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Lecture - 13 Testing of Fibre Reinforced Composite Materials (contd.)

Hello everyone. So, we will discuss the fibre matrix interface bonding strength measurement.

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Fibre-Matrix Interfacial bonding strength Measurement	
7	Fibre/matrix interfacial bonding is one of the major parameters which determines the mechanical performance of the composites
A	There are mainly <u>four types of bonding mechanism</u> which determine the fibre/matrix interfacial bonding strength
	 ✓ Adsorption & Wetting (a) ✓ Mechanical Keying (b)
	Chemical Reaction (c)
	(d)

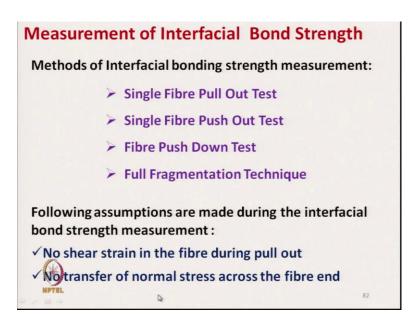
So, fibre matrix interface is the key factor which we need to know to assess the properties or quality of any composite material. So, fibre matrix interfacial bonding is one of the major parameters which determines the mechanical performance of composite. There are mainly four types of bonding mechanisms which actually determine the fibre matrix interfacial bonding strength.

These are adsorption and wetting. So, this mechanism is that the fibre has to actually adsorb the fibrous material should adsorb the matrix and it should get wet with the matrix material and it forms bonding. Mainly, this type of mechanism takes place in case of thermoset type of matrix, where the reinforcing material that is fibrous material get wet by the matrix material.

Next is that mechanical keying. So, the unevenness, the roughness present in the fibre

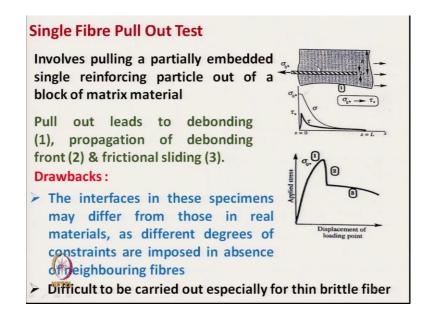
surface forms mechanical keying effect which increases the bonding strength. And third one is the chemical reaction, between the fibre and interface material and interface and the matrix material. And fourth one is electrostatic attraction. So, if we can create the electrostatic attraction between the reinforcing material and the matrix material we can create the bonding strength.

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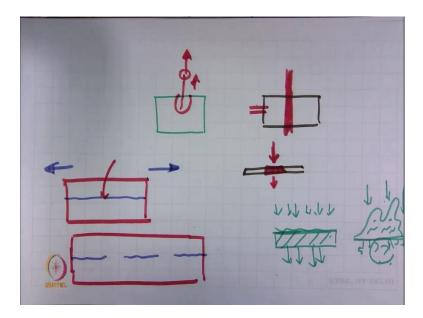
The measurement of bond strength is done using four different techniques, particularly for fibre reinforced polymers. So, these are single fibre pull out test, single fibre push out test, fibre push down test and full fragmentation test. So, the assumptions are during the interface bond strength measurement is that no shear strain in the fibre during pull out. So, once we try to pull out the fibre, so there will not be any shear strain in the fibre and no transfer of normal stress across the fibre end. So, with these assumptions we can test the bond strength.

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The single fibre pull out test it involves pulling a particularly fibre end which is partially embedded single reinforcing particle out of the block of matrix material.

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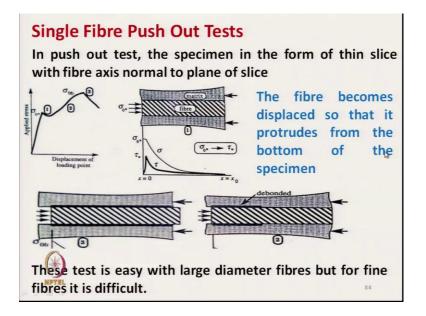


So, this is a matrix material block is created and the single fibre which is partially coming out from the matrix, this is only single fibre. And if we test, if we pull the fibre and once the fibre is coming out of this matrix from the composite material and this force is measured; that means, if the fibre breaks before it comes out then the test method will not complete. So, partially embedded single reinforcing particle like here particle is fibre out of the block of matrix material this portion, ok.

