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Module - 2 Lecture - 9 Template Methods – I

Welcome back to this course on nano structured materials synthesis properties self assembly and applications. We have had two lectures in module one and today is the seventh lecture of module two. In the previous lectures, we discussed the synthesis using CVD techniques that is the chemical vapor deposition techniques. The physical vapor deposition techniques and MBE techniques, which is molecular beam butoxy techniques. Today, we will start on the synthesis of nanostructured materials based on some template methods.

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The template based synthesis as the name suggests is based on some form of a material which will form the template and on which the nano structured material will made. Once it is made, the template will be removed and only the nanostructured material will remain.

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Now, this is schematically shown in a scheme which all of you must have seen, you must have seen pottery or Chinese pottery, specifically where the finished material is of clay or something, but how is this shape taking place start with the template. This template in this case is made of wood or it can be made of a metal and then in this case, we put the clay on to this piece of mood a wood and the clay fills all the holes and all the gaps within the a piece of wood.

Then, once you bake it and then you remove the piece of wood, what is left is this jar which is made only of the clay. So, this is precisely what you are doing to do in a template method, you are going to use a template and put some material which is clay. In this case, in our case, you may use any nano particle, any other material which can be a liquid which will form a shape. The shape is being given by the template, this piece of wood here will be given by some other template, which will be in the form of small structures.

Ultimately, when we remove the template, you will get in this case you get a large structure, you will get small structures which are independent on the shape of the template.

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So, what can be these templates in the molecular region in the nanostructure region, you can use many templates. For example, you can use cation exchange resins, which have micropores, that means the size of the pores or micropores, you can use zeolites. All of you know zeolites are alumina silicates, they have three dimensional structures, they may have channels within them or different zeolites have different size of pores. So, depending on which zeolite you use, you can either make a small particle or you can make a columnar structure.

You can also use silicate glasses as templates within which you can make a material which will have in itself some features which are left from the silicate glasses you can use ion exchange techniques. So, you have template in which there is a particular ion in the material and then you ion exchange with another ion and now you get a new material with the same structure as the initial template material. So, only one ion, one set ions have been exchanged in this methodology, you can also use gas deposition on a shadow mask.

The shadow mask here will act as a template and then this ordering of the gas molecules on this mass will give you the structure that you basically need. For that, you have to design this shadow mask because according to that, the gas deposition will take place and the structure that you need will be formed. So, these are a different classes of templates we discussed cation exchange resigns, where cations can be exchanged zeolites silicates and then other ion exchange possibilities.

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So, there are different types of pores, density of pores possible with different types of template or different type of membrane, which are going to act as templates. For example, if you have a polymer, a mainly poly carbonates are used for this purpose where you have ion tracks. That means use ions bombard this polymers and these ions leave tracks and these tracks of the order of 5 to 500 nanometers. These channels are ion tracks as we call and the density of these pores are of the order of 10 to the power 11 per meter square.

These are randomly arranged pores, you can also have mica which in natural naturally occurring in organic material. It is a mineral and this mica has layered structure and in this mica.

You can make this kind of etching to yield channels of 1 to 500 nanometer size in which again you can have random pores of the order of 10 to the power 10 or 10 to the power 11 pores per meter square. You can also use alumina, which is aluminum oxide Al 2 O 3 and in this alumina, you can make channels of the type of 10 to 500 nanometers and which is the pore diameter and again you have 10 to the power 11 to 10 to the power 13 pores.

Now, in this case you can have ordered pores, so earlier we were taking of random pores, but in the case of alumina and as well as in the case of block copolymers, we can get ordered pores and higher density of ordered pores. The channel width will vary from 10 to 500 nanometers in the case of alumina and 10 to 20 nanometers in the case, where you are using block copolymers basically block copolymers have two types of polymers. One may be hydrophobic, the other can be hydrophilic and they form two types of polymers joint together form around block copolymers.

There also you can create this kind of ion channel 10 to the power 10 to 20 nanometers thick, which will yield you ordered pores in which you can do synthesis leading to nano structured materials. These are precisely ordered because the pores in which you are doing the synthesis are precisely ordered, so you can have random pores or you can have ordered pores. Using these different types of membranes, we can yield random nanostructures or ordered nanostructures.

