Carbon Materials and Manufacturing

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Lecture - 37

Mechanical Property Testing Methods for Carbon Fibers

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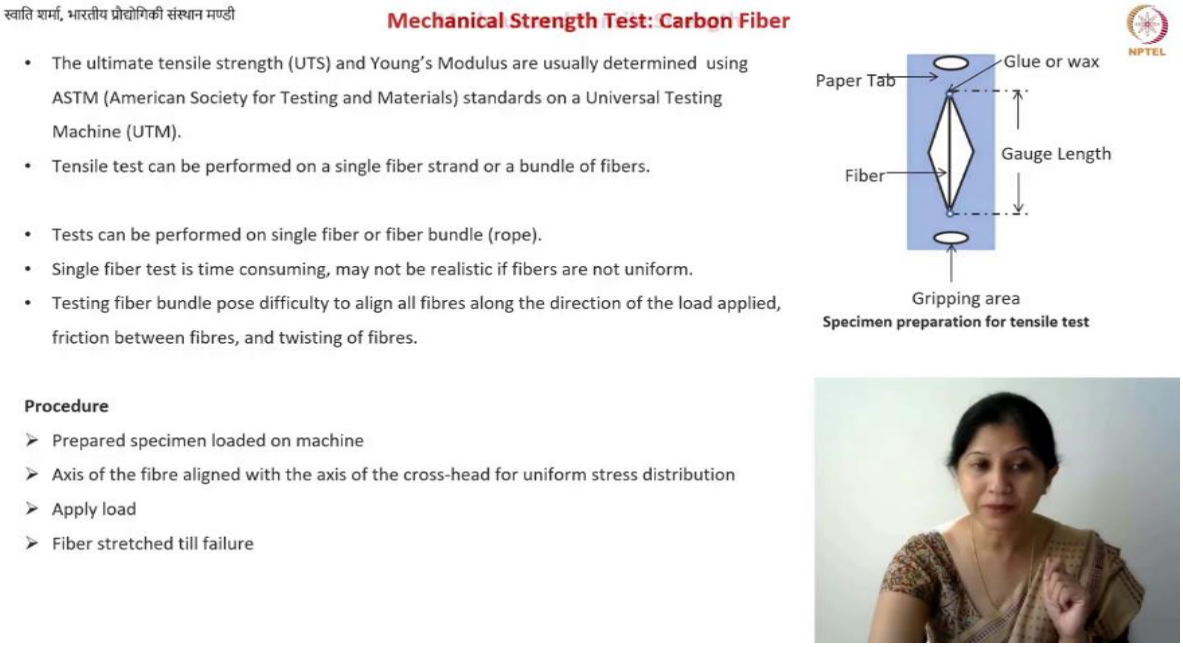

Hello everyone. In this lecture, we are going to discuss how to test Mechanical Properties of Carbon Fibers when we fabricate them. You know that mechanical properties are very important to us if you want to decide on the applications of certain carbon fibers or if you just want to purchase them. What there are two things that you will look for? Number 1 is Young's modulus, which is the slope of the stress-strain line in the elastic region and number 2 is the ultimate tensile strength this is the load at which your fibers will break.

If I fabricate let us say a new set of carbon fibers; I tried out a new polymer or I tried out some other special heat treatment conditions and now I want to see how different are these fibers. Are they better than the commercial fibers or not or any other information that I want to have for these fibers. I need to perform these two tests to find out the Young's modulus and to find out the ultimate tensile strength. So, how do we do that at large scale?

These tests are relatively simple. In fact, those who have done their bachelor's in mechanical engineering may have already performed these kind of tests in the lab. You will typically take let us say one mild steel rod, and then you will use a setup known as the Universal Testing Machine or UTM that is the common name, but there are also related instruments and there are several in house instruments.

So, the name is not that important. The idea is that this is a setup where you basically clamp your rod or fiber and pull it, and basically your machine will actually measure the load at which it breaks and will also measure at which point did it break; so the mechanical behavior. Also, it will measure the stress strain curve.