Pull out leads to debonding first it is debonding is taking place, then the propagation of debonding front propagation will be there, then once the debonding and propagation is completed then frictional sliding will be there. This test is actually replicable when the strength of the reinforcing material is very high, higher than the bonding strength.

So, the drawbacks are the interfaces in the specimen may differ from those in real material, because here in the specimen which is created using a single fibre; the interfaces which is created here it may be entirely different from actual composite material because, this is due to the absence of neighbouring fibres. Here there is no neighbouring fibres but in composite material we have other fibres which interfere with the pulling out force, and difficult to carry out specially for the thin and brittle fibre. So, for very thin fibre the strength will be less, so for those fibres it is very difficult to carry out and also for brittle fibre the fibre may be ease before we start the test.

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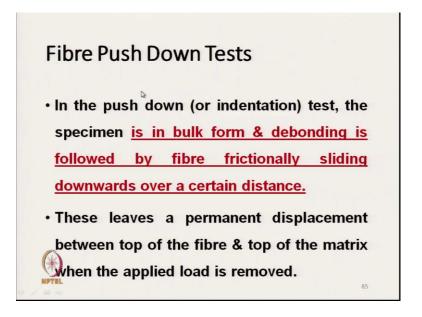


Next method single fibre push out test. What is that? Here the method is here, this is the composite and we use a single fibre here, and the fibre should be thick enough. After this the composite a slice is made, that is the this is the composite slice very thin slice of composite is there, and here we have a single fibre is a single fibre.

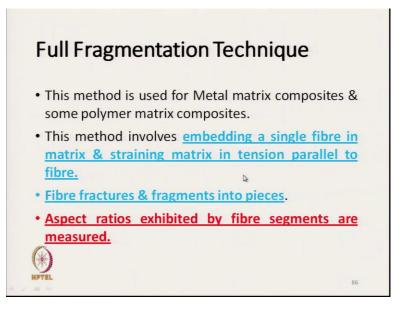
And in this case the fibre single fibre is pushed out by applying some force the fibre will

be pushed out, and this force is being measured. That is in push out test the specimen in the form of thin slice with fibre axis normal to the plane of the slice. So, this is the plane here and here the force this is the single fibre, single fibre and this is the slice thickness, slice thickness and fibre is being pushed out of the place. The fibre becomes displaced, so that it protrude from the bottom of the specimen from other side it will come out. This test is easy with large diameter fibre but for fine fibres it is difficult. So, the thickness of the fibre should be sufficient otherwise it is very difficult to push out. So, this shows again debonding propagation and frictional slide.

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Third method is fibre push down test. Here it is not the single fibre here bulk fibre. So, in the push down test the specimen is in bulk form and debonding is followed by the fibre frictionally sliding down by over the certain distance. The certain distance it will slide down and we can calculate the debonding force. This leaves a permanent displacement between the top of the fibre and the top of the matrix, when the applied load is removed. So, there will be permanent deformation.

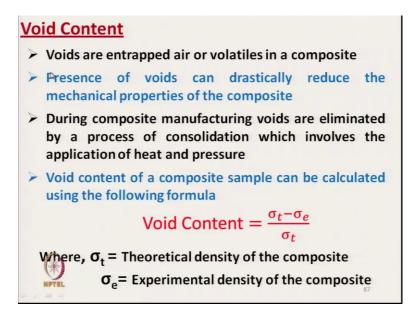


Next technique is that it is a full fragmentation technique. This method is used for metal matrix composite and some polymer matrix composite also. This method involves embedding a single fibre in matrix and straining the matrix in tension parallel to the fibre. Now, this is matrix and here we have fibre matrix. Now, this matrix is being strained and in case the extensibility of matrix is higher than the reinforcing material this is reinforcing material, there will be fragmentation of the reinforcing material.