So, these are various types of membranes and templates which are used to grow nano wires within these pores. So, you can see you can generate from around 1 nanometer diameter to up to 500 nanometer diameter wires and many wires, you can generate of the ordered of 10 the power 10 to the power 13, depending on which methodology you use to grow these nano wires in this templates.

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So, this is an example of a template mediated growth, where you are trying to grow carbon nano tubes on silicon nano wires tips. So, here the silicon nano wire is the template on which you are trying to grow carbon nano tube. So, the carbon nano tubes are these thin tubes which are growing out of these silicon nano wires and so the carbon nano tube is being guided by the silicon nanowires, which has been formed earlier. This is a close of view, where you can see this is the silicon which has wider or larger diameter and on top of that you have this carbon nano tube, the scale here is around 10 nanometer.

So, half of this is around 5 nanometer, so you can think of this is like 5 to 6, 5 to 7 nanometer thick carbon nanotube grown on around 15 nanometer thick or 20 nanometer thick silicon nanowires. So, these are very useful techniques the template based techniques are very useful to prepare a nanowires, which are precisely ordered in a particular fashion.

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To look at these carbon nano tubes more carefully, here is a high resolution transmission electron microscope micrograph. So, you have to use electron microscope, which has working under voltage of around 200 to 300 kilo volts. You know accelerated electrons come and hit the same which is loaded on a grid, which is which may be copper grid or something and the electrons. Then, make an image of the particles that they see and depending on the voltage the wavelength of the electron can be modulated and the wavelength of the electron can be of the order of 0.05 or 0.02 angstrom.

So, you can see objects which are very small and so here is a carbon nanotube and you can see the different layers of carbon and this is a typical thickness between the walls of two carbon layers of around 3.4 angstrom or 0.34 nanometers. In this slide, what you see is that you have this silicon nano wire and on the tip of the silicon nano wire, you have this carbon which is growing on top of the silicon nanowire. So, the silicon nanowire is acting as the template and top of that you have this carbon rather than a nanotube here is like more spherical.

So, these spheres of carbon since there are several layers in each sphere, this is a called onion like structure and so these are called carbon nano onions it is nano because you know they are of the order of few nanometers in diameter.

So, this is 0.45 nanometer, so around this whole thing may be around 10 nanometers or 8 nanometers, so here we are seeing silicon nanowire, which earlier we showed this is silicon nanowire where carbon nanotube is growing. Here, we are showing silicon nanowire as a template at the tip of that carbon onions are growing.

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These have tunnels like this or channels or you can have lipid bilayer vesicales or many other inorganic compounds which have tunnels structures. So, you have vacant spaces

between the cylinders in which you can grow nanowires, you can also have hosts which are two dimensional in nature and these are layered hosts. These can be made of materials, which are layered materials like molybdenum dioxide or Li Nb O 2 or lithium cobalt oxide, there can these can be oxides these can be halides chalconenide layers.

For example, molybdenum di sulphide tungsten disulphide molybdenum diselenide, these are all two dimensional layered materials. In between the layers, you can grow material which you want to grow and this layered the blue colored layers will then act as a template. After the reaction, you have to remove these templates to regain the material which are in between the layers. So, here you get one dimension linear structures when you make materials within the cylinders, here you get two dimensional structures when you make the synthesis suing these 2 D layered hosts.

This is the three layered host, so they form pores structures, so there are pores inside, so these are 3 D frame work host and may materials have zeolites can have this kind of cylindrical pores. They can also have these kinds of 3 D pores, which are of the order of 4 to 10 to 20 angstroms, few nanometers and you can have many other polymers bucky ball etcetera as 3 D frame work hosts.

Here, you can generate only particles and not layers and cannot generate wires, normally you will generate particles when you use 3 D hosts as templates for your synthesis. Here, you will you can generate wires and in this case you can generate 2 D layers using these particular hosts.

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This is another method, where you are using you are going to make metal nanostructured using a template based synthesis. So, you have say gold, one metal here and Aluminum another metal here and then you many structures to separate the layers, which will form in between. This you fill with the copper solution, there will be copper solution here which and in between you have membranes. So, what happens a depending on the redox potentials, the metal that you have chosen aluminum and copper will get reduced easily. So, it will accepts electrons to form this copper solution, which may be copper nitrate solution or copper sulfide solution.

