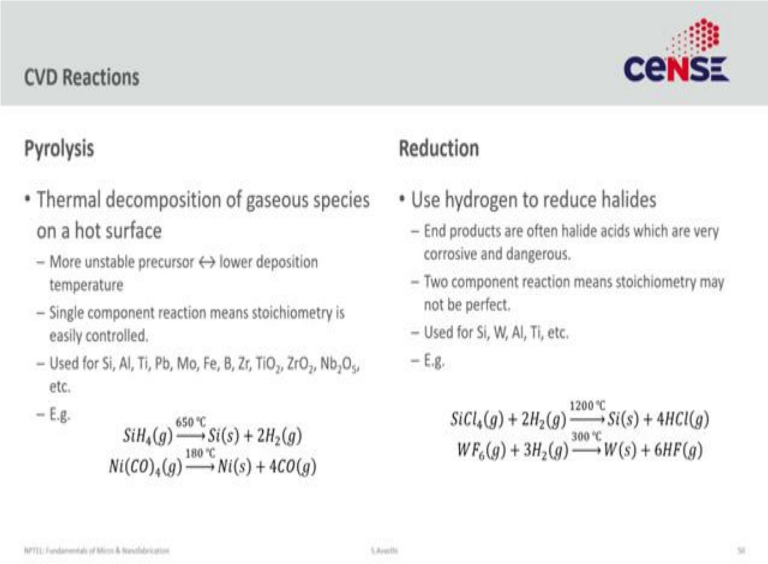

CVD Reactions

Pyrolysis

- Thermal decomposition of gaseous species on a hot surface
  - More unstable precursor ↔ lower deposition temperature
  - Single component reaction means stoichiometry is easily controlled.
  - Used for Si, Al, Ti, Pb, Mo, Fe, B, Zr, TiO_2, ZrO_2, Nb_2O_5, etc.
  - E.g.

$$SiH_4(g) \xrightarrow{650\,^{\circ}C} Si(s) + 2H_2(g)$$
$$Ni(CO)_4(g) \xrightarrow{180\,^{\circ}C} Ni(s) + 4CO(g)$$

Reduction

- Use hydrogen to reduce halides
  - End products are often halide acids which are very corrosive and dangerous.
  - Two component reaction means stoichiometry may not be perfect.
  - Used for Si, W, Al, Ti, etc.
  - E.g.

$$SiCl_4(g) + 2H_2(g) \xrightarrow{1200\,^{\circ}C} Si(s) + 4HCl(g)$$
$$WF_6(g) + 3H_2(g) \xrightarrow{300\,^{\circ}C} W(s) + 6HF(g)$$

Whatever has absorbed on the surface must react. There are different types of possible reactions. Common CVD reactions tend to be pyrolysis or reduction mostly because most semiconductors are not oxides, but are reduced elements. If you want to deposit silicon, you often start with say silane, heat it at high temperatures such that it would decompose into silicon and hydrogen. This silicon is solid, but at this point, it is molecular solid. Those molecules must rearrange on the surface for crystal growth.

In general, more unstable the precursor, lower the deposition temperature, or the temperature at which it would paralyze or break down. For example, nickel carbonyl breaks down at only 180^o C. For compound semiconductors stoichiometry can become a little challenging. Both silicon and nickel are elemental films. So, there is no question of stoichiometry, but if you are trying to deposit a nitride or an oxide, then you might need two components. That is when maintaining stoichiometry can become challenging.

If you can find a precursor that can pyrolyze (thermally decompose) directly into the material without you need to mix two components, it is a huge advantage. A single component reaction means you do not have to worry about the stoichiometry. Pyrolysis is often used for elements such as silicon, aluminum, titanium, lead, molybdenum etcetera, and in some cases for oxides, for example, you can get pyrolysis based precursors for titanium dioxide, zirconium oxide, niobium oxide. Those of you who are familiar may read up about alkoxides. In general, a class of alkoxides can pyrolyze into oxides.

The second very popular class of reaction is reduction. As the name suggests, you use hydrogen to reduce certain materials. As an example, you start with tetrachlorosilane ($SiCl_4$) and which is a gas and you react it with hydrogen at higher temperatures to form silicon and HCl gas. HCl gas floats away, silicon precipitates out and you form your film. You can do something similar to deposit tungsten. Start with tungsten fluoride, react it with hydrogen to form HF gas and tungsten. The disadvantage of or caution about this technique is it often has halide acids (HCl or HF) as a byproduct. Halide acids are very aggressive, corrosive, and hazardous gases. You have to be a little careful in designing a reactor that does this.