Now, this matrix is being strained to this dimension and the reinforcing material has got fragmented and by studying the fragmentation and aspect ratio we can actually calculate the debonding force. So, by applying the tension parallel to the fibre, the fibre fractures and fragmented into the pieces at different pieces it will be fragmented, and aspect ratio exhibited by fibre segments are measured, ok. So, these are the methods for measurement of debonding force,

Next important characteristics of any composite material is void content, because the amount of void content in the composite material affect its mechanical characteristics significantly.

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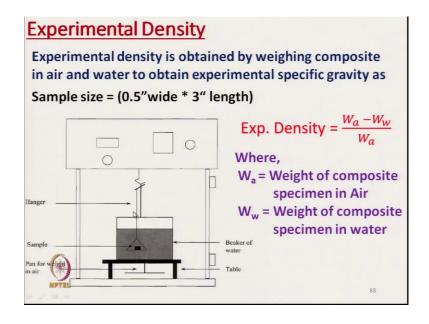
So, voids are entrapped here in the composite, ok. It may be entrapped here or any volatile material. Presence of void can drastically reduce the mechanical properties of the composite materials. During composite manufacturing voids are eliminated by a process of consolidation which involves application of heat and pressure. So, by application of heat and pressure we can eliminate the void from the composite material. So, void content of a composite sample can be calculated using the formula



Where, σ_t = Theoretical density of the composite

 σ_e = Experimental density of the composite

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The experimental density we can calculate by taking the mass of composite in air and the same specimen in water. So, this method can be used only if the density of the composite is more than the water that is more than one if it is there, then we can use this technique. It is expected that is the experimental density is

Exp. Density =
$$\frac{W_a - W_w}{W_a}$$

Where,

W_a = Weight of composite specimen in Air

W_w = Weight of composite specimen in water

So, here we measure the weight in water as well as in air.

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Theoretical Density It can be determined as follows Theoretical Density = $(1 - V_f)\sigma_m + V_f\sigma_f$ Where, V_f = Fibre volume fraction in the composite σ_m = Density of Matrix σ_f = Density of Fibre Fibre Volume fraction in the composite can be measured by i) Image analysis technique ii) Matrix burn off technique ii) Liquid digestion In liquid digestion method, matrix is dissolved using solvent and fibre volume fraction is calculated as Fibre vol. Fraction = $\frac{W_f}{w_c/\sigma_c}$ W_f = Weight of fibre
 W_c = Weight of Composite
 σ_c = Density of composite

And theoretical density we can calculate by knowing the fibre volume fraction So, V_f is the fibre volume fraction and density is σm is the density, this is the density of fibre. So, using this formula

Theoretical Density = $(1 - Vf)\sigma_m + Vf\sigma_f$

we can calculate the theoretical density of the composite material and fibre volume fraction in the composite can be measured by. So, we have to calculate also we have to measure actual fibre volume fraction of fibre V_f, it is done by using image processing technique or matrix burn off technique or liquid digestion technique. So, matrix can be removed by using the burn off method or liquid digestion method.

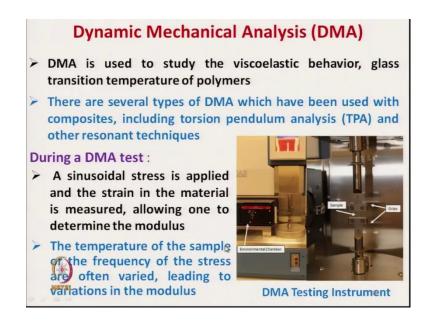
So, we can get the actual volume of fibre and the fibre volume fraction is

Fibre vol. Fraction = $\frac{W_f}{w_c}$

W_f = Weight of fibre W_c = Weight of Composite σ_c = Density of composite

So, total volume of fibrous material divided by total volume of composite material.