You will see that this is the slope then you can measure it later on. But what are these equipment doing and how do you actually calculate these properties and just get the data from the machine? That is what we are going to learn.

As I said that you will use the similar kind of setup that you would also use for a large-scale material, then what is the difference? Well, in the case of fibers our sample is much smaller. Also, if you are performing the test for a single fiber then the loads are much smaller compared to what you would use for a thick steel cylinder or rod. So, that is one difference.

The loading of the sample also becomes slightly different because you may not have the same kind of sample holder. So, in the case of steel rod you will just have two clamps and then you will tightly clamp your sample or specimen. But, in the case of carbon fibers, you may have certain chip-like structures in smaller sample holders where you can glue your fiber and then you can load this entire chip into your UTM.

So, these are some fundamental differences. But as such the calculations remain pretty much the same. So, the chip that I was talking about it, looks something like this. This is just a schematic again. You have a straight fiber, that is clamped on both sides. And how do you do that? In this particular case, you will glue it, you will wax it and then let it dry for a while.

You can also use epoxy raisins. So, this is what you are going to do. And you grip your fiber, you have it under complete tension inside your chip. Now, you also see something known as the gauge length. Gauge length is basically the length that is actually being measured between your clamps. This is the actual length of the fiber that is being tested. And then other things like gripping area. And this tab or chip, it may be made of plastic, it may be made of paper also. So, this is the first thing that you will have. Now, you will load this entire thing into your

UTM. You will typically have specific slots for such tabs and you can also buy these tabs or chips specific to your setup.

Now, one interesting and the most important question for us is that do we use single fiber or bundles of fiber? So, there are pros and cons of both. It depends also on what is the scale at which you want to have your application, do you want to use the bundle of fiber for your application, if you are doing any manufacturing application you want to make composite materials out of it or you already have some resin when you were making the bundle.

In that case, you will rather use the bundle because that is sort of more realistic. But if you want to test the mechanical property of single fiber for whatever application you have, in that case, you will use the single fiber. So, the bundle is basically this rope that I am talking about.

Single fiber tests can be time-consuming. And there is one more thing, your fibers must be uniform if you are testing single fibers.in that case most of your fibers the diameter range of your fiber should be pretty much uniform. So, this will often happen when you have mesophase pitch-based fibers.

in that case what you using melt spinning technique, and melt spinning give you thicker fibers, but they are more uniform because they are coming out of the spinneret and the capillaries and your spinneret are all of the same diameters. So, you pretty much get your fibers which are also more or less of same diameter.

But, that may not happen in the case of electrospinning because using the standard electrospinning technique, you end up getting this range of diameters in your fiber. So, if you test a single fiber then in that case you need to be very careful that the fiber has the average diameter. Whatever is your range of diameters can be very easily calculated using the scanning electron microscope and even there are other techniques that I will also discuss briefly.

Nowadays there are so many image processing software available, many of them are also free. They give you information about what is the average diameter of fibers in your sample. So, in that case, if you want to test a single fiber then you should take the one that falls in this average diameter range,

In general, if you want to utilize single fibers for a certain application, definitely you can perform the test on a single fiber. However, you need to know how realistic it is in terms of

your application, Now, if you test the bundles of fibers. It is more realistic. This is how you are going to use them for your manufacturing applications.

But there are also issues with that. For example, the fibers may get entangled, there may be twisting of fibers. So, you may not have all fibers. You need to have them in tension. So, you need to have them straight. But that might not be the case. Some fibers may get entangled and hence you may not be able to get the right values.

There may be also certain friction between the fibers. You can get rid of the friction by having some resin or some polymer between them, some sort of lubricant. But, if you do not want to have the lubricant and you want to test the properties of your virgin fibers, in that case, you may also having end up having some friction between the fibers.

So, these are some of the problems with the bundles, but then you need to choose what is important for you. So, now we come to the procedure. How do we do that? So, as I already you this chip. You prepare your fiber specimen and you load it onto the chip and that the chip is loaded onto the machine on your UTM.