Often this is a two-component reaction where you flow $SiCl_4$ and H_2 together. To get stoichiometric or pure silicon, you need this $SiCl_4$ and H_2 to be in some ratio. In this case, it is not too complicated, but in cases of a nitride or an oxide, this can be an issue. Reduction is often used to deposit silicon, tungsten, aluminum etcetera

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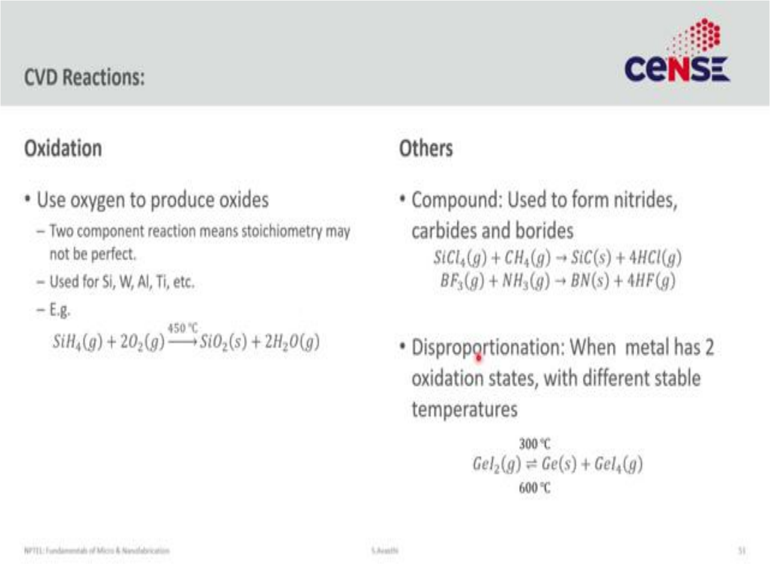

CVD Reactions:

Oxidation

- Use oxygen to produce oxides
  - Two component reaction means stoichiometry may not be perfect.
  - Used for Si, W, Al, Ti, etc.
  - E.g.

$$SiH_4(g) + 2O_2(g) \xrightarrow{450\,^{\circ}C} SiO_2(s) + 2H_2O(g)$$

Others

- Compound: Used to form nitrides, carbides and borides

$$SiCl_4(g) + CH_4(g) \rightarrow SiC(s) + 4HCl(g)$$
$$BF_3(g) + NH_3(g) \rightarrow BN(s) + 4HF(g)$$

- Disproportionation: When metal has 2 oxidation states, with different stable temperatures

$$GeI_2(g) \underset{600\,^{\circ}C}{\overset{300\,^{\circ}C}{\rightleftharpoons}} Ge(s) + GeI_4(g)$$

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Oxidation is self-explanatory. As most of the oxides are not semiconductors or at least not popular as semiconductors yet, oxidation as a technique in CVD is often less used, To deposit SiO_2 for example, you can take silane (SiH_4), put in oxygen and form silicon dioxide. Again, this is a two-component reaction. For stoichiometry, you may need to do some fine-tuning in how much silane and oxygen to flow. Oxides of silicon, tungsten, aluminum, and titanium can be deposited like this.

The general class of others includes replacement, disproportion, etc.. You can react BF_3 with NH_3 to form BN or $SiCl_4$ with CH_4 to form SiC. Disproportionation is used where the molecule both oxidizes and reduces at the same time. For example, germanium iodide can disproportionate into germanium solid which is the semiconductor you want to deposit and germanium iodide which is a gas that flows away.

What reaction you use is decided by the precursor you use and designing a precursor is a research problem in its own right. The goal in most cases is to design a precursor that is safe, volatile, decomposes, or forms the material cleanly, does not have side reactions which lead to impurities and the by-products of which are safe.

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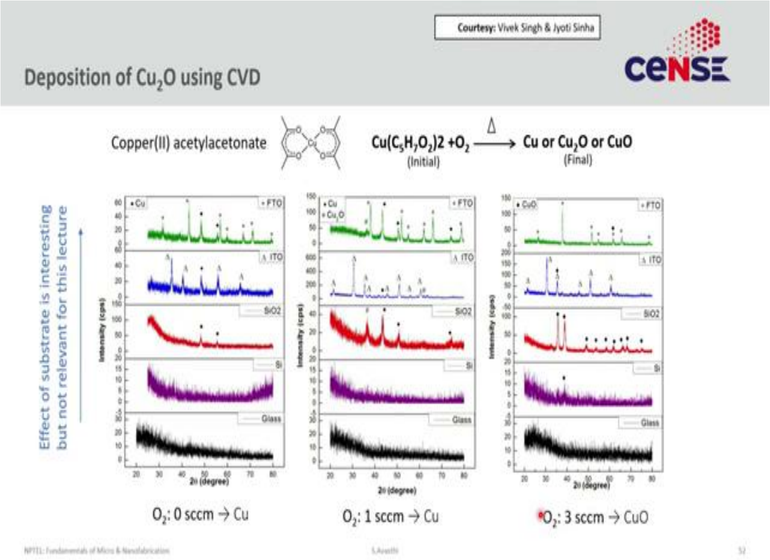

Courtesy: Vivek Singh & Jyoti Sinha

Deposition of Cu_2O using CVD

Copper(II) acetylacetonate

$Cu(C_5H_7O_2)2 + O_2 \xrightarrow{\Delta} Cu$ or Cu_2O or CuO

(Initial) (Final)

Effect of substrate is interesting but not relevant for this lecture

O_2: 0 sccm → Cu

O_2: 1 sccm → Cu

O_2: 3 sccm → CuO

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So, here is an example from my labs. We are using copper acetylacetonate which with oxygen and heat decomposes into copper. This is a two-component reaction in which you can form either Cu, Cu_2O (Cu^{+1}), CuO (Cu^{+2}), or something in between depending upon a lot of details, for example, how much oxygen you are flowing. If you do not flow any oxygen, you get Cu. In this example, on silicon dioxide substrate, we see these X-Ray Diffraction (XRD) peaks which correspond to Cu. At increased oxygen concentration, you start forming Cu_2O. For a certain concentration of oxygen, we start seeing a mixture of Cu_2O and Cu. This is evident from the second XRD pattern (with 1 sccm O_2) where you see peaks corresponding to Cu and Cu_2O. If you increase the oxygen even further, then you start forming a fully oxidized form of copper: CuO. This (with 3 sccm O_2) is what the XRD looks like where all the peaks correspond to CuO.