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So, after measuring the void content next important characteristics is the dynamic mechanical analysis. This is important for composite because composite during its application is subjected to various dynamic mechanical load and at different temperature different conditions are there. So, dynamic mechanical analysis is used to study the viscoelastic behaviour glass transition temperature of the polymer.

So, most of the plastic polymer that is fibre reinforced polymers are viscoelastic in nature. So, this viscoelastic behaviour is actually assessed using DMA method. There are several types of DMA which have been used with composites including torsion pendulum analysis and other resonant techniques, ok.

During a dynamic mechanical analysis test, a sinusoidal stress is applied the here that is

this is the grip, and in between the grip there will be a specimen here this is a specimen here and sinusoidal stress is applied, and the strain in the material is measured allowing one to determine the modulus. So, we can determine the modulus. The temperature of the specimen or the frequency of test is changed leading to variation in modulus. So, we change the temperature and frequency of stress and we measure the change in modulus also. So, here the test is carried out for storage modulus and for loss modulus.

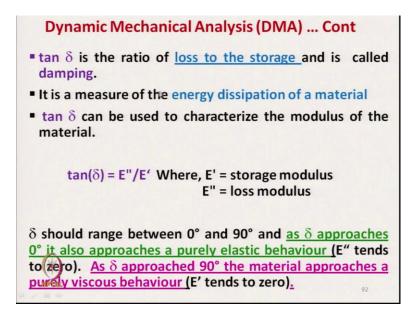
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Dynamic Mechanical Analysis (DMA) Cont
 DMA measures stiffness and damping, these are reported as moduⁱus and tan δ.
 Sinusoidal stress is applied, modulus can be expresed as
 In-phase component, the storage modulus (E') Out-of-phase component, the loss modulus (E")
 Storage modulus (E') is a measure of elastic response of a material. It measures the stored energy
• Loss modulus (E") is a measure of viscous response of a material. It measures the energy dissipated as heat.
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So, DMA measures stiffness which is actually in phase component that is storage modulus. Stiffness is nothing but the elastic component of the, and the damping also which is viscous component. So, by this method we can measure stiffness and damping these are reported as modulus and tan δ . The sinusoidal stress is applied modulus can be expressed in terms of in phase component that is storage modulus **E**' and out of phase component which is loss modulus **E**''.

The storage modulus is a measure of elastic response of the material it measures the storage energy, ok. And the loss modulus it measures the dissipated heat during testing which is viscous response of the material.

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Tan δ is the ratio of loss to storage, and is called the damping; so, loss modulus and storage modulus if we take the ratio, we can get the damping value. It is a measure of energy dissipation of a material. Tan δ can be used to characterise the modulus of the material and its expressed by

 $\tan(\delta) = E''/E'$

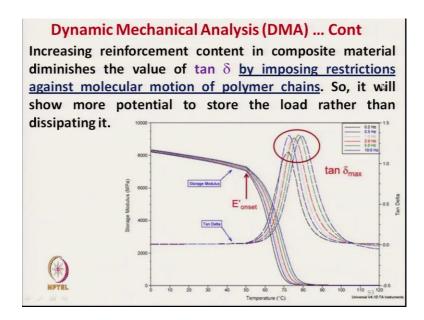
Where, E'=storage modulus, E'' = loss modulus

So, where **E**'' is loss modulus and **E**' is storage modulus, and δ should range between 0 to 90 degree, that this tan δ this angle should be between 0 to 90 degree as δ approaches 0.

That means, in that case it is also approach is the pure elastic behaviour. So, as it is 0 that means, it will be pure elastic behaviour where E double dash tends to 0. So, E''tends to 0 means it will become 0 this part. So, tan δ become 0. So, that shows the it is a pure elastic behaviour. And as the δ approaches 90 degree it will actually approach to towards the pure viscous behaviour.

So, from tan δ value we can actually get the idea about the material behaviour whether it is a pure elastic material or pure viscous material. So, for thermoplastic composite if we increase the temperature. So, it will gradually from the it will transmitted from the elastic behaviour to the viscous behaviour.

