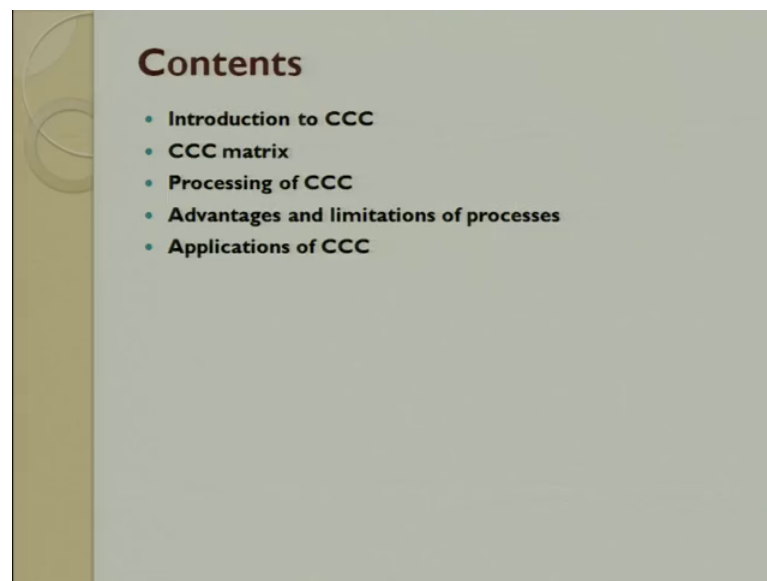


Manufacturing of Composites
Prof. J. Ramkumar
Department of Mechanical Engineering
Indian Institute of Technology, Kanpur

Lecture - 22
Carbon - Carbon Composites

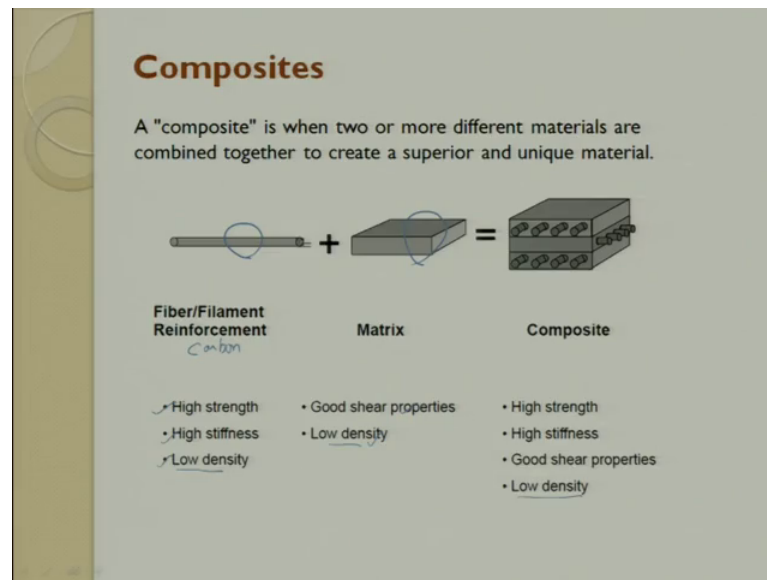
Lecture number 22. So, today we will be looking into Carbon-Carbon Composite, which is one of the star requirement in aerospace industry. And automobile also slowly we have started taking care of these carbon-carbon composites. Carbon-carbon composites are where in which the matrix is a carbon the reinforcing agent is also made out of carbon. So, in this lecture we will have a content introduction to CCC, and then we will have different matrices. Then we will see the processing techniques, advantages and applications.

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These will be the content.

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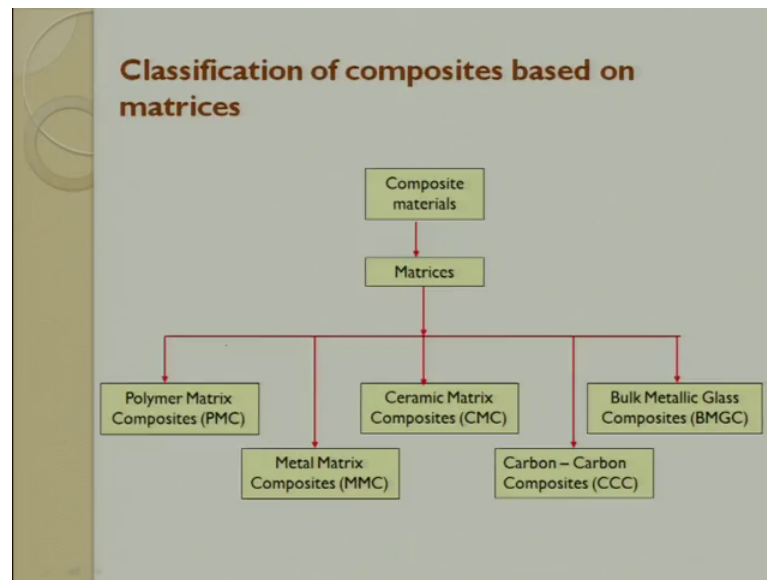


So, here a composite you are trying to use fibre, which is made out of which is made out of carbon material. Then you will also have a matrix which is made out of carbon material, these 2 put together we try to get a carbon-carbon composite. The function of the filament here is to give high strength high stiffness, and it also tries to give you low density. The matrix it has to have good shear strength property and low density. So, you see here this is also low this is also low.

Naturally the composite what we get is going to have a very low density. So, it will have good shear strength property from the matrix, high stiffness and high strength comes from the fibre what we use. So, we will use fibre which is made out of carbon. So, it can be a single wire, it can be a mat a 2 d or it can also be a 3 d. So, depending upon the requirements we choose a proper reinforcement matrix is made out of carbon and then we try to make composites. So, when I said matrix is made out of carbon.

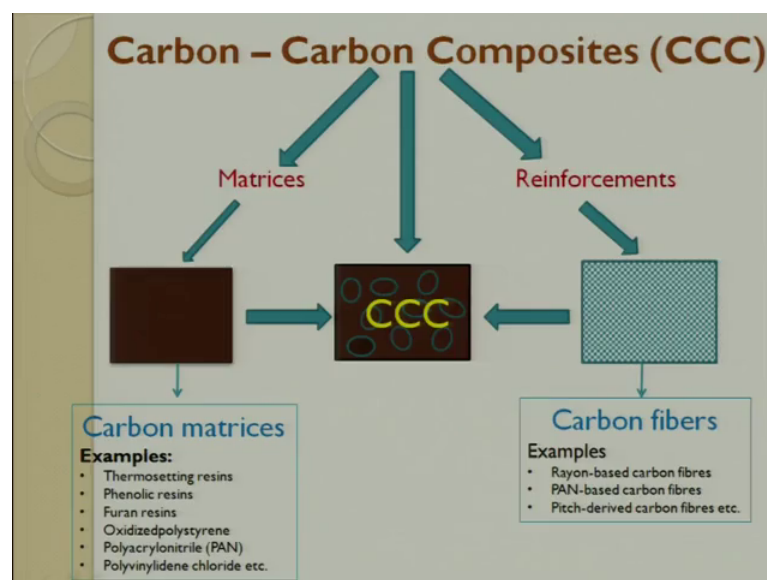
So, quickly since we have studied about the ceramic matrix composite, what should come to your mind is we will try to use a polymer we will try to do pyrolysis and we get converted into carbon. So, this which should be the basic thought we will see the process whatever we have lined up here.

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So, matrix the matrix can be polymer matrix composite, metal matrix composite, ceramic matrix composite, the most interesting and which is finding lot of applications are carbon-carbon composites. Then we also have bulk metallic glass composites. This is a next field which is coming up which is called as BMG composites. In this lecture we will more focus towards about a CC composites.

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So, a matrix; so what are the different types of carbon matrix. They are thermosetting resins since can be thought of phenolic resins. You have furan resins, then oxidized

polystyrene we have. Then we have pan resin then we have polyvinylidene chloride. So, this under etcetera you have many more resins which are available these are all the possible matrix for carbon. So, you in the carbon fibre can be made out of rayon based, can be made out of pan based, can be made out of pitch based; so rayon and pitch.

These 3 are the starting materials which are used for making a carbon fibre even today the it is a state of the art technologies are available, only with few people for making highly pure carbon fibres. Up to 90 92 percent or 95 percent it is easy to make pure carbon fibres it is easy to make anything, above that it is IP rides it is a preparatory item. Only very few companies across the globe hold the rights for making this carbon fibre, they make it out of rayon pan or pitch.

These are the set of matrices where and which this can help in making a carbon matrix. So, matrix reinforcement put together forms a carbon-carbon composites.

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Introduction

C/C composites refer to a family of complex advanced materials that consist of carbon fibers (mainly graphite form) fibers embedded in a carbon (mainly graphite form) matrix.

- Carbon Carbon Composites are those special composites in which both the reinforcing fibers and the matrix material are both pure carbon.
- C/C Composites are the woven mesh of Carbon-fibers.
- Carbon-Carbon Composites are used for their high strength and modulus of rigidity.
- It is light weight material which can withstand temperatures up to 3000°C.
- C/C Composites' structure can be tailored to meet requirements.

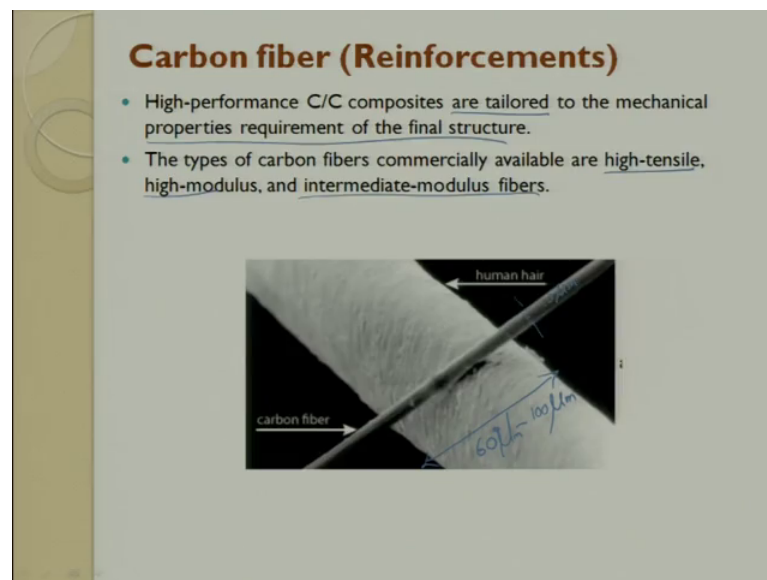
Diagram showing a woven mesh structure and a photograph of a cylindrical carbon-carbon composite part.

So, it is a family of complex advanced materials that consists of carbon fibre embedded in a carbon matrix. So, carbon-carbon composites are those special composites in which both the reinforcing fibre and the matrix are made out of pure carbon. They are made out of woven mesh of carbon fibre they are used for very high strength to modulus of rigidity they get.

And here they are they can withstand up to 3000 degree Celsius. This is very, very important. This property makes CC composite quite usable in aerospace industries. The space shuttle which comes back are now a day's made out of carbon-carbon composites which are light and weight, which can withstand very high temperatures and apart from that lot of filaments which are used for very high temperature furnaces they are also now a day's made out of carbon-carbon composite because they withstand very high temperatures. So, CC composite can be tailored to meet whatever is a customer requirement.