And now you need to align the axis of your fiber with the crosshead. So, again these are certain operating conditions of the machine. I am not going into the details and every machine may have a different set of operations, and how do you align it, and what kind of crosshead you have and so on.

But you basically need to align your fibers with the axis of your instrument. Then, you apply the load and apply the load till your fiber breaks. So, apply load till fracture. What do you do afterward? Now, how do you calculate the values from here?

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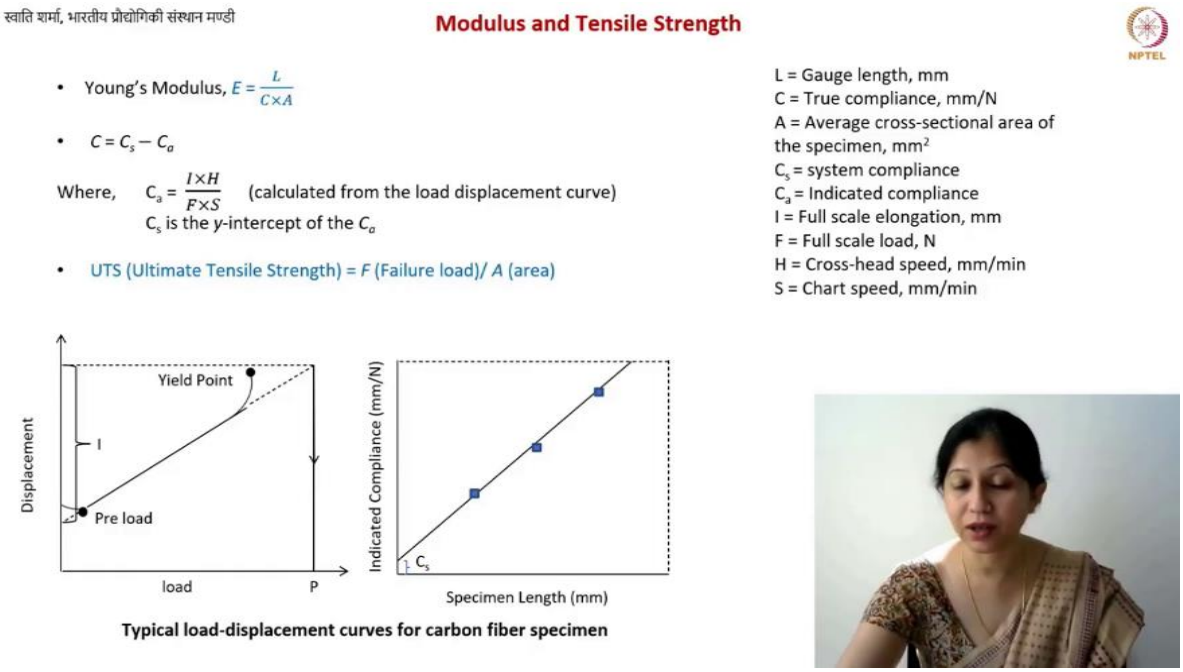

Here is the typical load-displacement curve that you will get for your carbon fiber specimen. So, you see in this load-displacement the first curve for example, you will have an elastic region; this is also interesting that in the case of carbon fibers you will typically get an elastic behavior, before they break.

But towards the higher end, you may have certain strain hardening. So, you may see certain strain hardening because the carbon chains realign themselves. This is always the case, you get strain hardening in the case of visco-elastic polymers as well that we had learned before.

Carbon fibers are not like polymer fibers. They do not have the same kind of geometry or microstructure. However, they do have these long sheet and chain-like structures. So, they do tend to align themselves at some point, and therefore, you may get a little bit of strain hardening. But even that is not very significant. Typically, you will an elastic sort of behavior.

as you increase the load, the displacement will also increase, and at some yield point, your fiber will not behave elastically anymore and soon after that you will have the fracture. Unlike the visco-elastic polymer where you have a larger region of strain hardening, that is not the case with carbon fibers and if at all you, see it is not going to be very significant.