The reaction and the type of film also depend on the substrate. We do not just have a silicon dioxide substrate; we have ITO, FTO, silicon, and glass substrates. There is also a huge dependence on the substrate. The reasons for this, however, will be clear in the following lectures. But, it does highlight the fact that CVD is a very versatile technique. You can get very different types of materials and there are a lot of levers you can tune. For a comprehensive understanding of CVD, you would require a lot of experience and essentially a course on its own. This course will not try to do that; we will highlight the basic concepts. So, at least you are empowered to read papers on CVD and learn further.

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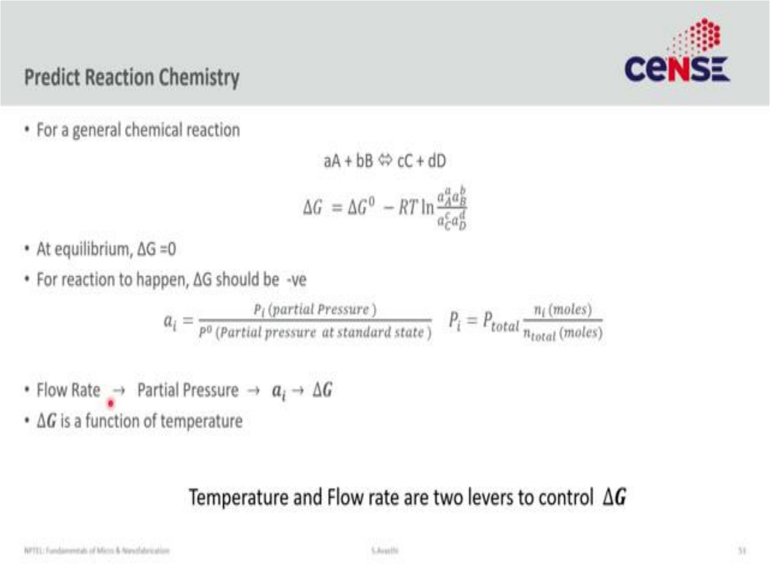

Predict Reaction Chemistry

- For a general chemical reaction

$$aA + bB \Leftrightarrow cC + dD$$

$$\Delta G = \Delta G^0 - RT \ln \frac{a_A^a a_B^b}{a_C^c a_D^d}$$

- At equilibrium, ΔG =0
- For reaction to happen, ΔG should be -ve

$$a_i = \frac{P_i\,(partial\ Pressure)}{P^0\,(Partial\ pressure\ at\ standard\ state)} \quad P_i = P_{total} \frac{n_i\,(moles)}{n_{total}\,(moles)}$$

- Flow Rate → Partial Pressure → $a_i \rightarrow \Delta G$
- ΔG is a function of temperature

Temperature and Flow rate are two levers to control ΔG

Let’s talk about how to predict this. In the example that we saw, a precursor reacts with the oxygen and it can either form metal or oxides of different oxidation states. Is there a better way to predict what would occur than just experimenting empirically? It turns out, yes. Any general chemical reaction, say, a molecules of A reacting with b molecules of B to form c molecules of C and d molecules of D. This reaction would have a Gibbs free energy change given by this relation. At equilibrium, the Gibbs free energy change is 0 and for spontaneous reactions, it is negative. ΔG_o (standard molar free energy change for the reaction) is often known - can be looked up from the tables or software.

Once you know the ΔG_o and the temperature of the reaction, the only thing you need to know is the activity of the various components involved in the reaction. The activity in case of gas-phase reaction is nothing, but the partial pressure divided by the total pressure. This is very easy to calculate as we have discussed in the previous lectures on CVD, by Dalton's law of partial pressures you can calculate those partial pressures if you know the flow rates. If you can figure out this a_i, then all you have to do is to plug these a_i’s back into this equation and calculate the ΔG and for negative ΔG, that reaction must occur spontaneously. Given a chemical reaction that must happen you can calculate ΔG, given the partial pressure of the components. The ΔG is a function of temperature.

To make a certain material, you need to figure out under what conditions of temperature and partial pressure your ΔG is negative and for those range of temperature and pressures, you will get the reaction to occur. This is how you can figure out your window of deposition.