You will have copper ions in this solution and it will readily take up electrons which are given by any metal. So, the metal which will give up electrons easily or this aluminum metal and this aluminum metal when it gives out electrons its form aluminum ions and goes into solution. The electrons that it gives out that is actually coming here reacting with the solution and copper two plus gets reduced to metallic copper and wherever it is get reduced, it is get deposited.

So, this wherever your copper, two ions are there they get reduced and they form copper metal layers in these cavities. Now, you have a particular order of copper metal which are separated from each other by this gap because no copper metal can deposit here, which are the membranes and only the gap which available where the solution can go can be reduced to copper metal. So, you will have copper metal regularly spaced between this regions and then again you will have copper here and again you will have copper here like that.

So, you can arrange systematically suing this template and here we call this the anatase aluminum, so aluminum is the anode and it is acting as an anode. So, this aluminum metal template is also call the anodized aluminum metal template is also call the anodized aluminum metal template, which is used for doing the templates base synthesis for metal nanostructures. It is possible to make nanostructures of those metals which have a much more is in accepting electrons from aluminum, so they have a higher reducing potential reduction potential compare to aluminum.

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Now, what other metal materials can you make using these templates based methods, you can make inorganic materials you can make organic materials inorganic materials like metals. Now, we showed copper metal, we can make gold silver, we can make organic compounds, and we can make polymers or metal organic compounds. Suppose, you have to make a compound of caladium with some organic, which may be a preordain based or naphthalene based etcetera.

So, you can make materials of different kinds using the template based synthesis, you can these materials that you are depositing using the help of a template. They may have avoid a ranging properties, some can be insulating some cam be semiconductors or metals or even super conductors that is those in which the resistance below a certain temperature goes to 0, such kind of materials are called super conductors.

So, you know you can deposit any kind of material if you choose the right conditions the right template and the right reaction whether it is a reduction or some other reaction. So, you can make a wide variety of materials with different properties and the size of these can vary if you have channels that is one dimensional channels, you have diameters from 5 to around 10,000 angstroms of these materials.

So, you can have wires which are from 5 angstrom to around 10,000 angstroms, so which is like 0.5 nanometers to around 1,000 nanometers, which is 1 micron. So, you can have from 0.5 nanometers to on micronsized line ararrays of nanostructured materials using templates if you want to make 2 D structures within the interlamellar spaces.

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The interlamellar space and your material that you will be synthesizing using this 2 D template or this 2 D host will be synthesized within these two dimensional layers, so which are which is also called the interlamellar space.

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So, in the interlamellar space, you can make layers which are of the order of three angstroms wide or three angstroms long up till 50 angstroms wide and 50 angstroms long. So, very large like micron sized nanostructures are possible in channels, but normally in the interlamellar spaces or 2 D structures is difficult to make very large layers of 2 D structures in the cavity diameter that is the 3 D structures. Here, you have cavities like this here also you can make vary the cavity size depending on that choose of your carbon sieves or the zeolites or the polymers what kind of cage you have.

You can have variety of sizes and hence you can make very large cavity diameters, typically they are on the larger side and it is very difficult to get very small three dstructures. So, depending on the shape of the material you have some restrictions on the type of size you will get and in 1 D structures, you can get very large values. In again 3 D structures you can get very large values however for 2 D structures or 2 D nano structures made within the interlamellar spaces of the template, you normally get sizes which of the order of the 3 to 50 angstroms.

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Now, you can also use surfactants as a template, so far we were discussing zeolites or metal oxides or metal chalconenides three dimensional structures, two dimensional structures which are like inorganic hard inorganic solids. Here, you can make aggregates of surfactants and these aggregates form from a molecules, which normally have a organic long tale with a some kind of ion at the head. So, it this surfactants are made of a head group which can be like an ammonium group or any other head group with say tri alkali group with the nitrogen.

This may be charged or it can be uncharged moiety and most of the time the head group is polar much more polar than the tale group. So, the tale and the head together form molecule which is called a surfactant and these surfactants can organized themselves to forms spherical micelles or they can form cylindrical micelles or they can form two micelles one inside and one outside. Then, there is a gap in between which go thought the sphere a this kind of aggregates of surfactants are called vesicles.

So, you can make use of these as templates and synthesized your material within this cylinder or within this sphere or within this what is called a bi continuous structure. So, depending on the type of surfactant its concentration and the solvent in which it is present, you can have different kind of structures.