And today what has happened is they put carbon-carbon fibre and then they have another material up and down. So now, CC composite is used also as a insert. They are added in between sandwich. So, these assume this is a metal. So, you have 2 metals and then in between you have carbon-carbon composites for required application. Again as I told you here it can be in the form of a woven mesh. So, this is a 3 dimensional version. So, in this is a pre form where and which is made out of carbon-carbon-carbon fibres and through which the carbon matrix is infused.

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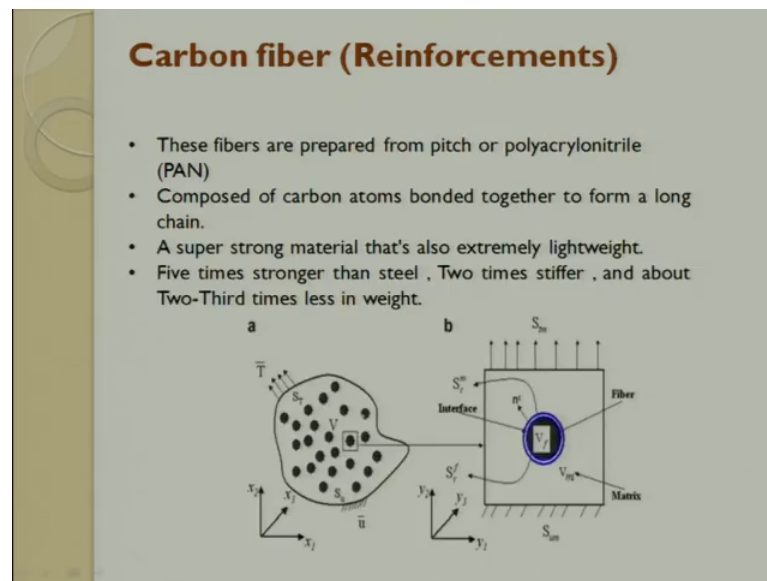


The carbon fibre which is used in the CC composite are tailored for to the mechanical properties of the final structure. So, for this the carbon fibre place a very, very important role. So, carbon fibres are having vey high strength of modulus, intermediate modulus fibre also available in this in carbon fibre. So, this is a human hair. And this is a carbon

fibre typically the human hair goes from 60 microns to 100 microns. This is the size of a human hair. It varies in this range, but you look at it this will be approximately 5 to 10 microns.

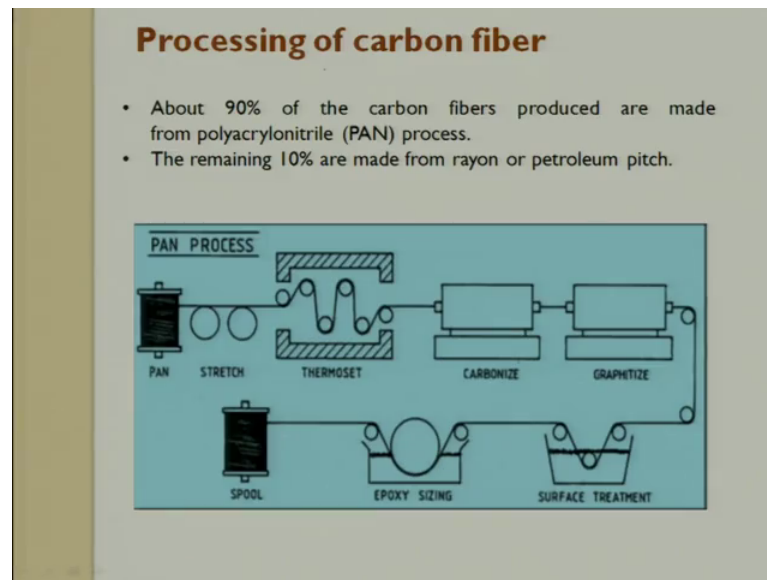
So, this is how the carbon fibre is made. So, here is the comparison between human hair and carbon fibre.

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The reinforcement, these fibres are the carbon fibres first are prepared from pitch or pan. The carbon atom bonded together to form a long chain. So, in pitch are pan what we do is we try to take it the carbon atoms are bonded together to form a long chain. This super strong material that also extremely light weight is a carbon fibre. They have 5 times stronger than steel, 2 times stiffer and about, two-third times less weight as compare to that of steel. So, this is carbon fibre. So, you see this is an interface right fibre interface. And this is the matrix. Carbon fibre has very good mechanical properties.

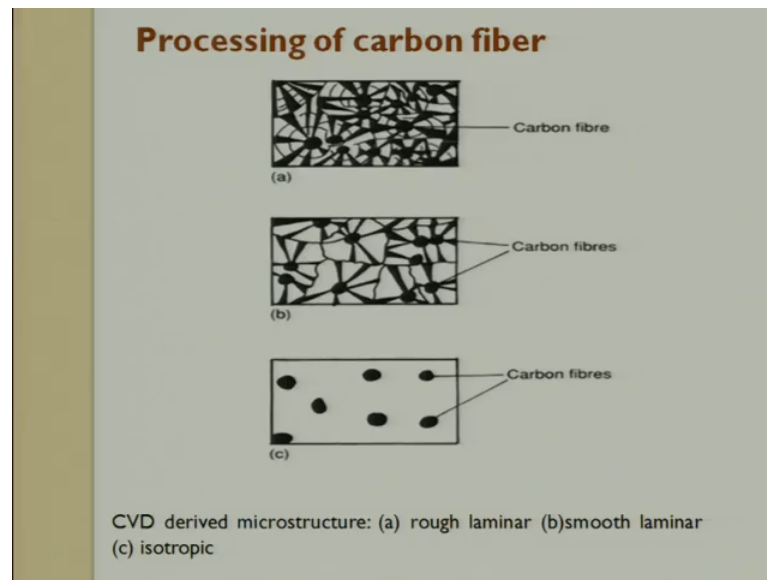
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So, how is the carbon fibre prepared? So, in a pan process, we take pan and then we try to stretch the fibre pass this fibre through a thermoset, and then we try to carbonize it. Then graphitize it, then we try to do a surface treatment, then epoxy sizing we do and then finally, we wind it in a spool. So, about 90 percent of the carbon fibre which are produced today are made out of pan process. The 10 percent is made from rayon process, and this is a very well established process.

So, because of this carbonizing and graphitizing, we need very high very high quality processing here. So, basically it has to be a furnace, and then there has to be gas carrier media which is there. So, these 2 should make sure that proper output is given. So, rest all treatment are straight forward. So, pan they will passes through spool. So, the carbon fibres are stretched. So, they are brought to high strain, and then it under goes in a thermoset. It expands and it is series of rollers, then carbonizing then graphitizing. Then you undergo this surface treatment epoxy sizing and then you try to do with spool.

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So, the processing of carbon fibre; so we have carbon fibres; so these are carbon fibres which are there you can say rough laminates. So, these are carbon fibres which are with rough laminates. You can also see with a smooth laminar you can also see with isotropic properties. So, these are the different processing of carbon fibres. So, with this you can see his are the microstructures which are made, and then carbon with laminate structure, laminar structure this is the smooth structure and isotropic structure is possible.

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Carbon matrix

- The carbon matrix in C/C composites has to effectively transfer the mechanical loads to the fiber reinforcements
- It acts as a binder to maintain the alignment of the fibers and fiber bundles and at the same time isolates the fibers from one another.

The most important criteria for the selection of polymer precursors to form carbon matrix are

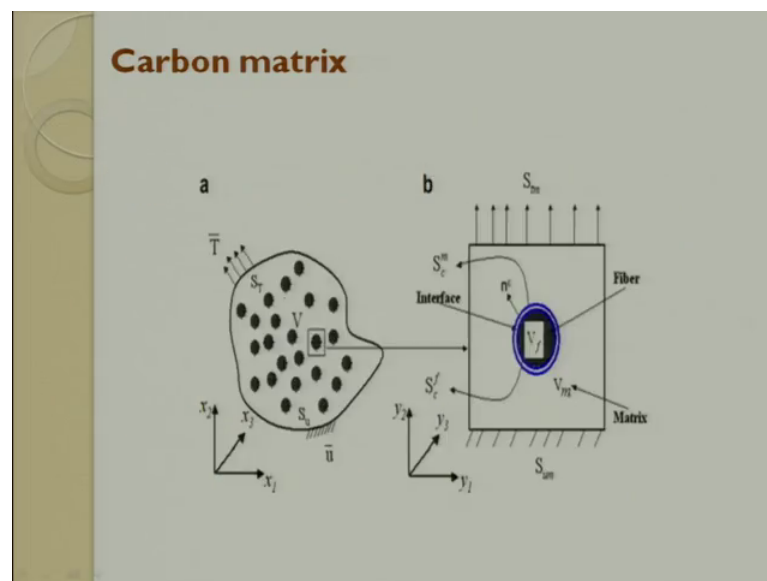
1. High carbon yield
2. Minimal shrinkage during pyrolysis
3. Amenable for all types of polymer composite manufacturing routes, such as resin transfer molding and filament winding
4. Low solvent content
5. High degree of pre-polymerization with lowest viscosity
6. Availability from multiple sources
7. Low cost
8. High pot and storage lifetimes

So, when you talk about carbon matrix. The carbon matrix in CC composite basic function is to transfer the mechanical load between the fibres, it has to bind the fibres. These 2 are well known. So, it acts as a binder to maintain alignment of the fibre fibres and the fibre bundle at the same time isolate the fibre from one and other. So, this is the basic function of a carbon matrix. The other important criterias for the selection of a polymer precursors to form, a carbon matrix should be the there it has to have a very high carbon yield. Why is that? Because I have, but a big list of polymers where and which you tried to do a proper processing it gets converted into carbon matrix.

So now, what should be the selection criteria? So, that is what we have listed it here. High carbon yield it should give it should have as minimal shrinkage during pyrolysis process. It should be amenable with all type of polymer matrix composite, when it follows a route of resin transfer moulding or filament winding. It should it should use low solvent content, it should have the highest high degree of pre polymerization with low viscosity. It should have the ability for multiple sources. It should be low cost it is part life that the storage lifetime should be very long.

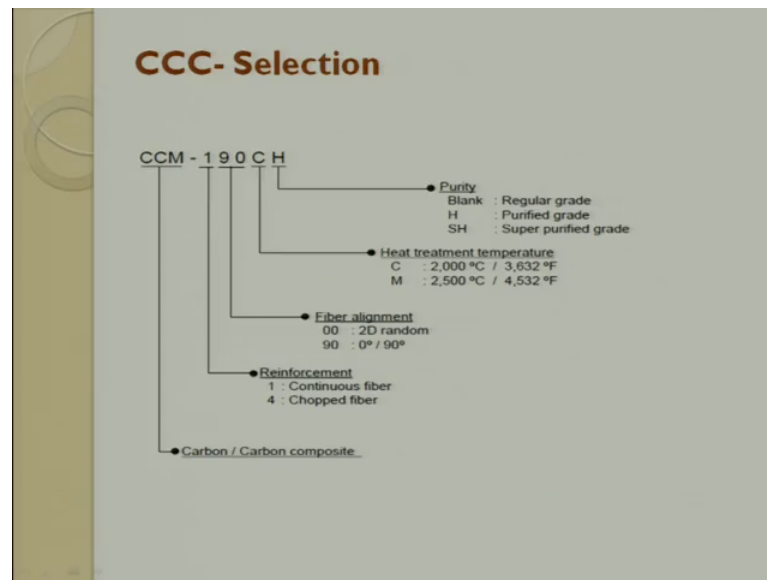
So, these are the conditions which are put in front of before choosing the polymer precursor.