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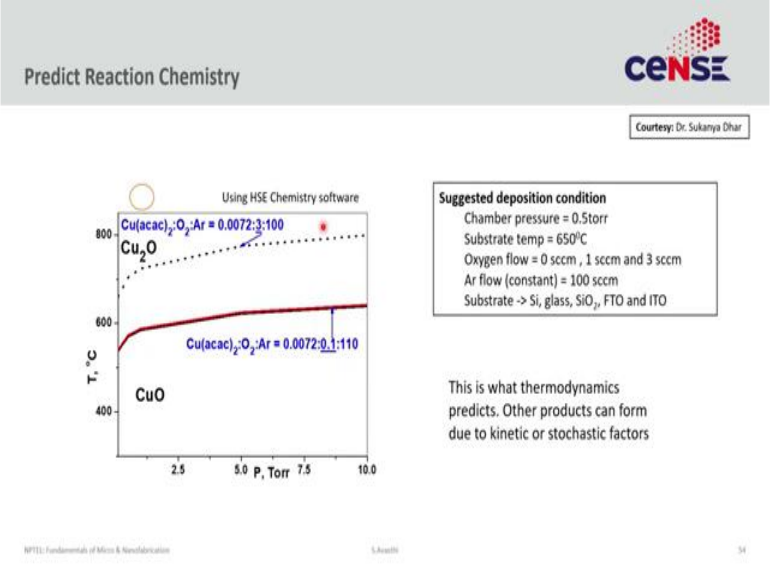

Predict Reaction Chemistry

Courtesy: Dr. Sukanya Dhar

Using HSE Chemistry software

Suggested deposition condition

Chamber pressure = 0.5torr
Substrate temp = 650°C
Oxygen flow = 0 sccm , 1 sccm and 3 sccm
Ar flow (constant) = 100 sccm
Substrate -> Si, glass, SiO_2, FTO and ITO

This is what thermodynamics predicts. Other products can form due to kinetic or stochastic factors

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We use a software called HSE chemistry to calculate the conditions of temperature and pressure for the formation of Cu_2O. At low temperatures, we form CuO, at high temperatures Cu_2O and a mixture in the middle. This becomes a very handy guide for experimentation. Sometimes experimentations can be little off towards left or right but this does provide a very good idea of which direction you should tweak your parameters to get the material you need.

Everything we have talked till now is about thermodynamics. This is what would occur given an infinite amount of time, but when we are doing CVD in a realistic reactor we do not have infinite time which is another way of saying we should also be careful about the kinetics. Kinetics and thermodynamics can sometimes give you different results. We will come to some kinetic factors later in the lectures.

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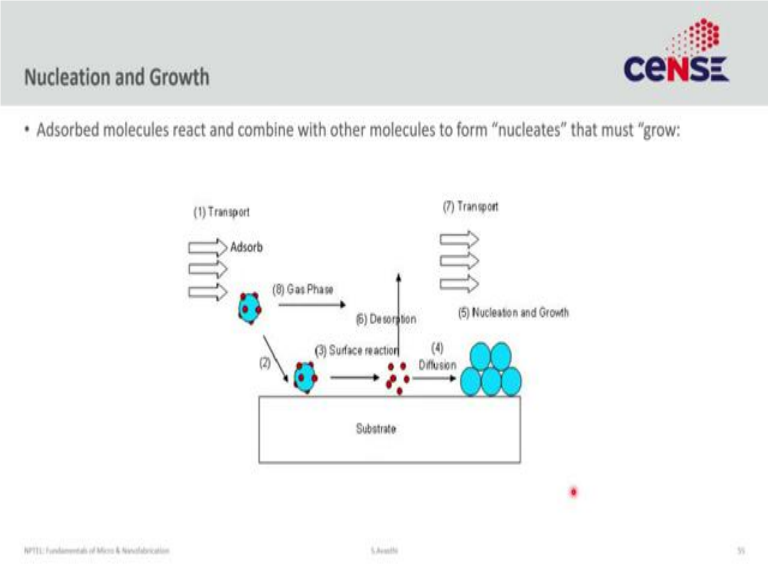

Nucleation and Growth

- Adsorbed molecules react and combine with other molecules to form “nucleates” that must “grow:

Let’s talk about nucleation and growth. To summarize, we start with the gas phase precursors that are absorbed on the surface, undergo a surface reaction, and leave the desired material on the substrate in atomic or molecular form. This material must reorganize on the surface. How it re-organizes on the surface is the discussion of nucleation and growth.

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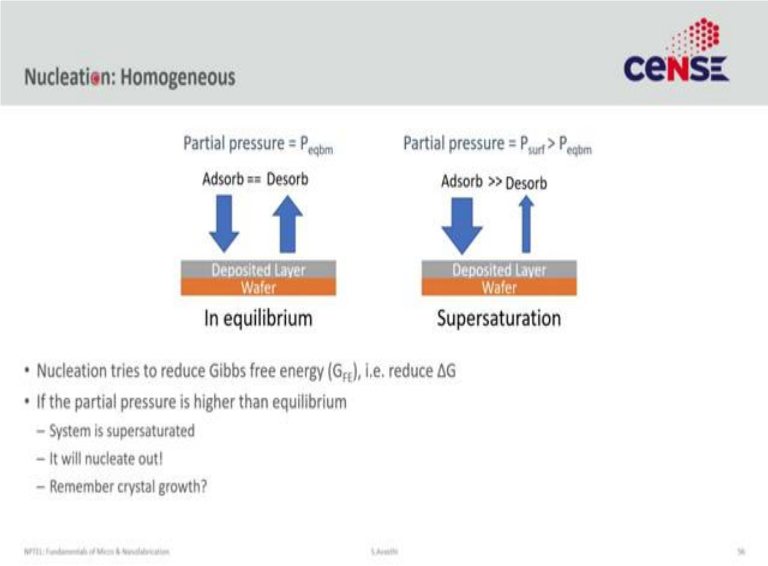

Nucleation: Homogeneous

- Nucleation tries to reduce Gibbs free energy (G_{FE}), i.e. reduce ΔG
- If the partial pressure is higher than equilibrium
  - System is supersaturated
  - It will nucleate out!
  - Remember crystal growth?