So, these are the kind of curves that you will get from your machine. And here I have mentioned a few things, a few terms that I will be using for showing you how do you calculate Young's modulus from these curves? Your Young's modulus is

$$E = \frac{L}{C \times A}$$

Now, A is the area of your specimen. Make sure that it is in mm^2. Actually, make sure that all the units are in same scale as units are always a problem when we do these kind of calculations, right. So, A is your area and L is the length of your fiber that is being measured. So, you can call it the gauge length. Now, what is this term C? C is calculated now by these graphs, by these plots.

$$C = C_s - C_a$$

C_a is the indicated compliance. As you can see here I have written it, indicated compliance that you can see in your second plot. It calculated by the expression

$$C_a = \frac{I \times H}{F \times S}$$

And what are these terms again? You can read in this glossary. Your I is the full scale elongation. H is the cross-head speed. These are very instrument specific parameters, but I am just trying to show you, how it is calculated. F is your full scale load and S is the what is known as the chart speed. So based on these things then the C_a value is calculated and C_s is nothing but the y intercept in the second plot that you see indicated compliance.

There you see, there is a certain intercept after which you start getting your values. You need to factor in factor that in; so, that you need to subtract. And this is how you will get the value of C which is $C_s - C_a$, and now you insert all the values in your Young's modulus expression and that is how you will get the value.

Now, the second important property is the ultimate tensile strength. This is very easy to calculate because, you will always know what is the load at, which your fiber breaks. And just that load divided by the cross sectional area of your fiber is what will give you the ultimate tensile strength of the fiber.

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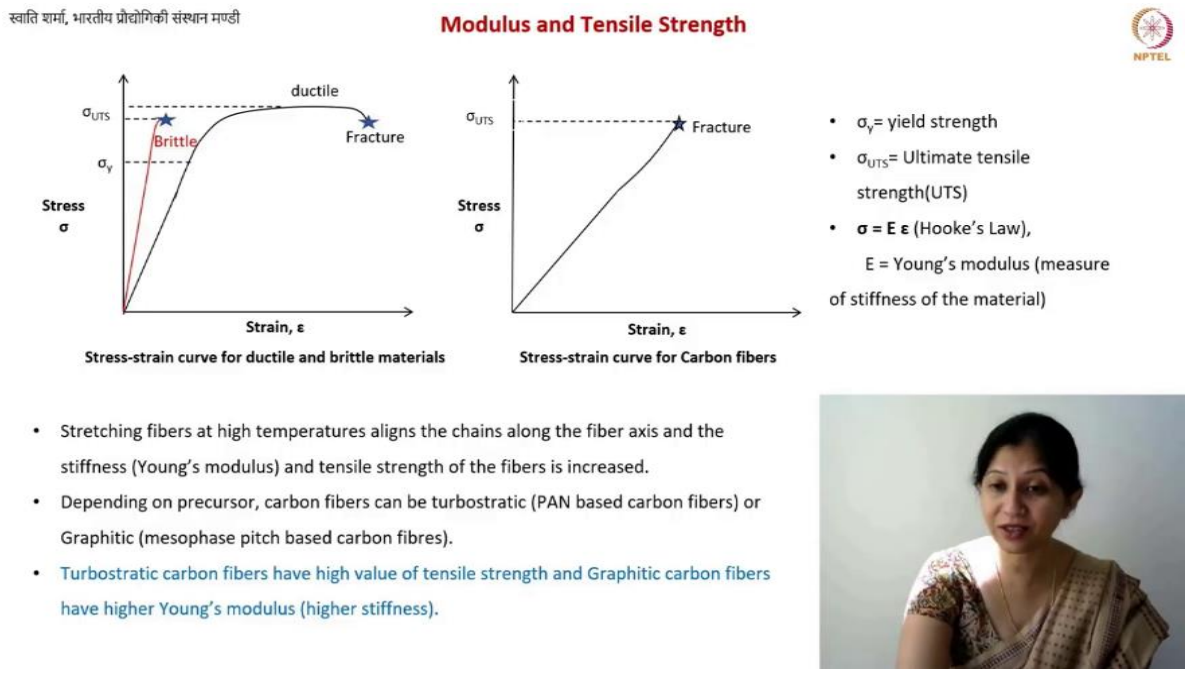

Now, there are some more curves. Again, we are still on the same topic of the modulus. Here in the second curve, you can see that the linearity is sort of compromised a little bit. That is because of the strain hardening as I mentioned.