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If you look at it this is a composite carbon-carbon composite, this is a carbon matrix. And these are the fibres which are used.

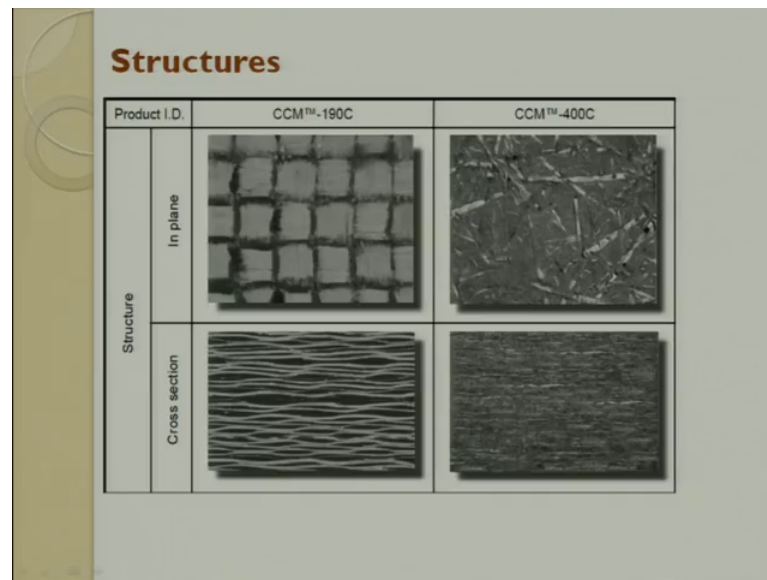
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If you generally the CC composite is has a nomenclature. So, this is the CCM it is written as carbon-carbon composite it says 1 or 4. So, one stands for continuous fibre, 4 stands for chopped fibre. So, you can put that signature digit there, then 90 is the orientation 0 90 the alignment can be put as 90 45 30 whatever it is. So, this is 0 0 means 2 d random and if you put 90 it is 0 90 orientation you can also have 45 the c is represent. This digit represents the heat treatment temperature. C means it is processed at a temperature between 2000 to 3632 degree Fahrenheit; if u put M, then M it is 2000 degree Celsius to 4532 degree fahrenheit. Or it is 2000 degree Celsius, and M is 2500 degree Celsius.

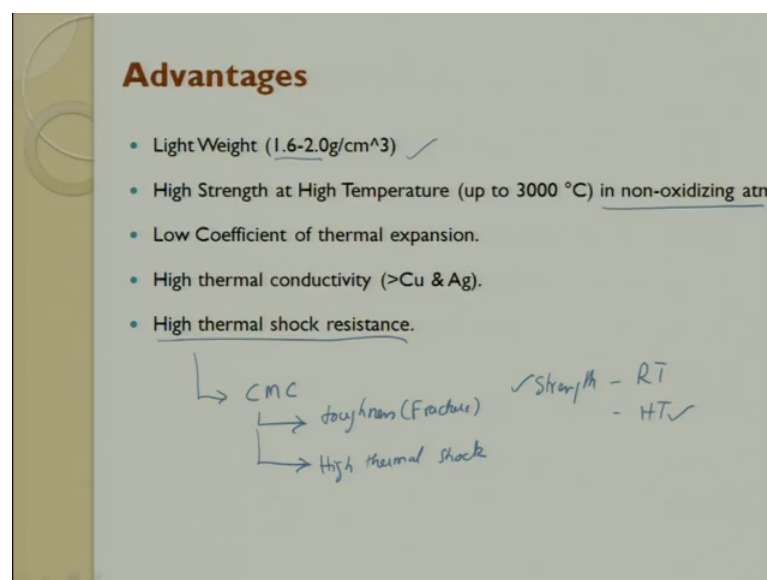
So, this h represents purity how pure is the carbon-carbon composite regular grade is purified grade SH is super purified grade. So, these are the grades. So, CCM composites can this is the signature which it follows the first digit talks for reinforcement next digit talks for alignment. Next digit talks for heat treatment and the last digit talks for purity of the composite.

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So, you can see here in plane and cross plane. So, here this is the product. So, you can see in plane, and this is also in plane, this is cross plane. So, you can choose this in plane and cross plane depending upon your requirement depending upon your final product you choose whether to go in plane or you go for cross section; so in plane and then you have cross section.

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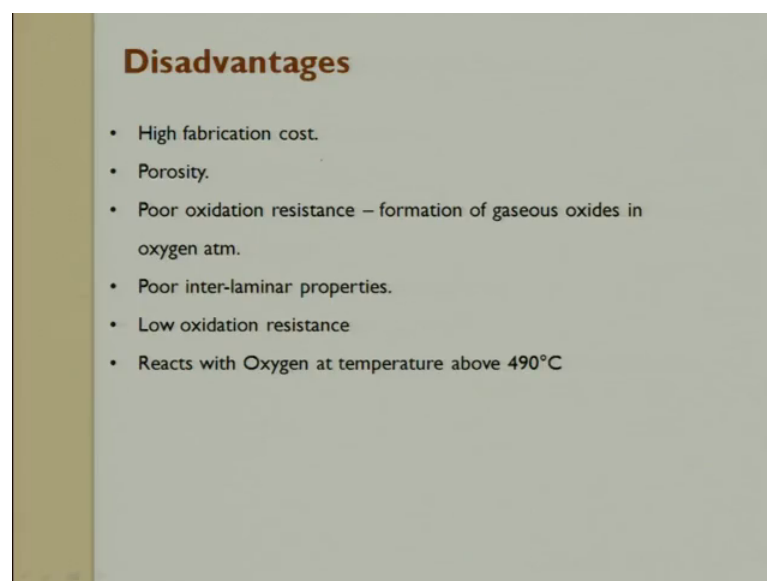
So, structures are there. So, what is the advantage of carbon-carbon composite. It is very light and weight you look at it is one point to 2 grams per centimetre cube. It has very

high strength at very high temperatures. For example, cutting tools and all you look you can (Refer Time: 13:52) they do not need strength at room temperature strength at room temperature, strength at high temperature. This is very important. Today our requirement are strength at high temperature requirements. Room temperature you can see the strength will be good hardness will be good, but very high temperature the strength the material get deformed moment at deforms.

Then it loses it is functional properties. So, high strength at high temperature is another big advantage it is for CC composite in a non oxidized atmosphere. Then it has to have low coefficient of thermal expansion, it has to have high thermal conductivity and it also has to have high thermal shock resistance. When we look at ceramic matrix composite, we said 2 properties are very important. One is the toughness property has to be enhanced or fracture toughness property to be enhanced. That is why we go from ceramics to ceramic matrix.

And the next is high thermal shock resistance. For a ceramic matrix composite when you go for carbon-carbon composite also we keep this as one of the biggest criteria, or this is one of the biggest advantage for choosing carbon-carbon composite. It has very low weight it has very high strength at high temperatures it has very low coefficient of expansion it has very high thermal conductivity. Today people are looking forward for heat exchangers made out of carbon-carbon composite.

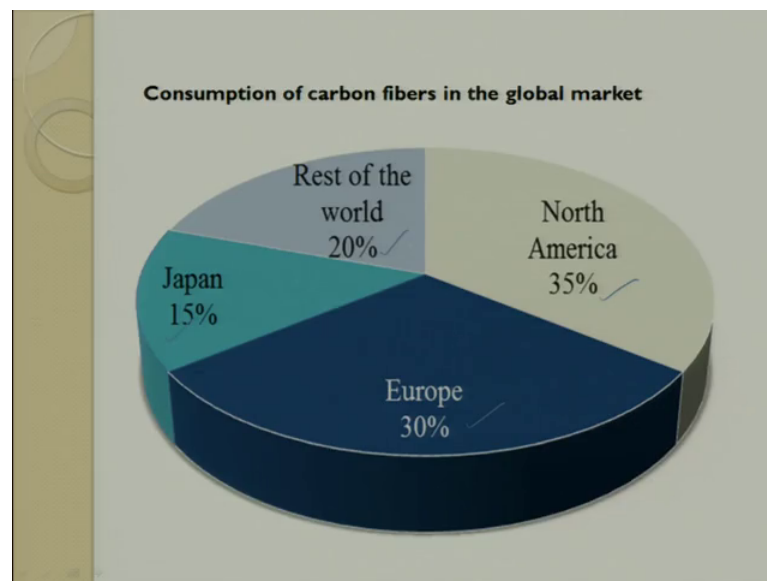
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But what are the disadvantages the disadvantages are the fabrication cost is extremely high. Because all the processes need furnaces these furnaces have should have gases in the absence of oxygen that is one the temperature what it operates should be very high which cannot be normally used in a in a furnace where heating coil is made out of steel or tungsten.

Next the process leads to lot of porosities. This process is followed is very similar to that of ceramic matrix composite. We will see that then it has poor oxidation resistance. So, when it is done when there is a few trace of oxygen, immediately the carbon-carbon composites the carbon matrix loses it is functional properties. It has very poor inter laminar shear strength. So, we have to be very careful before you seek it for structural applications. It has low oxidation resistance it reacts with oxygen even as slow at 500 degree Celsius it reacts and it form different compounds. So, the major disadvantages cost porosity and oxygen presence puts this carbon-carbon composite into still a very difficult component for manufacturing.

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So, if you look at it europe uses the maximum the north America uses maximum of 35 followed by europe, which they use carbon fibre to a large extent then we use Japan uses 15 percent and the rest of the worlds uses 20 percent. So this is; what is the consumption of carbon fibre in the global markets. So, slowly it is being used in Asian countries.

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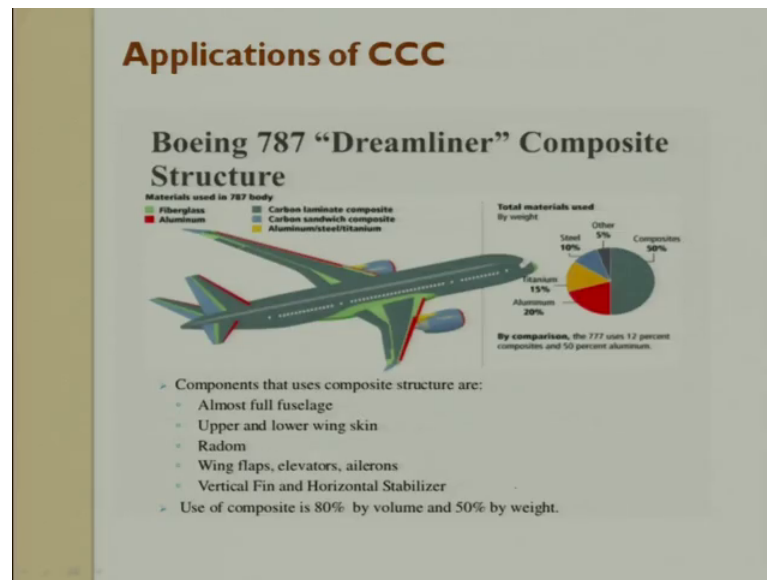


So, the applications of CC composites are it can be used for braking system where there it is going to be a high temperature. It can be used as a refractory material. It can be used as a hot pressed die for a turbo jet engine components they are today made out of carbon-carbon composite. So, wherever you can replace mat ceramic matrix composite we go for carbon-carbon composite. Ceramic has a fracture toughness property. So, here it does not have that, but; however, making CC composite also a challenge.