You can then synthesize a new material within the structure, so the surfactant now acts as a template within which you make your material, but it will be shaped like the templates. So, if you use the cylindrical micelle and do the synthesis inside the micelle then the shape of the product that you will get will also have a shape like a cylinder, so it will do what is called the templating effect.

Now, you can choose a wide variety of surfactants and so there is lot of choice how to control the diameter of this cylinder how to control the length of the cylinder how to control the diameter of this spherical micelle etcetera. So, surfactants are very rich source of templates, if you can make them aggregate the way you want them to aggregate or the way you want your final structure to be. Ultimately, we have to remove this template and the reaming structure should be the final structure what you want with your material left behind.

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So, this is an example where you have these surfactant molecules and these surfactant molecules can be organized. So, they are organizing and forming a cylinder and this cylinder you can synthesis something within this cylindrical space and then you remove the outside cylinder to give you the material that you want in the shape of a cylinder.

So, this is the templating factor this is aggregation of the surfactant molecules to form the template which looks like a cylinder. Then, you the b step you add or form your desired materials within this cylinder after the stage b after the stage c you remove whatever is outside. You are left behind with the material which you have made in inside this aggregated surfactant molecules structure. So, this kind of cylindrical micellar structure ultimately yields a nanowire, if the dimension of this wire the diameter is in nanometers and most of the time you get nanowires 5, 10 nanometers.

You can vary lengths from 100 nanometers to few microns very easily, this step you have to remove the surfactant molecules to get either you use appropriate solvents to remove the surfactants or you can heat this material. So, the surfactants molecules will all go away and you can left with the nanowire, so this the template based synthesis using surfactant aggregates to form nanowires.

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This is another example where you use these kind of surfactant molecules which are also called amphiphlic molecules. That means they have two properties one hydrophobic and hydrophilic both are there in the same molecule their two parts and here we are discussing synthesis of gold nano particles in micelles.

These micelles are made like as we said this is a cylindrical micelle made from surfactant molecules. So, in this case also you have micelles, but of course in this case these are spherical micelles and this spherical micelles are made of by some kind as you see two structures one thin tale and one solid rod and this kind of a polymeric structure is called a block copolymer. So, you have two parts to the polymer, so one part of the polymer how itself is a polymer is probably hydrophobic and the other part of the polymer is a another polymer made of more hydrophilic nature and the two parts are join together.

So, this is called a block copolymer and when these block copolymers arrange themselves like surfactants molecules at a particular concentration to form a micelle with all the parts of the block copolymer which are alike have come together. Those which are different are also close to each other, but away from the structures, so when you have this organization of a block copolymer to form a micelle, then you add your metal salt from which you want to make your metal.

In this case it is gold particles, so you have to add gold salt and this gold salt attaches to one part of the polymer which it likes say it attaches to say the more polar part of the block copolymer. So, all the dots as you see are attach to the more polar part of the block copolymer and then you can do a reduction or treading the code such that you get the nano particle at the center and that is explained more clearly in the next slide.

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So, what we were discussing is an A-B di block copolymer, which is first used to form a micelle using the surfactant like behavior of aggregation where like from the polar head groups in surfactants come together to form a micelle. The tales are pointing outwards in this case the block copolymer has two parts a polystyrene part and two vinyl pyridine part.

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So, the styrene part and two vinyl pyridine have different hydrophobicities.

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So, one of the parts the two vinyl pyridine which is more polar that forms the head group and arranges as close to itself and when you add Auric chloride acid which is the starting material to make gold nano particles. This auric chloride acid when you add to the solution of block copolymer bounds selectively to the vinyl pyridine based polymer and that is the vinyl pyridine part of the polymer is here. So, that is where the gold nano

particles, the salt of the gold nano particle gets attached and then a solubilized and then transform to the metal by reduction.

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So, when you use chemical reaction to reduce the gold ions to the metal, so in this step you finally, end up with the gold particle at the center and the block copolymer around it. So, this is a typical synthesis of using a di block copolymer as a template to make gold nano particles the di block copolymer appears to function like surfactants does and form a micelle. The auric chloride attaches to the head group and then can be reduced to form the gold nano particles at the center of the micelle.