We have visited nucleation before while discussing crystal growth. These concepts we relevant even for crystal growth. We will use the same terminology as supersaturation.

The basic idea was that at any given temperature and pressure any material will have a certain phase equilibrium for example, a solid or liquid will have a certain vapor pressure in a closed system. This is a kinetic equilibrium. In our system of vapor-solid equilibrium, there would be adsorption of vapor on the surface, and desorption of the vapor from the surface too. In kinetic equilibrium, these two rates would be equal, so, the net thickness of the film would not change.

To make this system go into a non-equilibrium state, specifically, in supersaturation, the adsorption rate needs to be higher than the desorption rate. We would then have a driving force for transporting the material onto the surface. In CVD, you form a molecular layer on the substrate by chemical reaction and these atoms get added to the film at a rate greater than the desorption rate. (Note: In CVD, the composition of the films is almost always different from the precursor. We will use P_{surf} and P_{eq} to denote the surface and equilibrium pressure of the species (molecular layer) produced after the chemical reaction and not of the precursors. The elemental composition of this species is similar to that of the film). So, $\Delta G < 0$ if the system is under supersaturation (precursor vapor pressure such that $P_{surf} > P_{eq}$), there will be a driving force for the atoms to go the solid phase. Thus, by maintaining the precursor vapor pressure such that $P_{surf} > P_{eq}$, you get a deposition of the film on the substrate. Remember that thermodynamics predicts the state of the system given infinite time. However, in the practical systems, there is certain kinetics to it.

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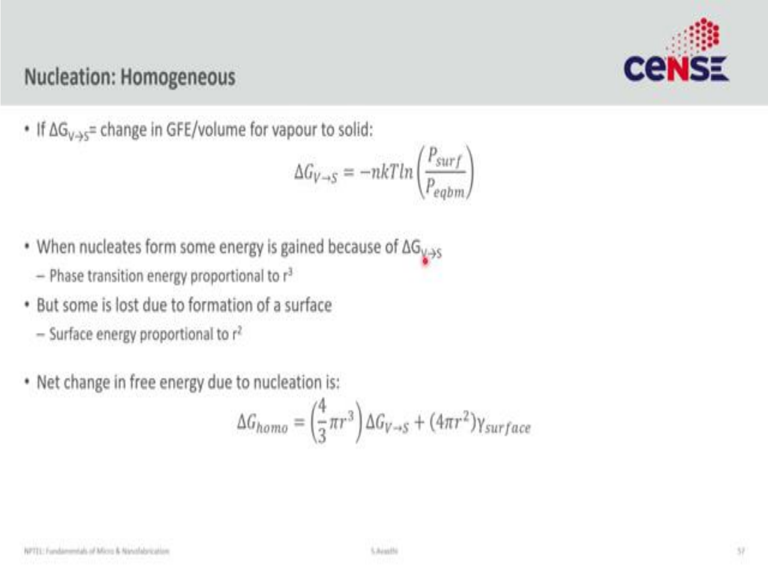

Nucleation: Homogeneous

- If $\Delta G_{V \to S}$= change in GFE/volume for vapour to solid:

$$\Delta G_{V \to S} = -nkTln\left(\frac{P_{surf}}{P_{eqbm}}\right)$$

- When nucleates form some energy is gained because of $\Delta G_{V \to S}$
  - Phase transition energy proportional to r^3
- But some is lost due to formation of a surface
  - Surface energy proportional to r^2
- Net change in free energy due to nucleation is:

$$\Delta G_{homo} = \left(\frac{4}{3}\pi r^3\right)\Delta G_{V \to S} + (4\pi r^2)\gamma_{surface}$$

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Homogeneous nucleation is the nucleation that does not happen on the surface but in the gas phase. Although this is not what we want in CVD, it is a good starting point in understanding heterogeneous nucleation that we typically see in CVD.

Imagine that you have pushed a certain precursor in a container such that the pressure of the precursor inside is higher than its equilibrium pressure. So, the system is under supersaturation. So, now, there is a driving force for these vapors to condense in solid (or liquid) phase. The Gibbs free energy (GFE) change associated with this transition is given by $\Delta G_{V\to S} = -nkT\ln(\frac{P_{surf}}{P_{eq}})$. So, when $P_{surf} = P_{eq}$, ΔG is 0, and the system is under equilibrium. As the partial pressure of the species on the surface increases, the GFE change becomes more and more negative.

If this chemical reaction happens in the gas phase, the reaction products would form a sphere or similar equilibrium shaped solid (can also be liquid, depending on the precursor, pressure, and temperature) and reduce the Gibbs free energy of the system. Since the volume of a sphere $V \propto r^3$, where the radius of this sphere/nucleus is r, the reduction in free energy of this nucleus is also proportional to r^3 ($\Delta G \propto -r^3$). However, this nucleus has a free surface and the creation of this surface increases the free energy of the nucleus. As the surface area of a sphere $A \propto r^2$, the surface energy penalty scales with r^2. ΔG_{homo} has a surface component that scales with the surface area of the nucleus and a volume component that scales with the volume of the nucleus.