So, heating elements are made out of CC composite because they can withstand very high temperature missile cone tip because when it enters inside or when it goes outside the atmosphere it does a huge friction coming out. So, missile cones are made out of cone tips are made out of CC composite. The rocket motor throats are made out of it leading edge of space shuttles are made out of it heat shieldings are made x rays targets are made out of it aircraft brake. This braking system is an automobile today even in bikes people are started using CC composites. Aircraft brakes reentry vehicles biomedical implants engine piston because very high combustion if you want to have.

So, the temperature which has to be withstand by the piston should be very high. So, they are started using CC composites then we have electronic sinks, and other automobile and motor bike bodies are made out of carbon-carbon composites.

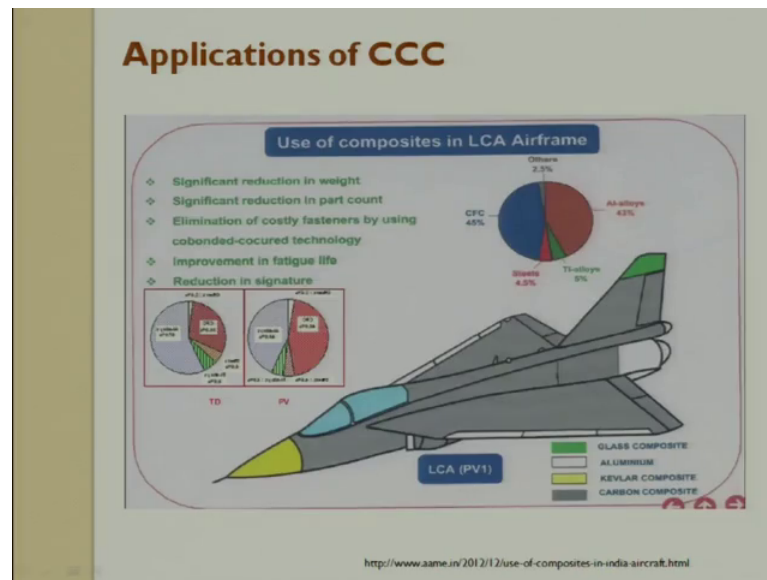
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If you look at Boeing dreamliner 7 8 7, the composites are 50 percent made out of this, and then we will have carbon-carbon composites carbon laminate composites are used in the tailed region. It is also used in the wings. The carbon sandwich composites are again used in the tails, and as well as in the wings we can see. And then it is also used in the turbo charge the casing is all made out of it. So, the components that are used for used uses the composite structures are almost the fuselage is made out of composite.

They are some are made out of glass fibre also, but now they are replacing it with carbon fibres of light weight. Then upper and the lower wings wing skin are made out of it radom is made out of it. Radom is in the front portion the wing flap elevators and the ailerons are made out of it; the vertical fins and the horizontal stabilizers. So, these are the horizontal vertical stabilizer which are made out of it. So now, the complete plane are moved towards carbon-carbon are made out of carbon fibre composites, and they are also slowly moving towards carbon-carbon composites.

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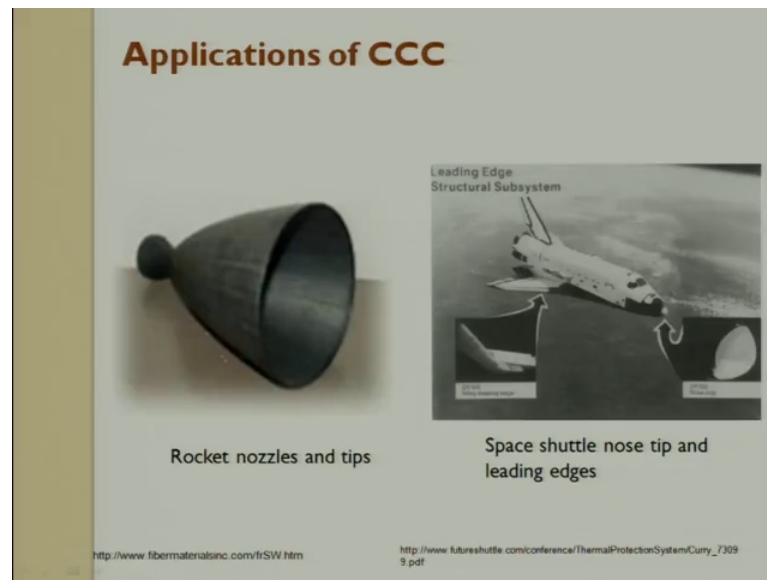


So, if you look at LCA airframe, these LCA airframes, they are made out of carbon composites you can see these are the portions which are made out of carbon composite. And slowly they are trying to replace this carbon composite with carbon-carbon matrix composites. So, here it was carbon composites where it was not carbon-carbon; that means, to say here only fibre was used. And polymer was used and slowly when in the high temperature engine. And other places they are replacing this carbon composite with carbon-carbon fibre composite.

So, significant weight reduction is there. Significant reduction in the part count also since it is going to composite complex shapes can be made and integration of inserts in the parts can be made of number of parts are gone down. So, the fasteners are reduced because basically when you use composite materials you can think of using adhesives rather than making a hole. So, when we look into machining you will understand when you drill a hole there will be a lot of de laminations coming into. So now, a days it has gone to a adhesion bonding, when fatigue life is improved because we try we try to choose proper interface between the matrix and the fibre. So, we try to enhance the fatigue life that crack growth can be arrested. And to large extent the signatures are reduced in the LCA frame. So, this is the carbon fibre composites 45 percent it is tried using.

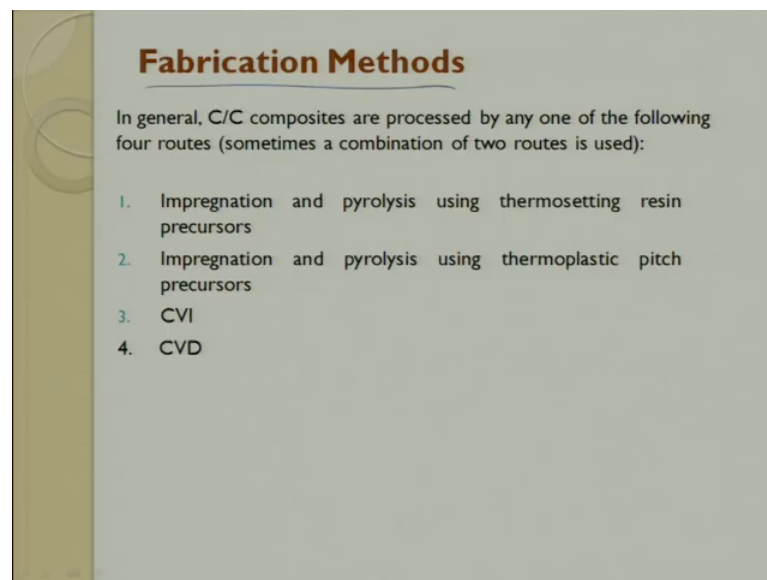
So, this is the rocket nozzle tip which is made out of carbon-carbon composite.

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So, this withstands very high temperature. So, a space shuttle nose tip are made out of carbon-carbon composites. (Refer Time: 21:34) of very high temperature.

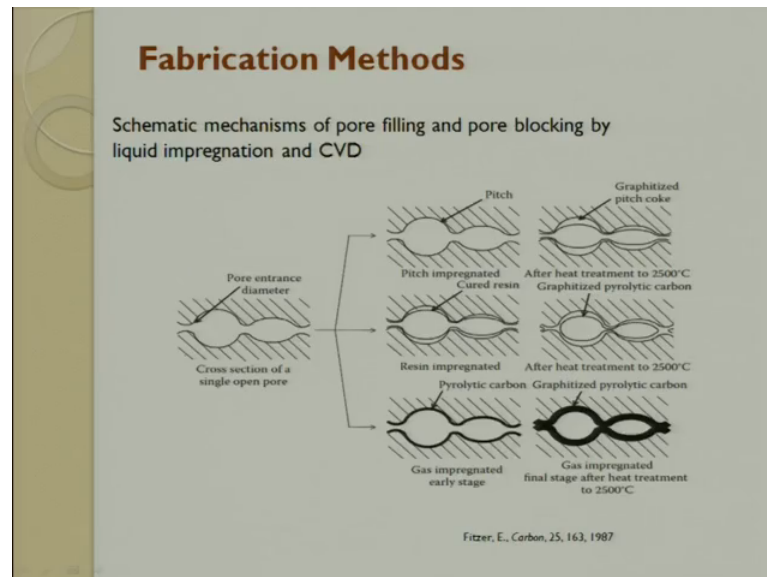
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So now slowly let us get into the fabrication methods of making CC composites. So, here there are 4 methods which are predominantly used. 4 routes sometimes a combination of the 2 routes are also used. See manufacturing there is nothing called as a unique solution. So, depending upon the availability resources depending upon the output required. A combination is always thought of and we tried to get the required output.

So, here now we have the 4. So, one is impregnation and pyrolysis using thermosetting resin impregnation and pyrolysis, using thermoplastic pitch precursors then we have a CVI route we have CVD route.

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So, fabrication Method here is a schematic diagram which is given; so the schematic mechanism of pore filling and pore blocking by liquid impregnation and CVD process. So, here what we do is we these are the pores pore entry entrance diameter, and this is the cross section of a single pore.

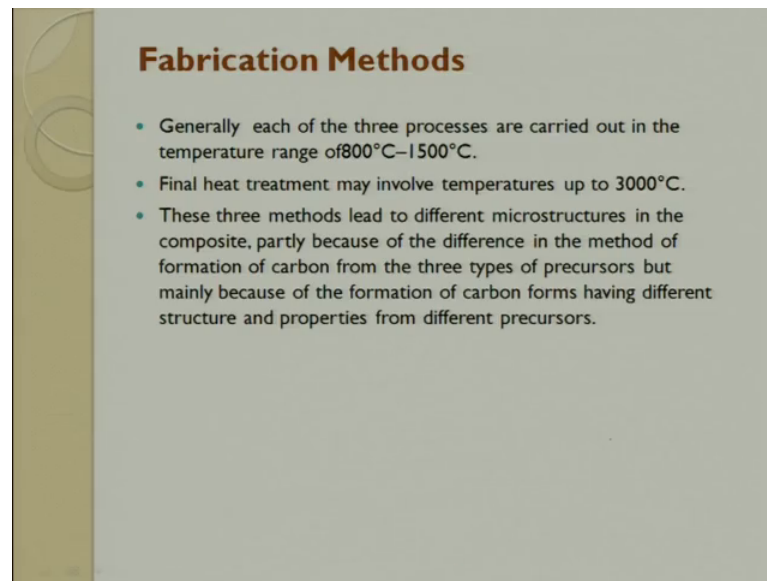
So, this is pitch impregnated. So, what we have is this is pitch impregnated pore is there. So, then what we do is we try to graphitize this pitch coke we try to do it. So, what we do is we take this pitch impregnated, then we heat it to 2500 degree Celsius. So, then the resin is impregnated. Then this pyrolytic carbon is formed and then the gas impregnation early stage is like this. So, then what we do is graphitization of the pyrolytic carbon happens, then after heating at 2500 degree Celsius, there is a graphitized pyrolytic carbon which is getting formed and the gas impregnation final stage heat treatment to 2500 degree Celsius, we make a carbon-carbon composites by this process.