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So, this diagram shows in x axis we increase the temperature and y axis is the storage modulus. So, increasing the reinforcement content in the composite material diminishes the value of tan δ , by imposing the restriction against molecular motion of the polymer chain. That means, if we incorporate the reinforcing material the composite will become elastic, so it will reduce the tan δ value. But if we increase the temperature this picture it shows if we increase the temperature at lower temperature the storage modulus was high, but once we increase the temperature the storage modulus suddenly drop after certain time the **E'** onset, so it starts dropping that means, it is becoming viscous.

On the other hand, if we increase the temperature the damping behaviour a tan δ is increasing and it is reaching to the maximum value. So, by imposing the restriction once we add the reinforcing material increase the content of reinforcing material, so by imposing the restriction against the molecular motion of the polymer chain it will show more potential to store the load rather it is dissipating. So, it will actually allow the composite material to store the load, otherwise in absence of reinforcing material it will try to lose the all this load value, ok.

Now, after all this test which we have discussed those are destructive in nature that means, we test the composite which will not be reused but in most of the applications, the composite materials are applied in structural material. If we want to use the test methods which is destructive in nature we cannot use for any structural material. But during the

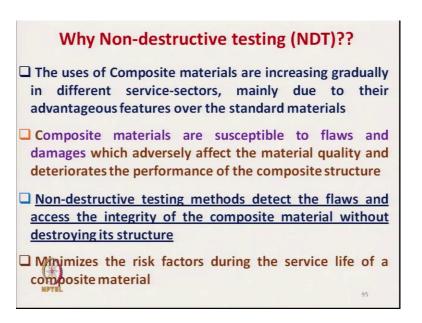
applications of all this composite in different structure if we want to use we have to select some non-destructive test of composite materials.

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So, there are different non-destructive methods which will actually help us to understand the characteristics of composite materials. So, why do we need to test the composite in non-destructive mode?

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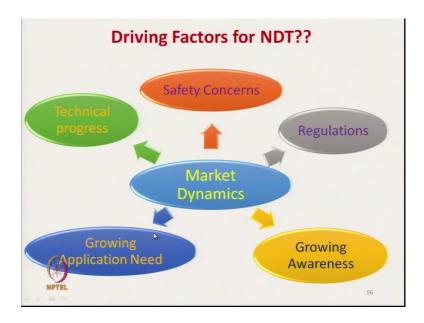


So, the use of composite materials are increasing gradually in different service sectors mainly due to their advantageous features over the standard materials. Composite materials

are susceptible to flaws and damages which adversely affect the material quality and deteriorates the performance of the composite structure. Non-destructive testing methods detect the flaws and access the integrity of the composite material without destroying the structure, which is extremely important.

So, we have to assess the integrity of the material or integrity of the structure without destroying the structure. So, we can intermittently test the performance of the structure by non-destructive methods. And it minimises the risk factors during service life of a composite material. So, during its service life of aircraft structure aircraft body if we want to test whether there is any crack or any failure is there, so we can use non-destructive test method of the total structure. Here we cannot use the destructive test method.

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So, the driving force is there the safety norms. So, there are different safety norms are there. Suppose a particular structure we have to understand whether there is any internal damage took place or not, in the structure which is already built. So, there is the norms, we have to test we have to understand the characteristics of the material during test. So, due to the safety norms we have to test the material but if it is destructive in nature we cannot test. So, non-destructive testing is required.

The regulations are there, government regulations are there, its growing awareness of the safety of the structure is there. So, we must test the material, test the structure intermittently, growing application need, technical progress is that so we can test the

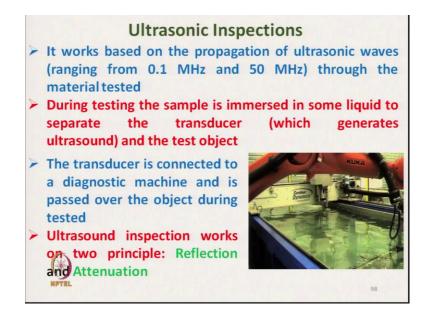
material without destroying the structure. So, due to the technical progress; so, these are the market dynamics.