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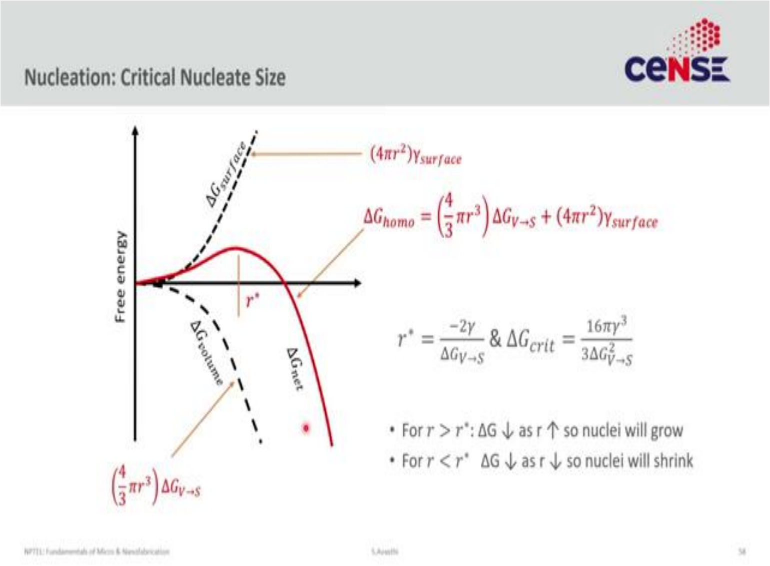

Nucleation: Critical Nucleate Size

$\Delta G_{homo} = \left(\frac{4}{3}\pi r^3\right)\Delta G_{V\to S} + (4\pi r^2)\gamma_{surface}$

$r^* = \frac{-2\gamma}{\Delta G_{V\to S}}$ & $\Delta G_{crit} = \frac{16\pi\gamma^3}{3\Delta G_{V\to S}^2}$

- For $r > r^*$: ΔG ↓ as r ↑ so nuclei will grow
- For $r < r^*$ ΔG ↓ as r ↓ so nuclei will shrink

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Here, we plot the volume free energy change component ($\frac{4}{3}\pi r^3 \Delta G_{V \to S}$) and the surface free energy penalty ($4\pi r^2 \gamma$) and the net free energy of homogeneous nucleation (ΔG_{homo}) as a function of the radius (r). $\Delta G_{surface}$ goes up slower than ΔG_V. So, for very very large r's ΔG_{homo} would be negative. However, for small r's, the penalty of making a surface is higher than the free energy reduction from the volume $\Delta G_{V \to S}$. So, for small r's there a penalty of formation ($\Delta G_{homo} > 0$). What this means is that under supersaturation, nuclei do not want to form when they are small, but nuclei do want to form when they are large. But, from a gas-phase chemical reaction, you can't directly make a very large nucleus.

Then how does that work? There is a Gibbs free energy barrier to nucleation because of the surface energy component. However, this is a *thermodynamic* energy barrier. Any small nucleus with $\Delta G_{homo} > 0$ is unstable and will *eventually* disintegrate by desorption. Kinetically, the small nuclei keep forming and disintegrating in the system biased towards vapor to solid transition. Statistically (by chance), some of the nuclei grow a little larger by coalescing with smaller nuclei and become *thermodynamically* stable. Smaller nuclei keep shrinking the larger nuclei keep growing. There is this critical radius r^* which is the boundary of this difference. If the nucleus has $r > r^*$, it will grow and if it has $r < r^*$, it will shrink because above r^*, ΔG_{homo} reduces with increasing r and below r^*, ΔG_{homo} reduces with reducing r.

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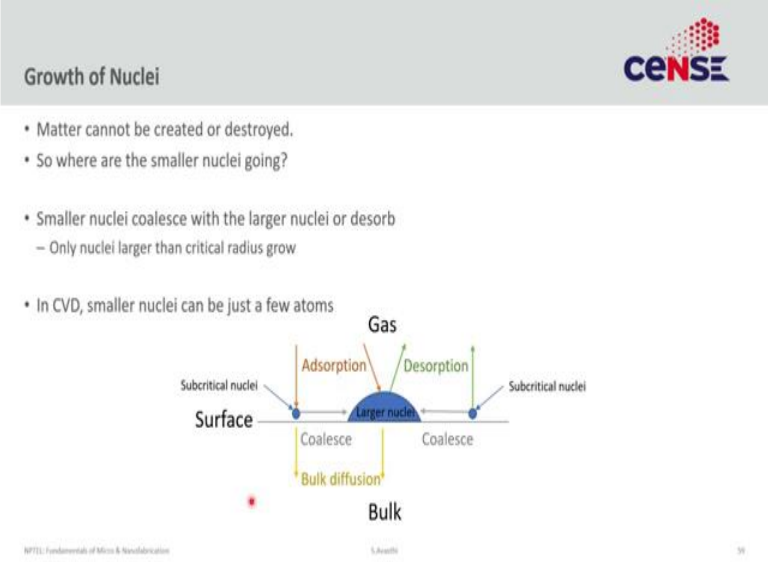

Growth of Nuclei

- Matter cannot be created or destroyed.
- So where are the smaller nuclei going?