So, here is a pore this is a cross section of pore. So, this can be done by 3 routes. So, one we tried to put the pitch here. And the pitch is impregnated here right and then what we do is here we try to graphitize the pitch coke. Then how do we get this done we heated to whatever it is we heated 2000 we heated to higher temperature 2500 degree Celsius, then

we it forms like this. And then we try to pitch coke is formed. Then here what we do is we try to use the cured resin right. And then this cured resin is graphitized pyrolytic carbon is pushed into. So, we get something like this.

So, then pyrolytic carbon is like this and this is the earliest stage of impregnation and this gas impregnation final stage after 2500 degrees we get the required carbon-carbon composite.

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So, the fabrication method generally each of the 3 processes are carried out at a temperature between 800 degree Celsius to 1500 degree Celsius. The final heat treatment goes up to 3000 degree Celsius these 3 methods lead to different micro structures in the composite partially, because of the difference in the method or the formation of carbon from the 3 different types of precursors, but mainly because of the formation of carbon forms having different structures and properties for different precursors. So, these are the 3 precursors with these precursors. We are trying to get different types of CC composites. The thermosetting resin based processor for carbon-carbon composite.

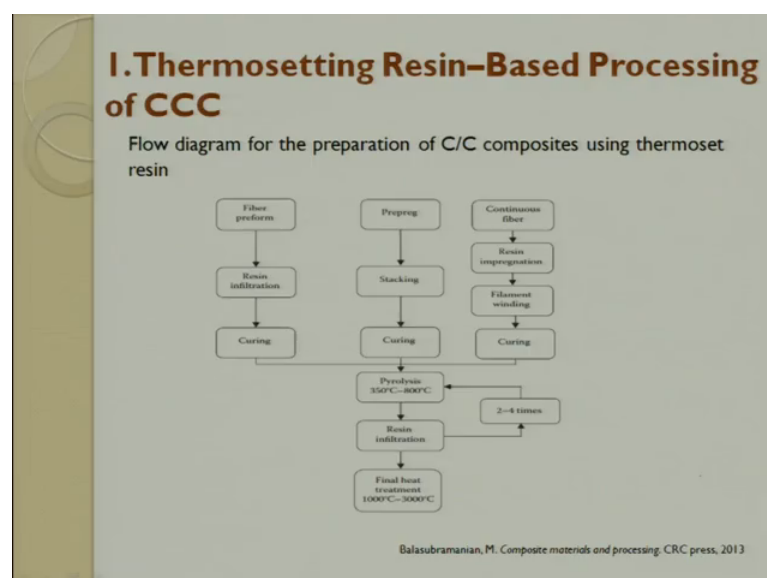
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I. Thermosetting Resin-Based Processing of CCC

- The resins are usually dissolved in an organic solvent or furfuryl alcohol with a catalyst/curing agent before infiltration.
- The resin in the impregnated composite is cured and then pyrolyzed by heating it to a temperature in the range of 350°C–800°C
- Frequently hot pressing at pressures up to 10 MPa and temperatures in the range of 150°C–350°C for periods up to 10 h are used to enhance density during the curing process.
- The pyrolyzed composite is subsequently graphitized at temperatures in excess of 1000°C

The resin are usually dissolved in an organic solvent, with a catalyst slash curing agent before infiltration the resin in the impregnated composite is cured, and then pyrolyzed by heating it to a temperature of 350 to 800 degree Celsius. Frequently hot pressing at high pressures up to 10 mega pascals and the temperature from 150 to 350 degree Celsius for a period of 10 hours it is done had to get intense density in the curing process. Then the final pyrolysis of the composite is subsequently graphitized at 1000 degree Celsius we get a CCC composite. So, in this if you see very clearly time pressure and temperature. Are very important and then here in the absence of oxygen is very, very important.

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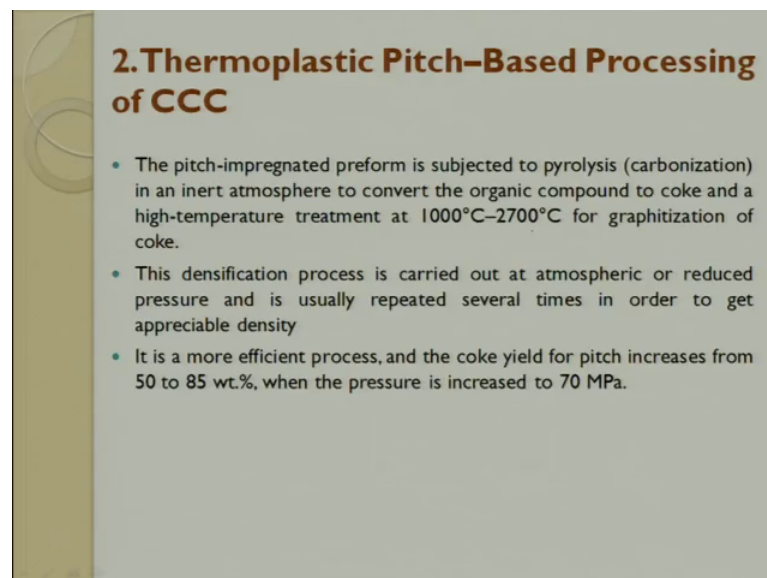


So, if you look at it following is the flow diagram for CC composite using thermosetting resin. You have a fibre preform this is resin is getting impregnated into it, then we do up curing. The next stage is get a prepreg then stacking of the prepreg you do curing. So, prepreg is where already polymer is fitted into the resin. So, you have a continuous fibre then resin impregnated. We do filament winding and then we do curing. So, when all these things are cured you go to pyrolysis. So, pyrolysis is done up to a temperature of 800 degree Celsius, then what we do is moment the pyrolysis is over then there might be pores or some gap in between.

So, again what we do is we try to do a resin infiltration. And this process is repeated multiple times until you get the final required dimensions strength or thickness or whatever it is. Then once it is done then finally, we do one more heating from 1000 degrees to 3000 degree Celsius to get the required output. Through this processing route we try to get a carbon-carbon composites manufacture using thermosetting resin. So, preform pre peg and then we use continuous to get the carbon-carbon composite made.

So, we can also use thermoplastic pitch based processing, so for carbon-carbon composites.

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2. Thermoplastic Pitch-Based Processing of CCC

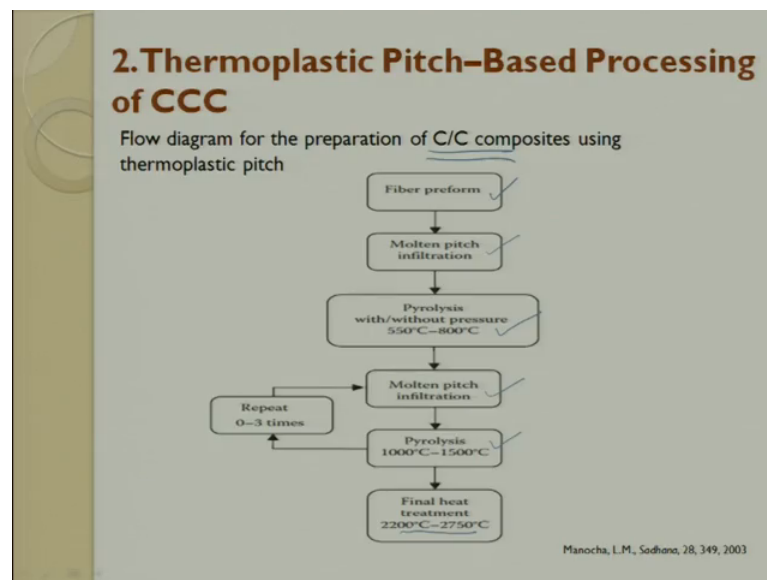
- The pitch-impregnated preform is subjected to pyrolysis (carbonization) in an inert atmosphere to convert the organic compound to coke and a high-temperature treatment at 1000°C–2700°C for graphitization of coke.
- This densification process is carried out at atmospheric or reduced pressure and is usually repeated several times in order to get appreciable density
- It is a more efficient process, and the coke yield for pitch increases from 50 to 85 wt.%, when the pressure is increased to 70 MPa.

So, the pitch impregnated preform is subjected to 2 pyrolysis carbonisation; in an inert atmosphere to convert the organic compound to coke, and the high temperature treatment at thousand to 2000 degree Celsius for graphitization of the coke. So, first it forms pitch

into is converted into organic component to coke, then we try to do this coke to get graphitized of this coke to get the required output.

So, the densification process is carried out at atmospheres at atmospheric or at reduced pressure is repeated several times to get desired density, it is more efficient process the coke yield of for pitch increases from 50 percent to 85 percent by weight. The pressures which are used are 70 mega pascal the pressures are also very high.

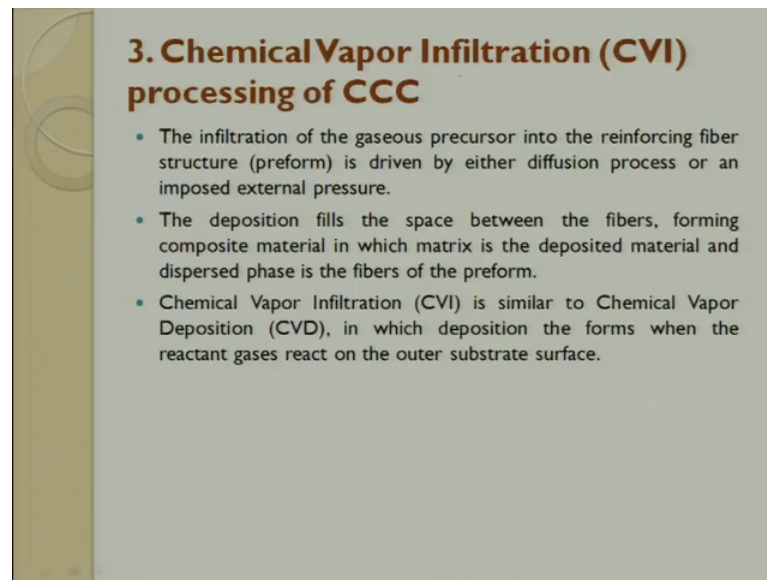
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So this is; what is the route flow chart for making it. So, fibre is taken in a preform and then we put the molten pitch in filtration is done fibre preform, molten pitch infiltration is done then pyrolysis is taken care the molten pitch infiltration happens. Then we do one more round of pyrolysis and we keep adding repeating the steps n number of times. And then finally we do a final treatment at this at 2000 to 2000 2200 to 2700 degree Celsius, to get the required output by this way we try to make carbon-carbon composites.

So, basically the resin is taken to a very high temperature. It is graphitized to get the required output. So, this is for thermoplastic pitch based processing.

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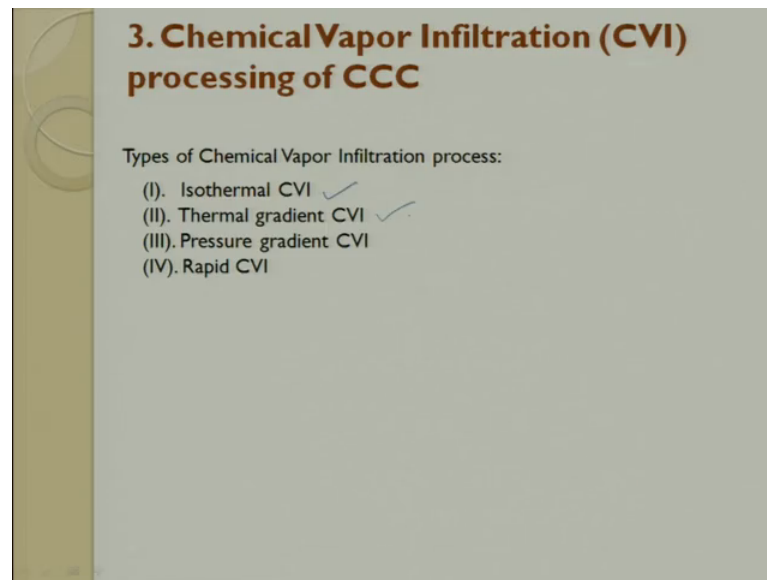
3. Chemical Vapor Infiltration (CVI) processing of CCC

- The infiltration of the gaseous precursor into the reinforcing fiber structure (preform) is driven by either diffusion process or an imposed external pressure.
- The deposition fills the space between the fibers, forming composite material in which matrix is the deposited material and dispersed phase is the fibers of the preform.
- Chemical Vapor Infiltration (CVI) is similar to Chemical Vapor Deposition (CVD), in which deposition the forms when the reactant gases react on the outer substrate surface.