What is now important for us is that different types of carbon fibers have slightly different behavior. Why? Because they have different crystallinity. So, if you take PAN-based fibers, you typically have turbostratic carbons. what is turbostratic again? You have the carbon sheets, but they are still randomly oriented on top of each other, they are not graphite-like crystallite, they do not have AB AB A type arrangement.

However, when you get your fibers from mesophase pitch then you have the more graphitic. And that means that at least you have some regions of AB AB A type crystal structure.in that case you can imagine that you have a higher Young's modulus for these kinds of fibers, for mesophase pitch or graphitic type fibers. Why? Because you have you have nicely aligned sheets.

They behave more elastic. If you give them certain strain they may change their position slightly, but then they will try to come back because the molecular attractive forces are pretty strong, you have a good crystal structure. So, the crystal structure always tries to come back to its normal position.

And hence you here will see it has good elasticity, higher value of elastic modulus, that you see when you have graphite-like crystallites. But when you have turbostratic sheets, these are randomly oriented sheets; randomly oriented sheets mean they may also contain some non-6 membered rings.

That also means that if you distort them, they may not be able to come back to their normal position. However, at some point, if you keep on distorting them maybe they will find some new normal position or they will try to realign themselves. That is what we call strain hardening.

So, this is sort of the mechanism and this is the reason why you get better elasticity or better modulus in the case of graphitic fibers. Again, it depends on your application, what is it that you want? For many applications for many manufacturing applications actually you want a good modulus. But biomedical applications would require a good ultimate tensile strength, for example, if you are making bone implants, you might want your structure or your bone-implant not to be too stiff because you are not applying too much load there. What is more important there, is flexibility. What you want is that you should be able to press the overall structure a little bit.

So, here then I have written a summary that some carbon fibers especially PAN-derived carbon fibers have more of a turbostratic arrangement of sheets, they have a higher value of tensile strength. While the graphitic carbon fibers or graphite carbon fibers, those derived from mesophase pitch will have a higher stiffness or higher Young's modulus.

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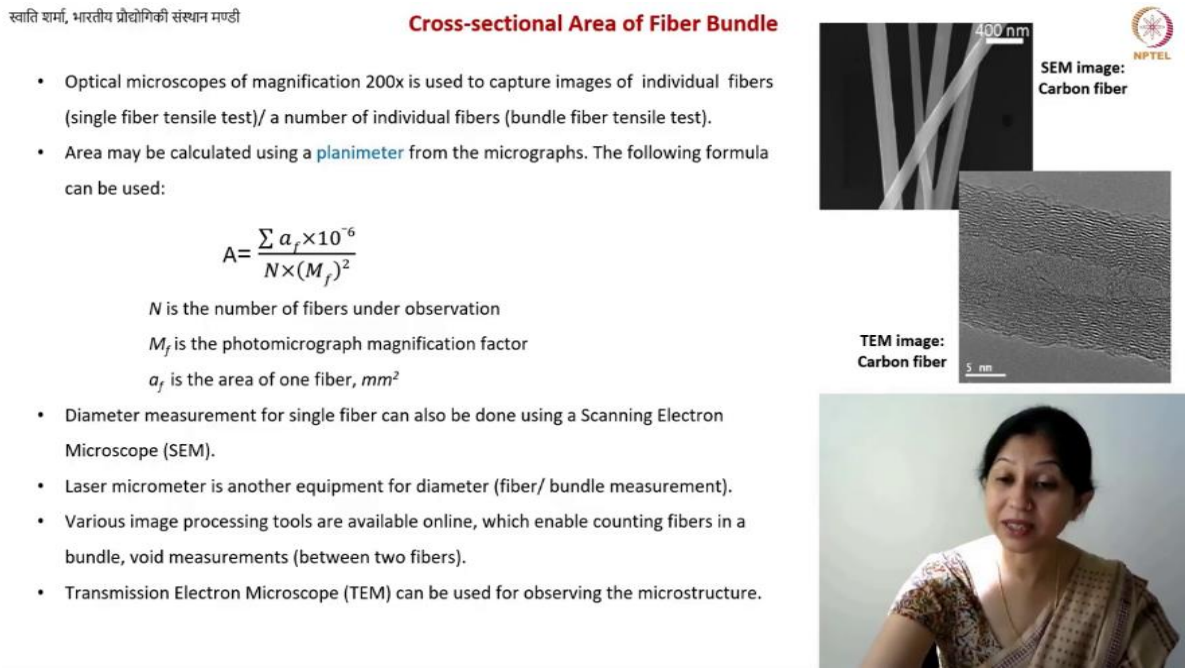