- Smaller nuclei coalesce with the larger nuclei or desorb
  - Only nuclei larger than critical radius grow

- In CVD, smaller nuclei can be just a few atoms

Now, the matter cannot be created or destroyed. So, when the smaller nuclei shrink, where are the atoms going? As we have discussed, they might disappear by desorption. The second thing that can happen to the smaller nuclei is they can coalesce into larger nuclei. This is what I am trying to show here. Larger nuclei would suck up more and more of the nuclei that are on the surface and keep becoming larger and larger. In CVD, when we say small nucleus, that small nucleus maybe just a few atoms. The other thing that can happen to these nuclei is bulk diffusion of the atoms. So, if you, for example, are depositing phosphorus on silicon, the phosphorous atoms can also diffuse into the silicon wafer. You want the atoms to coalesce into forming larger nuclei because that is what is giving the growth; rest everything the desorption, the bulk diffusion subtracts from the growth. So, you have to find that temperature and pressure window where this process is the dominant one.

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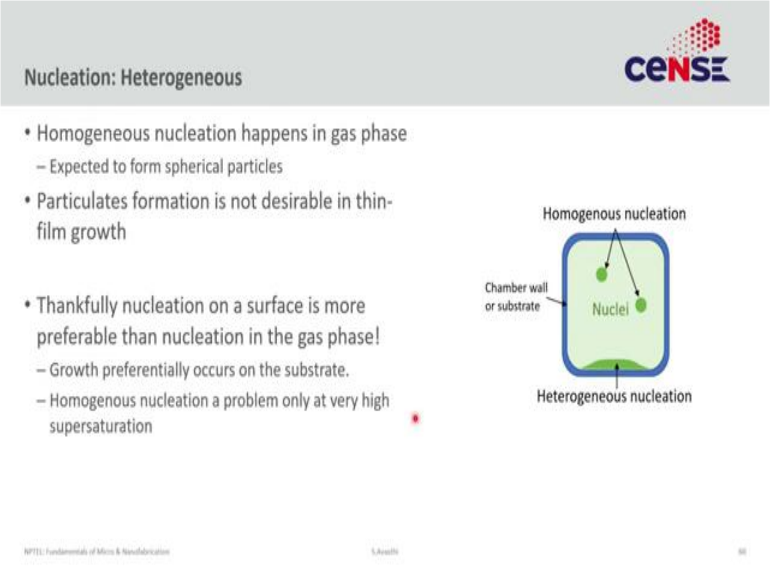

Nucleation: Heterogeneous

- Homogeneous nucleation happens in gas phase
  - Expected to form spherical particles
- Particulates formation is not desirable in thin-film growth
- Thankfully nucleation on a surface is more preferable than nucleation in the gas phase!
  - Growth preferentially occurs on the substrate.
  - Homogenous nucleation a problem only at very high supersaturation

In CVD, we want the chemical reaction and nucleation to occur on the substrate and not in the chamber. Nucleation that happens on the surface/heterogeneities instead of in the bulk of the generating phase is called heterogeneous nucleation. In this slide, homogenous nucleation is when this green vapor coalesces into a nucleus that is suspended in the air. This is what happens when mist, for example, forms from the vapors in the atmosphere. In CVD, the primary mechanism is that you have a susceptor or a wafer surface and on that, you start forming the nuclei and that nuclei may not be spherical and shape. In this figure, the large nucleus is not drawn as a sphere. If it is not a sphere, does the mathematical model that we have developed for homogeneous nucleation hold here?

It so turns out that heterogeneous nucleation is more probable than homogeneous nucleation. This is important because if you are depositing silicon using silane and if the gas-phase reactions start happening more than substrate surface reactions, then instead of getting a silicon film you will just have a shower of silicon particles as silane will break down into silicon atoms, and those silicon atoms will form these particles and those particles will rain down on your wafer, but not give you a film. It does not happen because homogeneous nucleation is less probable than heterogeneous nucleation, so you get deposition on the wafer before nucleation in the vapor.

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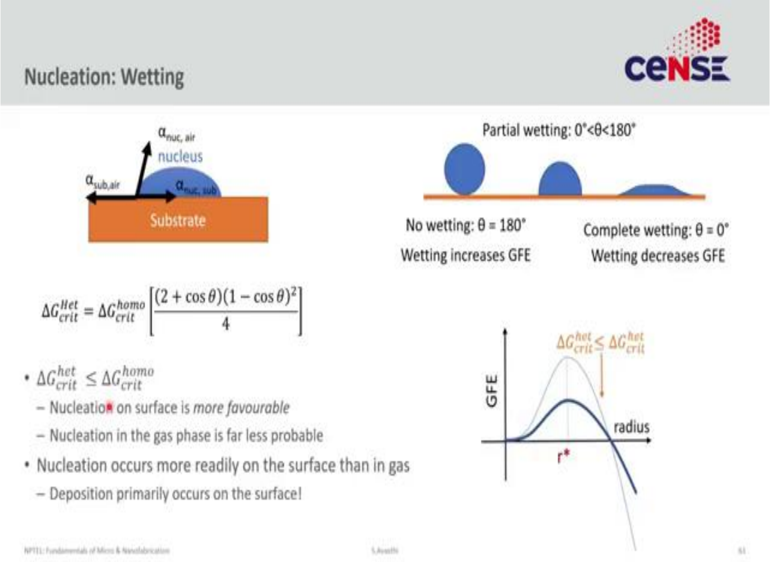

Nucleation: Wetting

$$\Delta G_{crit}^{Het} = \Delta G_{crit}^{homo}\left[\frac{(2+\cos\theta)(1-\cos\theta)^2}{4}\right]$$

- $\Delta G_{crit}^{het} \leq \Delta G_{crit}^{homo}$
  - Nucleation on surface is *more favourable*
  - Nucleation in the gas phase is far less probable
- Nucleation occurs more readily on the surface than in gas
  - Deposition primarily occurs on the surface!