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And different test technologies are there. The main technologies are radiography, thermography, shearography, ultrasonic inspection. So, ultrasonic inspection is there where we use ultrasonic sound wave and assess the characteristics. So, I will discuss all these methods one by one.

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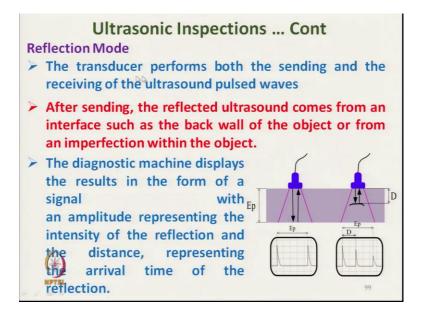
First is ultrasonic inspection. So, this method works based on the propagation of ultrasonic

wave which is ranging from 0.1 to 50 megahertz that with that frequency through the material tested. So, the ultrasonic wave is propagated through the material.

During testing the sample is immersed in some liquid to separate the transducer and the test object. So, that transducer is there which will generate the ultrasound and test specimen should be actually separated using some liquid. So, some liquid has to be applied on the surface of the test material. The transducer which generates the ultrasound is connected to a diagnostic machine and is passed over the object during test. So, the transducer is passed over the object the ultrasound inspection works on two principles one is reflection mode, another is attenuation mode.

So, once in it works on reflection mode the ultrasound is actually it is falling on the material and due to presence of some defects it will get reflected and that reflection is measured. And in attenuation the ultrasound is passed through the material and from other side the recording is taking place.

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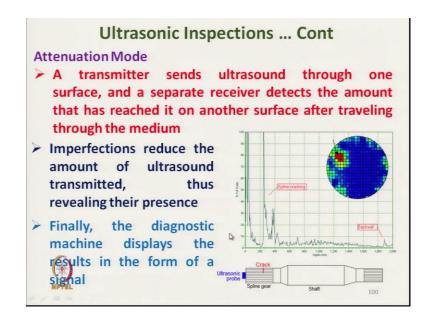


So, once it is working in reflection mode the transducer, transducer performs both the sending and receiving of the ultrasound pulse wave. So, there are transducer, this is the transducer, once it is in work in the reflection mode this transducer actually perform both the it is sending and receiving function. So, this sensor it is sending the ultrasound and it is receiving the ultrasound. And the time here it is measuring the peaks, ok. This portion it is showing there is no defect. So, if the ultrasound is passing through this composite, this

portion composite material and from other surface it is getting reflected and the peaks even peaks are shown here.

But once there are some defects or some cracks present inside the composite material the ultrasound before its reaching to the other surface it is getting reflected from that portion from the different zone, and its showing peaks in different form. So, by analysing this graphs one can locate one can actually identify the defects present inside the structure. The diagnostic machine display the result in the form of signal which is with the an amplitude representing the intensity of the reflection and the distance representing the arrival time of the reflection. So, from there we can actually identify the defects.

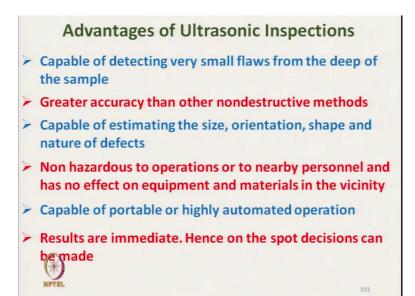
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Once the ultrasound system works in attenuation mode, a transmitter sends the ultrasound through one surface and the separator actually that there will be separate receiver which will detect the amount of sound, amount of the wave which will reach to the other surface. Depending on the defect present, in the structure the amount of wave transmitting through the composite will change the imperfections reduces the amount of ultrasound transmitted thus revealing their presence. So, any defect present inside the structure can be identified.

Finally, the diagnostic machine displays the results in the form of signal. So, we can get the signal. So, it works on reflection mode and attenuation mode.