For all these calculations, you need is the cross-sectional area of your carbon fibers. If you have a single carbon fiber, and that is thin enough and it is easy to measure just by using scanning electron microscopy. You can just take a picture of that.

However, if you are using a fiber bundle, and that bundle may not have a perfectly circular diameter, and it is probably in the millimeter length scale. So, in that case, you may need to use some other techniques because it may be beyond the field of view of your scanning electron microscope.

In that case, what do we do? We use an optical microscope first of all for taking a basic image. So, you will for example take 200 times magnification and then you will capture an image. Some of the UTM's are also equipped with the microscope and you use this formula to calculate your cross-sectional area.

So, now, what you see the important thing here is that af the first term that is just the area of a single fiber. What is important here is you are dividing this entire thing by the magnification factor. So, how many times does it? Which is 200 in this case. And N is the number of fibers that are under observation. So, this is pretty much for a bundle for a relatively sort of thick bundle of carbon fiber.

So, this is how you calculate your cross-sectional area. Again, you should look for the units and have the right units. Now, the diameter measurement as I mentioned for the single fiber can be done using SEM. So, you see this is an SEM image and scale bar is 400 nanometer. You

can see that these fibers are electro-spun fibers and you can already see that they are not really uniform.

So, some of them are let us say about 100 nanometer and some are 300 nanometer in terms of diameter. So, in that case, you will need to calculate the average diameter of your fibers. But you can use scanning electron microscope for this purpose. There is also something known as a laser micrometer, where you have a laser beam, and then you have the fiber.so you shining the laser on top of the fiber.

Now, let us say, this is your fiber and the laser shines from here, then the region where you have the fiber, you will sort of get the shadow of this fiber and that is how you will measure the diameter of the fiber. So, that is known as a laser micrometry that also you can perform for single fiber or a bundle of fibers again.

There are also many other methods which be used. Even laser micrometry can be used for, if there are any visible voids inside your fiber or there are gaps between two fibers as you can see in this SEM image, so you can also use that. If there are gaps between two fibers, there the laser will pass through that gap.

Of course, the laser wavelength has to be optimum so that you can get a good contrast in your image. But these are some of the techniques that you use what you also use. Yes, transmission electron microscope. So, here is an image of a carbon fiber.

But you do not use it just for diameter measurement. You will typically use transmission electrode microscope only when you want to see the microstructure of the fiber like how the chains are aligned, how does it look inside and for that purpose you also need to have very thin fibers. If any structure is thicker than 20 nanometer, then it is too thick for TEM. So, you will get just a black image and you will not be able to see much.

But in this particular case, you see that the fiber diameter is approximately 20 nanometer. And you can see how the chains are aligned. Actually, these are the chains that I call turbostratic.

So, you can see that these are these wavy chains, and if you actually measure the distance between these chains, then it is not it is higher than what you will get for AB AB A type graphite crystals. So, these are some of the methods for calculating various values for carbon fibers.

Again, this depends on your very specific equipment, specific measurement technique and specific type of carbon fibers whether you want to do it for single or for a bundle of fibers, you

can accordingly choose your parameters and you can accordingly choose your specimen as well.