To understand heterogeneous nucleation, you need to understand wetting. If you put a drop of water or any liquid on top of a substrate, then there are three types of surface tensions that are acting; between the substrate and the nucleus, between substrate and air and between the nucleus and air. And, under equilibrium all these balances and for their balance, the nucleus-air interface makes a certain angle with the substrate at the point of contact. This angle is called the contact angle. The balance of these forces decides the contact angle and hence, the shape of the nucleus. If a material does not wet surface at all or is hydrophobic, and you put water on top, then water just sits and there is no wetting and $\theta = 180^o$. The other extreme is if the water wets the surface perfectly then the contact angle is essentially 0.

Look at this formula, which we shall not derive and I shall not ask you in exams. Notice that $\Delta G_{het}^{crit} < \Delta G_{homo}^{crit}$ for any contact angle θ (wetting), as the angle-dependent part of the equation is less than 1. $\Delta G_{het}^{crit} = \Delta G_{homo}^{crit}$ for the case of $\theta = 180^o$ because for $\theta = 180^o$, you form a complete sphere and that is exactly what happens in the homogenous nucleation. For this case of no wetting, the substrate surface does not influence the nucleation. As you go from $\theta = 180^o$ to smaller angles, the value of this term reduces, which reduces ΔG_{het}^{crit} in comparison to ΔG_{homo}^{crit}. The nucleation on the surface is always more favorable and that is the reason we can deposit films and do not have a shower of particles.

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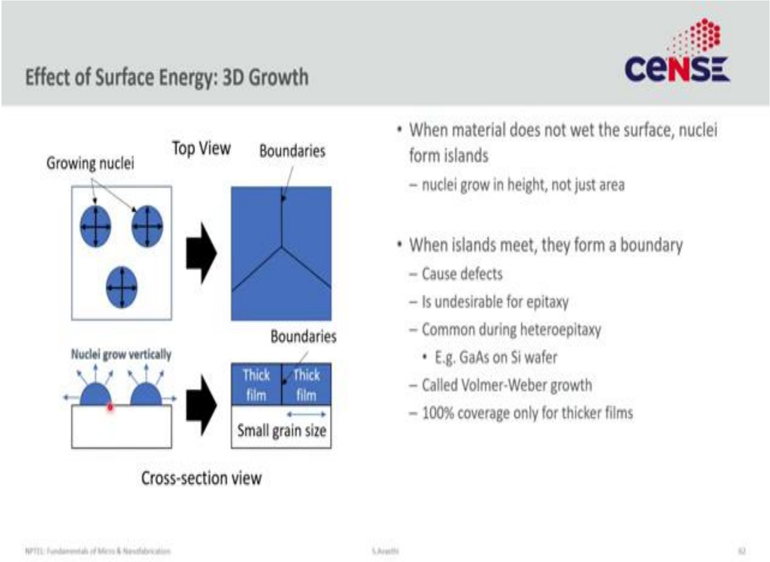
Effect of Surface Energy: 3D Growth

- When material does not wet the surface, nuclei form islands
  - nuclei grow in height, not just area
- When islands meet, they form a boundary
  - Cause defects
  - Is undesirable for epitaxy
  - Common during heteroepitaxy
    - E.g. GaAs on Si wafer
  - Called Volmer-Weber growth
  - 100% coverage only for thicker films

If the material doesn’t wet the surface (high surface energy and contact angle), as more and more material comes it tends to stick to itself and not to the substrate. So, you would form these small spheres on the surface instead of an (atomically) flat film. These nuclei would grow vertically as well as horizontally. As they become larger and larger, at some point, they coalesce and form a boundary. Often this is a grain boundary or a domain boundary. This sort of process where the nuclei grow as (spherical) islands instead of layers along the surface is called Volmer–Weber growth or 3D growth. It is undesirable for the epitaxy because you do not want these boundaries. These boundaries are bad for devices. These nuclei that are growing are not connected, have no way of communicating the crystallographic orientation with each other, grow in different orientations and when they meet, you have a defect in the crystal structure at the boundary.

The other disadvantage is that 100 % coverage is only achieved for the thickest films; for thinner films, you form these islands that are disconnected from each other. This is, however, a common case during heteroepitaxy. For example, if you are trying to deposit gallium arsenide on silicon, this may occur. However, when you grow silicon on silicon, the problem of wetting is not that severe and you form good films.

In the next lecture, we shall look at 2D growth, which is what you want in heteroepitaxy. We will look at nucleation, the rate of nucleation, the density of nuclei which decides your grain sizes, and certain other aspects epitaxy.

Thank you.