The next one is chemical vapour infiltration process. In chemical vapour infiltration process the gaseous precursors is infiltrated into the fibre preform. This is driven by either diffusion process or it is imposed by a external pressure. So, either you push it or there is some phenomena which succeed capillary action. You get it done the deposition filling fills the space between the fibre forming and the composite matrix in which the resin is the is the deposited material. And the dispersed phase is the carbon phase the chemical vapour infiltration is a very similar process to that of chemical vapour deposition which we have seen in ceramic matrix composites.

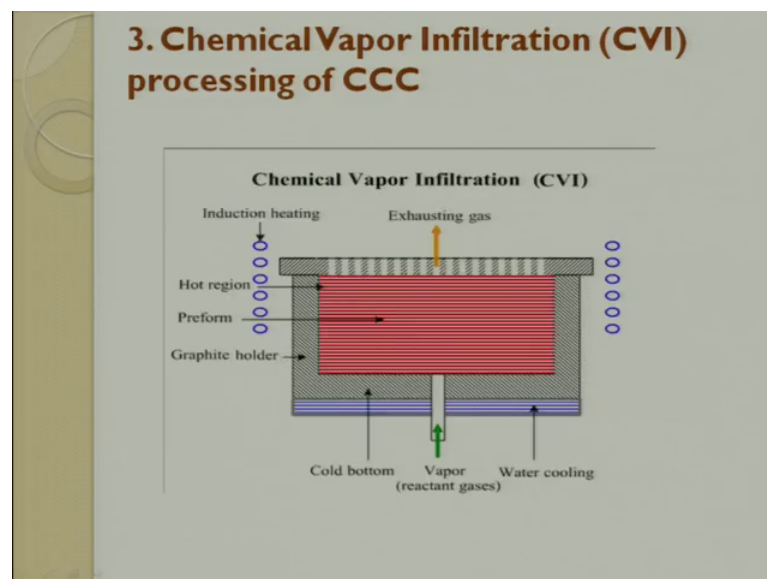
So, we use here precursor gas then we use carrier media carrier gas and then what we get is CVD. And this CVD is done on a substrate. So, on a substrate or on a fibre we try to get the required output. So, this is c v this is CVD process or CVI process here. So, what is the difference between the previous process and this process, in the previous process what we do is we take a fibre we take molten pitch which is infiltrated. So, all this things happen somewhere close to room temperature itself. But here in CVI what we do is we try to take the precursor the precursor is a gaseous precursor which is tries to pass through the resin fibre.

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So, I said told you last time in ceramic matrix composites, we have a isothermal CVI process, we have a thermal gradient CVI process, we have a pressure gradient CVI process, we also have a rapid CVI process for making carbon-carbon composites.

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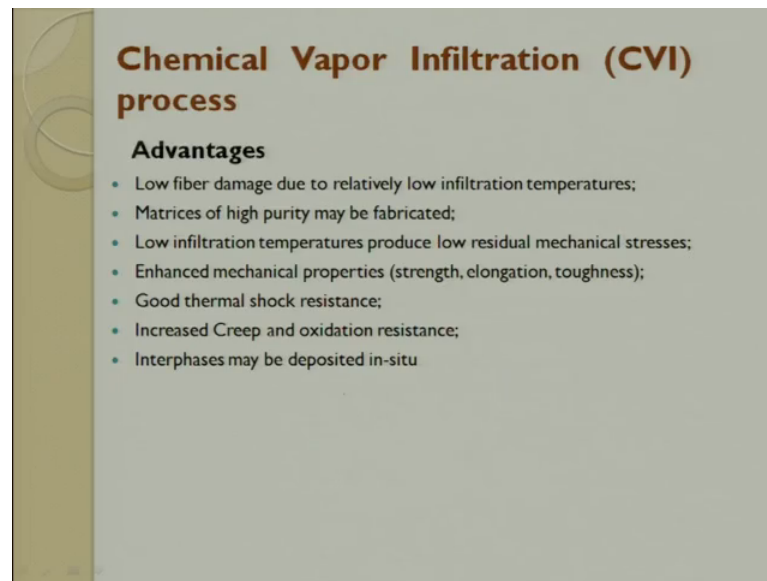


So, here through this the reactant gas is passed through. Here is the preform carbon fibre which is kept, this gas goes into reacts and then makes a carbon-carbon composite when they reacts. Whatever happens is only at a nation stage or in a green stage then after this it has to undergo 1 or 2 times of more graphitization. So, that the carbon-carbon

composites is formed. And here these vapours are infiltrated at regular intervals. So, that you try to have a proper densification of the carbon-carbon composite.

So, these are the induction coil for heating. This is the hot region preform is used and this is a graphite holder so that the graphite can withstand very high temperature otherwise they would have gone for a metal one metal or a steel 1. So, and here in the bottom they use a water jacket and then for cooling.

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Chemical Vapor Infiltration (CVI) process

Advantages

- Low fiber damage due to relatively low infiltration temperatures;
- Matrices of high purity may be fabricated;
- Low infiltration temperatures produce low residual mechanical stresses;
- Enhanced mechanical properties (strength, elongation, toughness);
- Good thermal shock resistance;
- Increased Creep and oxidation resistance;
- Interphases may be deposited in-situ

So, what is major advantage low fibre damage happens here, then highest purification can be done low infiltration temperatures are required, enhanced mechanical properties can be done. Good shock resistance happens increased creep and oxidation behaviour comes. Interface can be deposited in c 2 we want to enhance.

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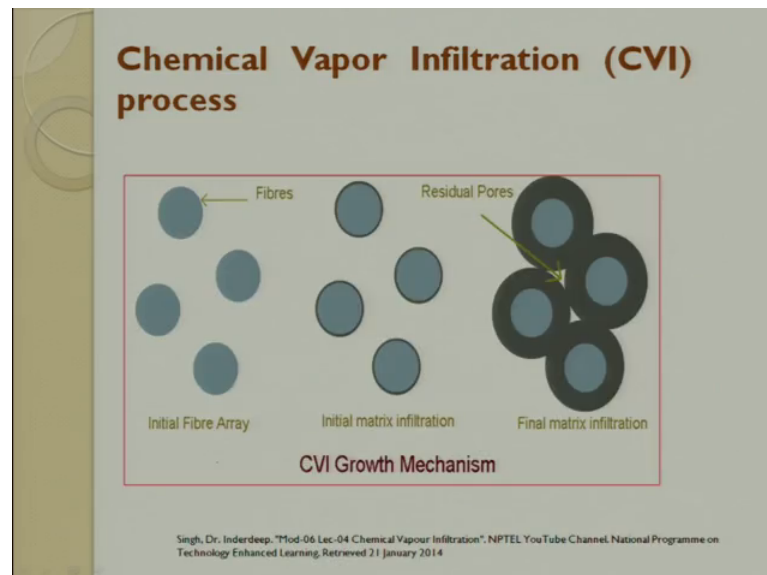
Chemical Vapor Infiltration (CVI) process

Disadvantages

- Slow process rate (may continue up to several weeks);
- High residual porosity (10-15%);
- High capital and production costs.

What are the disadvantages of this process? It is a slow process. It can take even several weeks to have it. It has a porosity is always part of the product. So, this porosity will try to give a very less or this will have to compromise a mechanical property. It is capital intensive.

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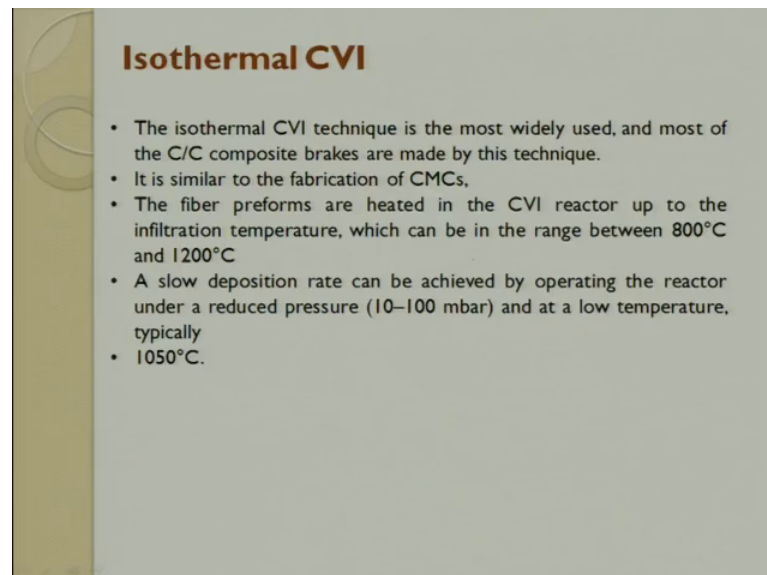


In CVI process this is how the CVI growth mechanism is there. You have initial fibre which are erase these are initial fibre erase. These are initial matrix infiltration happens right. And then what happens is this is first let us take this is one stage, and in the next

stage you keep on pumping it second time third time 4th time. So, you see that this initial matrix which was infiltrate the thickness increases. And we were talking about the residual pores of 10 to 15 4 percent these are the pores which are there because completely this portion is locked.

So, once it is locked the further gases cannot go through. So, this portion is a residual pores which is prone to happen here in CVI process the best pore; that means, to say the lowest porosity what you can get is around about 6 percent which are reported in the research publication, but generally it is 10 to percent.

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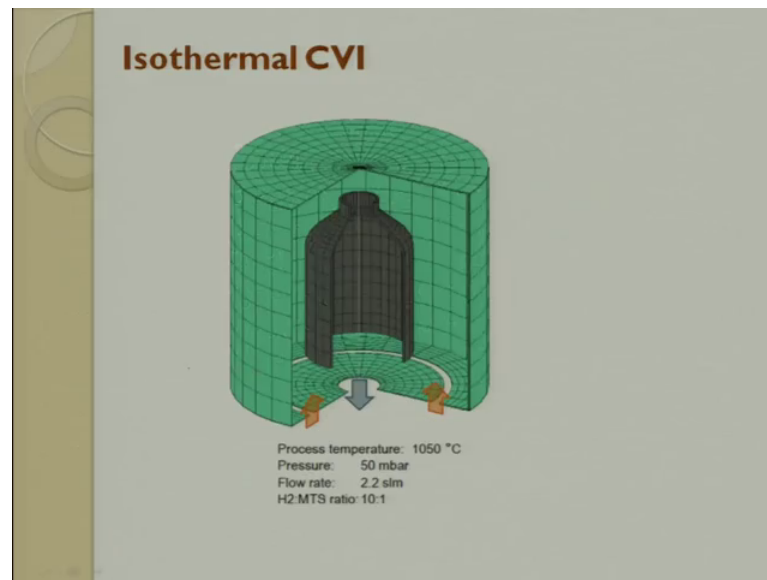
Isothermal CVI

- The isothermal CVI technique is the most widely used, and most of the C/C composite brakes are made by this technique.
- It is similar to the fabrication of CMCs,
- The fiber preforms are heated in the CVI reactor up to the infiltration temperature, which can be in the range between 800°C and 1200°C
- A slow deposition rate can be achieved by operating the reactor under a reduced pressure (10–100 mbar) and at a low temperature, typically
- 1050°C.