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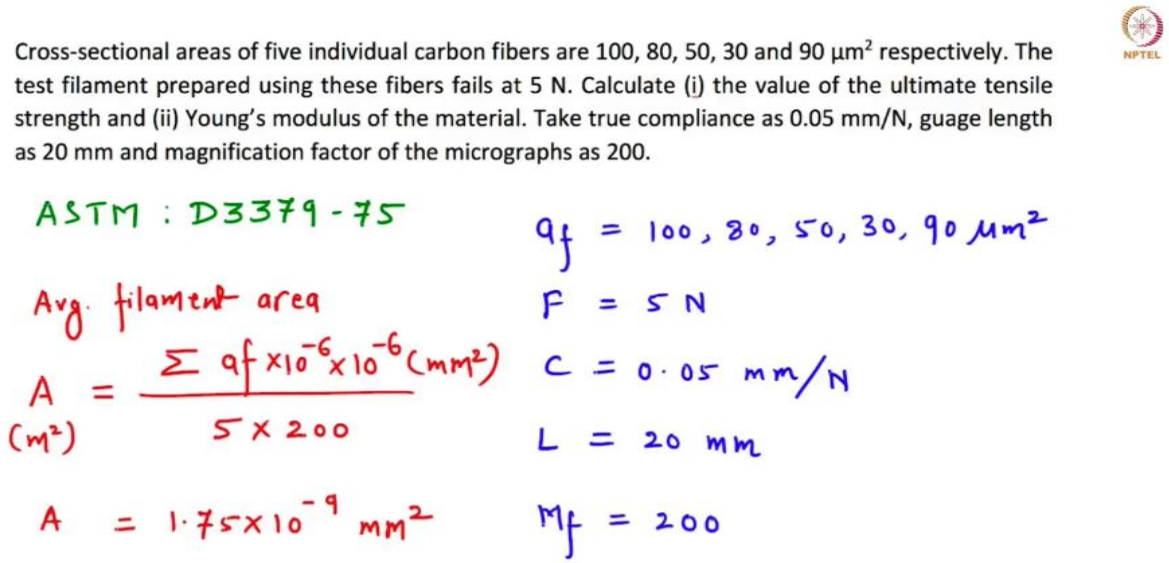

Now, we are going to solve one numerical problem. And after this hopefully it will become more clear to you whatever we have learned in the class. So, you know that we have the ASTM standard methods for mechanical property testing.

The one that we are going to use here, this is the number of that protocol D3379-75. So, you can also search for it yourself and then you will actually find more detailed analysis. So, in case, something is missing in this problem, then you can find it here.

So, what are the things now that are given to us? So, first of all we have these fiber fiber areas, so cross sectional areas, which are the values of af. So, we will see when we write down the formula. What do we have here? 100, 80, 50, 30 and 90 μm^2. By the way we have taken only 5 fibers here because this is just an example, but when you are actually doing the experiments then you should take a minimum of 10 fibers,.

So, this is one thing that is given to us. What else? We have the failure load i.e. 5 N, which we will require when we want to calculate the ultimate tensile strength. What else do we have?

We have the true compliance of the system. Now, true compliance C I am not calculating here, the value is already given to you, but you generally have to calculate it for your specific system. So, here the value is given to us, which is 0.05 mm/N. What else? The gauge length, gauge length is very important. We will write it as L. So, this is very important as well. So, generally you will take it between 20 and 30 millimeter, but also by changing the gauge length sometimes the final answers of the Young's modulus that you will get that will slightly vary.

So, this is also an important factor. What else is given to us? The magnification factor Mf of your micrograph, that is your 200. So, now, let us do the first calculation. We need to calculate the Young's modulus and also we need to calculate the ultimate tensile strength. In both cases, what do we need? We need the average filament area.

When I say average filament area this means this is the test filament; test filament means this is the bundle or the rope that you are testing. So, filament the here does not mean that this is an individual fiber. You can also do the testing for individual fibers, but slightly different methods are used for that. So, here we are talking about the filament which is actually a bundle.