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Now, another template that people use quite often are what are called nucleopore membranes, a nucleopore filter. Basically, it is a filter made of a plastic or polymer usually polycarbonate and which has got holes on it which are few microns in diameter. So, this is a typically a nucleopore membrane and can be used as a nucleopore filter, so how do you create these holes you expose the membrane to radiation and that radiation weaken the plastic. Then, you add certain chemicals or acid which can remove or make holes in those specific areas which are weaken by the radiation.

First, you weaken the plastic at certain positions depending on the design which you want where you want the holes to be created of microns size or some micron size. Then, once you expose them to radiation is a pattern manner, then you expose them to some chemicals and the weaken part of the plastic or the polymer which is pattern, then we will get generate these holes which can be used a nucleopore filter.

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So, these nucleopore membranes of filter can be used, people have used to synthesis compounds within those pores like polypyrrole, poly 3 methylthiophene and nanoporous nucleopore membrane was used. That means the hole switch will made where of the diameter of few nanometers and then this membrane which is basically a polycarbonate which was treated to some rays or radiation. Then, it was some acid or other chemical where used to make holes and then that pore nanopores membrane was used as a template.

This has cylindrical pores of the order of 300 angstroms to 10,000 angstroms in diameter which is 1 microns. So, from some 13 nanometers to 1 micron that is the diameter of the pores that you can create in these nucleopore membranes.

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They are linear cylindrical pores very much like the case that we discussed earlier, this can be considered linear cylindrical pores. If this diameter is what we see in the nucleopore membranes, this diameter will be around 330 nanometers to about 1 micron, but they were created from one sheet. In this case, there are different cylinders, but in the nucleopore membrane, there was one full sheet and then holes are made by selectively exposing to radiation at certain spots and then those weeks were then made in two holes by chemical treatment.

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So, that is the nucleopore filter, which you can start from a polymer membrane like a polycarbonate membrane.

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Here, you can get cylindrical pores of the size of 13 nanometers to 1 micron and the monomer solution is separated by the polymerization agent by these membranes out offset. Before polymerizing, you have only the monomer solution and have this membrane and once you add the polymerizing agent, then the nucleopore membranes will be in these pores. You will have this polymerization taking place and you will you can make compounds like polypyrrole and poly three methylthiophene has been shown to be made in these pores membranes.

These are basically made of polycarbonates films with holes drilled in them not by any drilling machine, but using some radiation. Then, we are chemically treating them to make pores cylindrical type of structure in which by doing reactions with monomers, you can polymerize and get nanostructures, polymeric nanostructures.

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Now, these also use a biomaterials for templating, so far we have discussed inorganic materials organic materials which polymeric materials we can also use biomaterials for templating. In general, we can call them as bio templates, some of the examples of such bio templates can be for example, bacterial cell surface protein. So, on the surface of the bacterial cell there are several proteins and these proteins will have some structure which will control the formation of other materials.

If these are used as templates, you can have a small size nucleic acid compounds which are of the size of nano and micrometer and they can be thought of. They can be used as templates during the formation of other materials, you can have hallow a compartments like structure in viruses and then you can use those viruses as bio templates. So, there are cases where hollow bio molecular compartments like viruses have been used to synthesis materials within the viruses. So, that is another example of bio templating, then we can we come cross a term which are called which is called S layers basically from single polypeptides.

If you can make many copies, multiple copies of a single polypeptide, then it can spontaneously highly regular nano porous lattices. So, you have a polypeptide chain and that polypeptide chain spontaneously aggregates to form regular super lattices that is there are there are ordered in space over a large dimension. This can be of various symmetry, so there can be you know rectangular symmetry hexagonal patterns of these polypeptides which arrange themselves.

So, one single polypeptide make multiple copies of that which spontaneously aggregates in some pattern form and then leaves behind a leaves along with it some nano pores like this. Then, you can do synthesis in these nanopores which are having as the templates these polypeptide chain many numbers of polypeptide chain starting from a single polypeptide. So, in this if you do synthesis, and then it will be called bio templating because your template is a bio molecular, its polypeptide chain and hence these are different examples of bio templating.

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So, typically you make an S layer, that means you make a layer made up of this polypeptide chain which organized itself. So, it forms say a pattern and this pattern is we are showing in one dimension, but it can be like this in two dimensions. Here, what we are showing schematically is that these S layer which is organized as a pattern is on a TEM grid, because we want to use the TEM. So, you make this pattern on the TEM grid, so once it is on the TEM grid, then you coat this S layer with gold.