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And the advantages of ultrasound is the ultrasonic inspection principle is that it is capable of detecting very small flaws from the deep of the sample. So, in case of there is a very very small crack inside the composite material it can detect greater accuracy than other non-destructive method. Capable of estimating the size orientation shape and nature of the defect; so, for any source of defect whether it is a void or crack something else it can detect.

Non-hazardous, because it is a only ultrasound is created. So, it does not affect the human body to the operator or to nearby personnel and has no effect on equipment and material in the vicinity. Capable of portable or highly automated operation; so, this can be actually transmitted the total instrument can be taken to the structure and tested. Results are immediate we can get the result immediately. Hence on the spot decision can be taken. So, we can take decision on the spot because we get the result immediately.

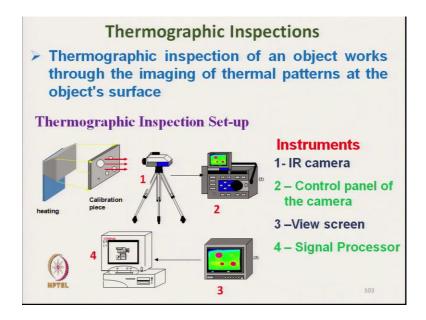
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Main disadvantages of the ultrasonic inspections are its expensive and the extensive technical knowledge is required for the development of inspection process. So, for that it is becoming expensive; thin, rough, irregular shaped sample are difficult to be inspected what does it mean. So, rough sample, irregular sample, because here we need some liquid to be applied on the surface. If the surface is rough then the application of the liquid is difficult. Surface must be prepared by cleaning and removing loose scales and paints.

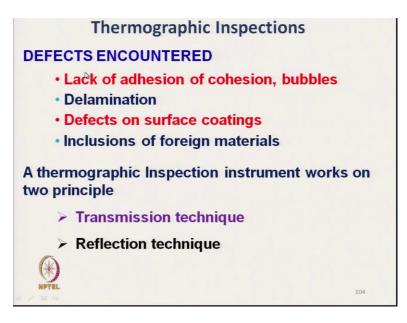
So, once we try to test or try to apply this technique in the existing big structure where it is painted or something is there in the surface it is very difficult to use. Liquid media are needed to provide effective transfer of ultrasonic wave energy between the transducer and parts being inspected. So, we need to apply liquid medium that is disadvantage. It is very difficult to apply the liquid medium in some application where when it is vertically oriented or some crack orientation in those applications it is difficult. Inspected items must not be affected by the liquid medium. So, the liquid medium which we use it should not get affected by this liquid, it should not affect the item the composite material, this liquid should not react with the matrix or the reinforcing material.

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So, next technique is that thermographic inspection technique. Here in this technique the thermographic inspection of an object works through the imaging of thermal pattern at the objects surface. So, from other surface of the material we can actually get the thermal image. So, here it consists of IR camera which takes the image, control panel of the camera to; so, this is the control panel we can control the camera, view screen and signal processor. These are the components.

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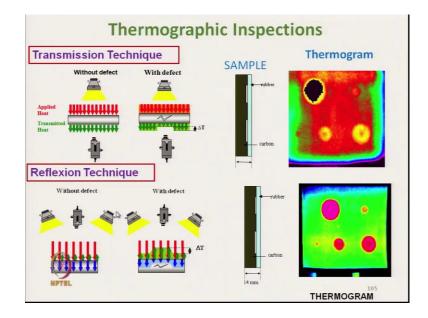


The defects encountered here, lack of adhesion of cohesion or bubbles; so, this type of

defects. So, bubble present or lack of adhesion, we can measure using thermographic inspection, delamination we can measure, defects on surface coating, inclusion of foreign material in the composite material we can get by using thermographic inspection.

A thermographic inspection instrument works on two principles one is transmission technique, another is reflection technique.