So, this filament area or A, you also see the term af, that is what we are going to calculate and that is what is useful for all the calculations. What is the formula again?

We have the summation of all areas, but these areas individual fiber areas they are taken in millimeter square and what is given to us is in square micrometer.

So, we have to factor in some 10^{-6}. And then there is another 10^{-6} which is already there in your formula and that is because we get the area values finally in square meter.

What else is there in the formula? Now, here we need to enter the value of N, which is the total number of fibers. So, let me just write the total number of fibers, this is the N. So, here we have just taken 5 fibers and then the magnification factor.

$$A = \frac{\sum af \times 10^{-6} \times 10^{-6}}{F \times Mf}$$

So, now from here you will write down the values af. And then after all of this I have calculated the value of the area is, so it comes out to be 1.75×10^{-9} mm^2. So, I have already done the conversion from square meter to millimeter. So, these conversions are something that you have to be careful with. So, this is the value of the area. Now, we will do the rest of the calculations.

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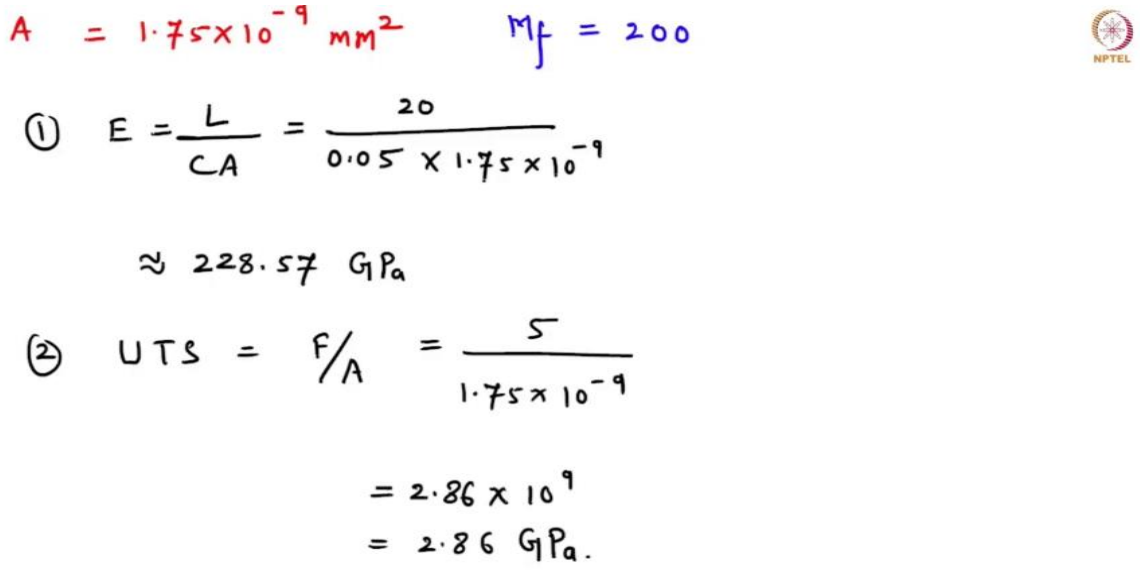

let us first calculate the Young's modulus. So, Young's modulus the formula equals

$$E = \frac{L}{C \times A} = \frac{520}{0.05 \times 1.75 \times 10^{-9}}$$

You remember L is your 20, it is in millimeter, and also in the formula we need it in millimeter, and then the value of true compliance also is given in mm/N which is the correct unit that we use in this formula. In case it is not given in the correct unit then you need to change it.

What else? The area A again 1.75 into 10 to the power minus 9 and here we take it in square millimeter. So the value of Young's modulus that I have calculated comes out to be approximately 228.57 GPa. So, this is your solution to the 1st part.

The 2nd part is the calculation of ultimate tensile strength, which is actually the easy one, all you need to do

$$UTS = \frac{F}{A} = \frac{5}{1.75 \times 10^{-9}}$$

You will get 2.86 GPa.