So, what is isotherm which we are discussing? There were 4 different types of CVI. So, what is isotherm CVI, isotherm CVI is the most widely used process and it is very similar to that of a ceramic matrix composite. So, hear the fibre preform are heated in a CVI reactor up to a infiltration temperature in the range of 800 degrees to 1200 degree Celsius.

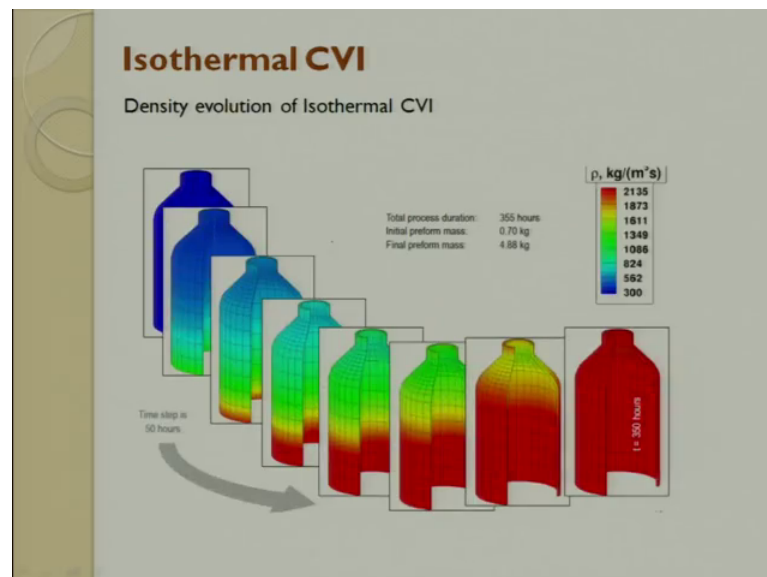
So, the in the fibre preform is heated reactor up to and infiltration temperature where the temperature ranges from 800 degree Celsius to 1200 degree Celsius are also deposition rate can be achieved by operating the reactor under reduced pressure, and a lower temperature of 1050 degree Celsius.

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So, this is a isotherm CVI. So, you have a process temperature of 10, 50 degrees Celsius. The pressure is 50 millibar, the flow rate is 2 point 2 S L M. And then h₂ is to M t s ratio is 10 is to 1. So, here this is an isotherm we push in gas inside the furnace and we start using it.

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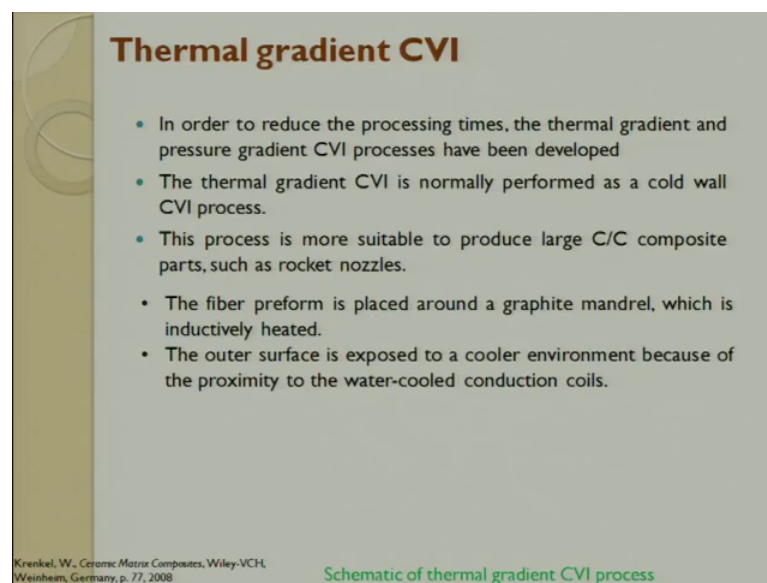


So, if you look at the density evolution of CVI. So, over a period of time we see that 100 percent the densification keeps on increasing. So, the process duration you see here it is almost 355 hours it is done. So, that you tried to get a final shape. So, this is the product

which was supposed to be made. So, for making this process it takes it take 355 hours. So, the initial preform was round about point 7 k g's the final proform mass was around about 4.88 k gs, around about 5 k g's.

So, there are 50 steps which are involved. So, every time what we do is we keep on pushing gas inside after every densifiquie after one cycle we try to push the gas once again. And then we start improving the densification. So, you can see the densities which is increase from 300 to 2135 millimetre cube.

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Thermal gradient CVI

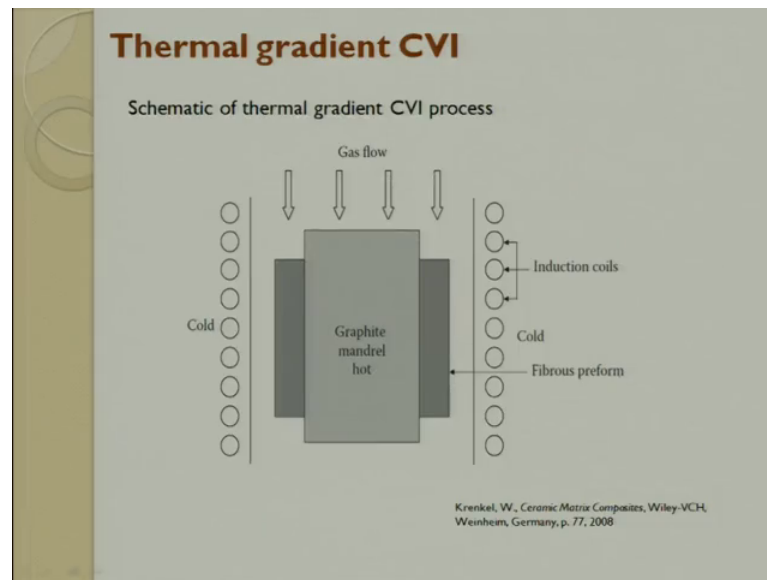
- In order to reduce the processing times, the thermal gradient and pressure gradient CVI processes have been developed
- The thermal gradient CVI is normally performed as a cold wall CVI process.
- This process is more suitable to produce large C/C composite parts, such as rocket nozzles.
- The fiber preform is placed around a graphite mandrel, which is inductively heated.
- The outer surface is exposed to a cooler environment because of the proximity to the water-cooled conduction coils.

Krenkel, W., *Ceramic Matrix Composites*, Wiley-VCH, Weinheim, Germany, p. 77, 2008

Schematic of thermal gradient CVI process

The thermal gradient CVI process in order to reduce the processing time; so if you see here it takes isothermal it is round about 355 hours. So, in order to reduce that time what we do is the thermal gradient under pressure gradient has been developed. So, in thermal gradient CVI is normally performed as a cold wall CVI process. This process is very suitable for large carbon composites such as the rocket nozzle are made out of it. The fibre preform is placed around a mandrel here. And which is heated. The outer surface is exposed to a cooler environment, because the proximity of water cooled cooling is possible here.

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So, this is; what is the schematic diagram for thermal gradient CVI process. This is a graphite mandrel. So, you place your preform the gas is flown from the top. So, the heating is maintained in the furnace. So, this is a fibre freeform this is a schematic diagram of thermal gradient CVI process. The cooling jackets are here. You can see the induction is given the circles are induction, and then after that you will have cooling, then you have a preformed fibre carbon fibre preform is reinforced preform is there you have a mandrel on which the preform is bound.

So now, you pass gas through it. So, you try to get the carbon getting infiltrated into the 3 form. So, you produce a carbon-carbon composites.

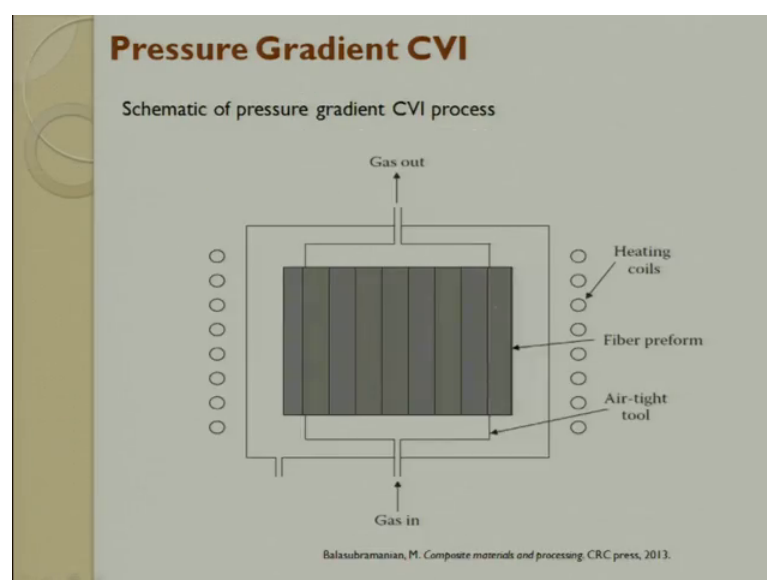
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Pressure Gradient CVI

- This method is a modified version of the isothermal CVI process.
- A C-fiber preform (often carbon felt) is placed in an isothermally heated furnace with an outer graphite tool, which only leaves a small gap for the forced gas flow of the precursor gas.
- A pressure difference that forces the gas flow through the pores is created across the wall of the structure
- This process is also limited to the production of single, simple shaped component at a time.
- Hence, this method may not be suitable for the commercial production of C/C composite parts in large numbers

So, you also have a pressure gradient which is a small variation modification of the isothermal CVI process. So, here carbon fibre is placed in an isothermally heated furnace with an outer graphite tool, which only leaves a small gap for the forced gas to flow. The pressure difference that forces the gas flow through the pores is created across the walls of the structure. The process is also limited to the production of a single or a simple shape only. Here, this method may not be suitable for commercial production of CC composite parts for a large area. So, this method is not used.

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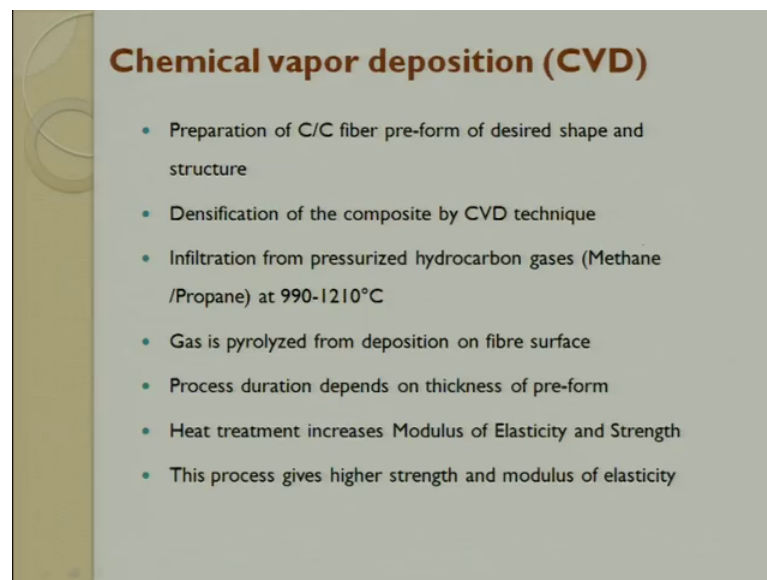


So, the schematic diagram is seen here. So, you have this is a fibre preform. This is the gas in, this is the gas out. So, you have heating coils and here it is sir tight. So, that you try to make simple geomentries out of it. So, there is a pressure gradient which is maintained such that you get the CVI done for making carbon-carbon composite.