So, suppose you are able to coat this S layers with the gold because they are these bio molecules are functionalized and you can have thelo groups and to the thelo groups you can attach gold. So, suppose you can coat gold on these surfaces, so all the entire pattern now is covered with gold, but then you want gold in the center, you want it be pattern at the center. So, what you do is you can pass electron beam which is shown here the electron radiation and all the gold from here then enters into the cavities in between the gaps between these to ordered sub units of the polypeptide chain or the S layer.

So, you can have gold particles in these gaps which are which are like kind of melted and reunited to form droplets on being exposed to an electron beam. So, this is an example of bio templating, so you have a s layer pattern based with single polypeptide chains and you have gold coating on top of them.

Then, you irradiate with electron and all these gold forms droplets and they are arranged regularly, because this phasing is fixed and so you can have now regular pattern of gold nano particles made using a bio template route.

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Then, you can use magneto tactic bacteria as a bio template what is this magneto tactic bacteria these are bacteria which as which are found mainly in this, see and the word magneto tactic tells you that they have some magnetic property associated with them. So, these are in biology this is a group of prokaryotes and they are especially interesting since they orient and migrate along with the magnetic earth magnetic fields which is called the geomagnetic field lines. So, that means the bacteria can find out where is the earth north pole and the earth south pole, so the magnetic lines of force of the earth, the bacteria can find out. So, how can it find out that is because it has got some magnetic particles inside the body.

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So, this migration of these bacteria based on the earth's geomagnetism is related to the magnetic structure or the magnetosomes inside these bacteria. These magnetosomes are nothing but ferric oxide Fe 3 O 4 particles, which are also called magnetite particles which are bound inside the membrane of these cells and these are called magnetosomes.

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So, to explain to you what is a magnetosomes and how this happens is if you consider this to be a cell, so there is outer membrane of the cell and there is a cytoplasmic membrane inside you have the Cytoplasm. So, it is known that inside the cytoplasm there are these magnetosomes, so these are the magnetosomes and the inside the magnetosome you have this Fe 3 O 4, which is magnetic in nature. It will be attracted or interact with magnetic field and this kind of particles are present within the cell of the bacteriaof the magneto tactic bacteria.

That helps it to guide to move the bacteria based on the magnetic field, now this Fe 3 O 4 particle cover with the membrane which is called the magnetosomes membrane. So, here what we show is how does this Fe 3 O 4 form inside the cell, so what is known today is that there the Fe 3 plus ions from outside the cell. So, it is in a solution which is outside and can come in through this cell membrane and form Fe 2 plus, but it is the exact mechanism is not known how F e 3 plus comes within the cell wall from F e 2 plus within the cell it enters this magnetosomes.

So, we have pictured the magnetosome like this where one it comes inside you get ferric oxide which is hydrated. So, all this iron is Fe 3 plus and here Fe 2 plus is there, so they together Fe 3 plus and F e 2 plus give you Fe 3 O 4.

So, ultimately you get a particle which has got Fe 3 O 4 inside outside there is a membrane and the whole thing is called a magnetosome and this magnetosome with Fe 3 O 4 inside the cell. Many of these are there and they control the movement of the bacteria in the presence of a magnetic field and how the formation exact formation how does the ions move way inside. Still certain questions, but lot of it dependents on the pH and other redox behavior in these magnetic tactic bacteria.

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This is real TEM pictures of these bacteria, so you can see these particles magnetosomes with the magnetic particles they align themselves in S, some particular direction inside the cell and this direction is guided by the earth magnetic field. So, you can find many different types of the shapes of these magnetosomes, they can be little bit hexagonal like here or they can be like cuboids. The most important thing is most of the time they will be aligned in a certain manner and this alignment is important for the bacteria to navigate based on the earth's magnetic field.

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So, this is an example of bio templates, where the cell is the template the magnetosome is template inside which the bacteria is forming inorganic materials Fe 3 O 4 is an inorganic material. The formation of that is guided by the structure of the magnetosomes, now next after this we will come to the next lecture, which is on template methods. Again, part two with which we will continue in our next lecture, which will be the lecture eight of module 2.

Thank you.