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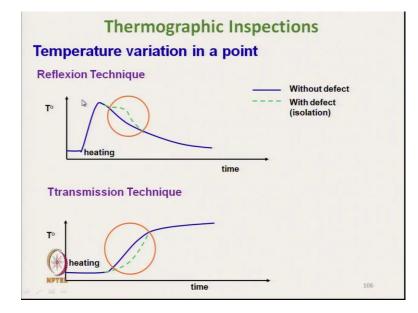


So, this is the technique for transmission mode where this is applied heat is applied, ok, and once the heat is applied the heat will be transmitted evenly throughout the sample. And this sample in the left side it shows the sample without any defect. And, once the heat is applied, this is the heat source heat is applied with the sample with defect here due to the presence of defect like crack or any other thing the heat transmitted in other side will be non-uniform. And by using the thermal imaging camera, we can image we can take image the temperature distribution on the other side. And here this is showing the image on the other side and this shows the defect present in the composite.

Once this method works in the reflection mode here the heat source and the camera is in the same direction. In left side it showing the specimen without any defect that means, the reflection of heat is uniform, but on the other hand with defect as there is a defect present here the heat reflected will be totally different in the defective zone than in normal zone.

So, in case of transmission mode the heat transmitted in the defective zone will be less

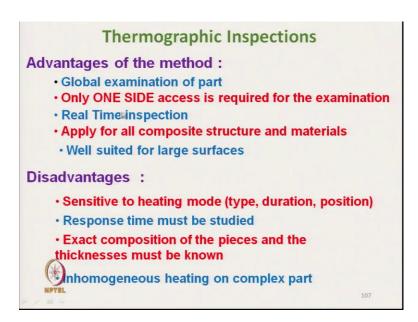
than in the normal zone, but on the other hand in case of reflection mode the heat transmitted heat reflected in the defective zone is more than the normal zone.



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This is shown by these graphs, here it is the time and it is a temperature. In case of reflection mode the temperature at the defective zone is higher than the normal portion whereas, in case of transmission mode this is lower, heat in the temperature in the defective zone is lower. So, by analysing this graphs and the picture we can get idea about the defects present inside the composite structure.

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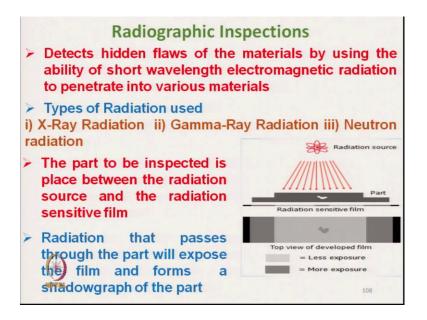


So, the advantages of the method is that, it is a global examination of parts. Only one side access is required for the examination, that is the heat is applied from one side and if it is the reflection mode is used only one side access is enough. Real time inspection; apply for all composite structure and material, so we do not need to apply any liquid medium here. Well suited for large surface for large surface we can use, but main disadvantages of this technique is its sensitive to heating mode. So, if we change the mode of heating that is type of heating, duration of heating or position of heating the result will change.

Suppose we change the distance of heat source from the composite material, the heat transmitted on the other side or heat reflected will be entirely different. The response time must be studied, ok. And exact composition of the piece and the thickness must be known because if it is the thermoplastic composite and the melting point is low that will affect the composite. In homogeneous heating on complex part, that is the main drawback of this technique because in case of complex part the heating will not be uniform.

Let us see, suppose this is a composite which is straightway uniform. So, once it is heated, the heating will be uniform throughout the surface and the heat transmission will be uniform. Suppose there is no defect perfect composite, another composite of say complex structure this is the composite material without any defect this is the composite material. Now, if we apply heat here. So, heat transmission will not be uniform which is very difficult to assess, whether this difference in temperature on the other side is due to the structure or presence of any defect inside the structure. So, that is why it is very difficult to assess the composite defect for inhomogeneous complex material. So, there will be inhomogeneous heating on the complex part of the composite.

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And next technique is the radiographic inspection technique. So, this technique we will discuss in the next class. So, till then thank you.

Thank you for patient hearing.