The next process is called as rapid CVI process. Rapid CVI process technique developed by France this is still in a very, very nation states can be applied for industries language that densification which is done is very good. In this process of the precursor of carbon fibre preform acts as a carbon susceptor, and is fully immersed in a liquid hydrocarbon such as cyclohexane or a toluene. So, it is immersed inside and then we do the process. Out of the 2 hydrocarbon the carbon yield is higher toluene. The complete densification of CC breaks can be achieved within 10 hours by this process.

So, what we do is we try to take a porous carbon fibre preform, which acts as a susceptor and it is fully immersed inside a hydro liquid hydrocarbon and then you dry It to get the process done very fast.

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Chemical vapor deposition (CVD)

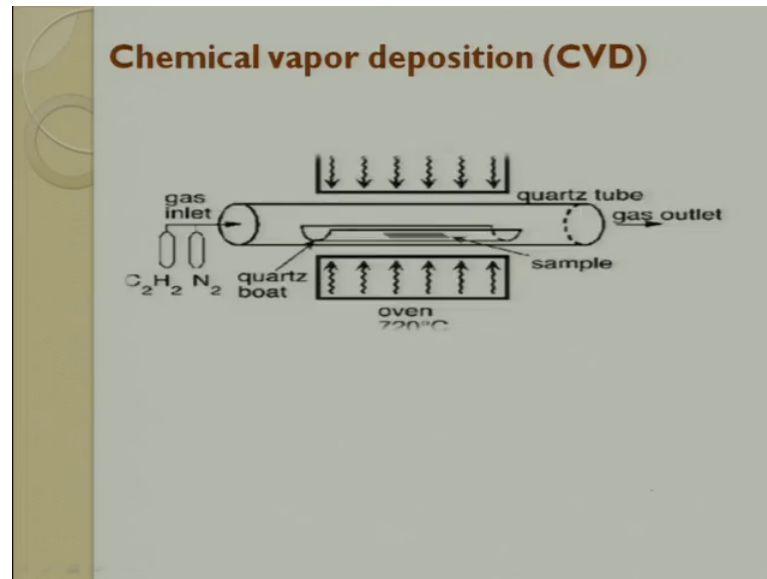
- Preparation of C/C fiber pre-form of desired shape and structure
- Densification of the composite by CVD technique
- Infiltration from pressurized hydrocarbon gases (Methane /Propane) at 990-1210°C
- Gas is pyrolyzed from deposition on fibre surface
- Process duration depends on thickness of pre-form
- Heat treatment increases Modulus of Elasticity and Strength
- This process gives higher strength and modulus of elasticity

So, the other one is chemical vapour deposition. So, it is infiltration here it is deposition. Infiltration is pushing through, deposition is you deposit on top of it. So, here the preparation of CC fibre preforms of a desired shape and structure is done. The densifications by the densification of the composite by CVD techniques are pretty good. The infiltration from the pressurized hydrocarbon gas is around 900 degrees to 1200

degree Celsius. The gas is pyrolyzed from the deposition on a fibre surface. The process duration depends upon the thickness of the preform.

The heat treatment increases the modulus of elasticity and strength, this process gives highest strength and modulus of elasticity when we use is CVD process.

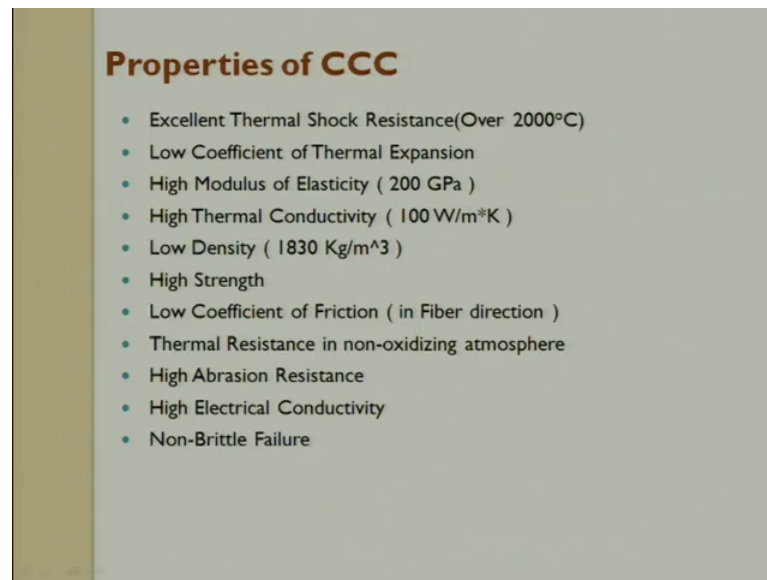
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In a CVD process we use gases, and then we use a carrier gas. It is passed through a furnace and where and which there is a maintained temperature of 720 degree Celsius. This is a quartz tube and there is a quartz boat which is their parent with sample is given by CVD the carbon is getting deposited on the fibre and we make a carbon-carbon composite. In this process this is quite slow and second thing is here there is a possibility of other impurities coming from.

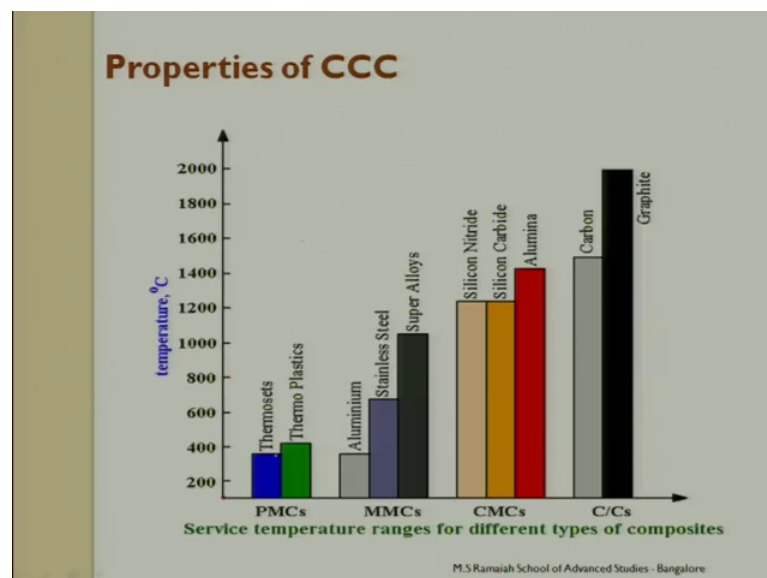
So, there has to be a proper control on the output. The limitation of this the hydrocarbon gas which is infiltrated into the inter filament surface and it also it forms an interfilament surface and a crack. Sometime this gas deposits on the outer shell and leaves lot of pores. The infiltration and the densification is required at the it takes a long time to do this process.

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So, if you look at the properties of the CCC composite. It has excellent thermal shock, low coefficient of expansion, high modulus high conductivity, low density high strength, high coefficient of very low coefficient of friction, high resistance in non-oxidized atmosphere, high operation resistance electrical conductivity. And non brittle fracture, these are some of the properties which are good for carbon-carbon composites.

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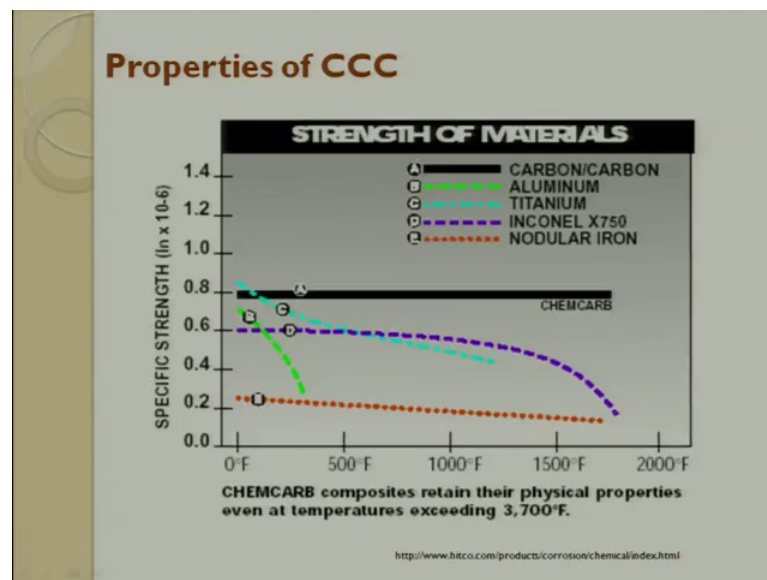


So, if you look at temperatures polymer matrix composite it can with stand a thermoset can go up to 300 or 400 degree Celsius. This can go up to 5 degree Celsius when you

look at metal matrix composite, it can go from 400 degrees Celsius, it can go depending upon your matrix requirement it can go up to 1000 degree Celsius. This is the service condition where it can work. Ceramic matrix composite it can go up 1300 degree Celsius, it can silicon nitride silicon carbide. Aluminium can go up slightly higher temperature of 1400 degree Celsius.

Look at carbon-carbon composite, it can go up to 1500 degree Celsius service condition easily a temperature around and when you do a graphitisation, when it is graphite it can go up to 2000 degree Celsius. So, this makes it a very, very viable matrix for high temperature applications.

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So, if you look at specific strength at varying temperatures, you can see that carbon-carbon composite the specific strength is almost constant up to 2000 degree Fahrenheit, whereas aluminium there is a decline. Titanium you can see over a period of time it is decline inconel for slightly higher temperature, and nodular iron with also there is a decline which happens over a period of time.

So, the carbon-carbon composite maintains the strength specific strength to a for a very high temperature as constant. So, it also depends upon the orientation. If you have this is a flexural strength for short fibres for cloth type and then for roving type plus minus forty roving type, plus minus 15 unidirectional fibre carbon fibre we use, and then we

tried to have multiple orientations, we can go have a flexural strength as high as 700 Newton per millimetre square.

So, if you look into it, this is as a climax for this carbon-carbon composite. So, if you see here carbon fibre reinforced plastics strength, versus temperature the carbon fibre false here. And moment you convert this matrix into a carbon. So, this is carbon fibre reinforced this is carbon fibre reinforced in a carbon matrix you can see here it works very nice from thousand 4, 1000 to 2000 degree Celsius it works excellently well. There is no compromise in strength and. In fact, you see here this is the major advantage of carbon-carbon composites used in very high service temperature conditions. With this we come to the end of this carbon-carbon composites fabrication, and we have seen different properties.

So, predominantly here we use thermoset matrix thermoplastic matrix, then we use CVI. In CVI we have seen 4 different variations to reduce the cycle time, and to enhance the quality of the output. We can also do exclusively CVD, but there is lot of limitation in this process. So, that is why we do not use CVD to a large extent, we follow only CVI process to get the carbon-carbon composites.

Thank you much.