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Module – 04 Mechanical properties of non – metallic and composite materials Lecture – 20 Strengthening of materials, fatigue, and creep

Welcome to my course Non-metallic Materials and this is module number 4, Mechanical properties of non-metallic and composite materials. And we will be taking lecture number 20 which is Strengthening of materials followed by fatigue and creep characteristics of non-metallic materials.

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In the last lecture, we introduced the concept of toughening of the ceramics and the concept of fracture toughness which was related to the storage of elastic energy upon certain application of tensile stress into the ceramic. And then, at a particular critical length of the crack, this crack will start to propagate and it will have two surfaces and the crack, the elastic store energy is released.

So, we estimated what is the critical crack length and thereby estimated the condition of the stress intensity factor when they will qualify for critical stress intensity factor. So, in

that way, we define fracture toughness of the non-metallic especially ceramic materials. Now, we will have to make the ceramic toughen so that it can withstand the stress without the propagation of the crack. So, in this lecture, we will be defining the different types of concepts which are adopted for making the ceramic toughened.

So, mainly three types of mechanism I will introduce, the first one is crack deflection and then, crack bridging and final one is transformation toughening. Now, in the next lecture, I will be taking the composite aspect of the non-metallic materials. So, some of the concepts I have taken from that lecture. So, once we proceed, then this idea will get clarified more and more.

So, after that I will describe the so called R-curve behaviour which is very special for toughened ceramics and then finally, I will describe two important time dependent failure mechanism, the first one is fatigue, when at much lower fracture stress you will see that the ceramic material they fails when they are undergone a cyclic loading tension and compression or tension-tension or compression-compression so, that I will define and the creep, it is a high temperature property under a constant stress, the material gets strained and it is a function of time.

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And let us first start with the toughening of ceramic materials and as you know, there are a variety of approaches that has been taken to enhance the fracture toughness and resistance of fracture in case of ceramic material. In metal, it is not a very major problem, but in ceramics, it is really a major problem because most of the ceramics they have very limited fracture toughness value.

And the essential idea behind this toughening mechanism is basically to increase the energy needed to extend a crack. So, you remember that we described the fracture toughness, this is related to this energy term G c and we told that if it is completely brittle, this is replaced by surface energy term and when little bit plastic deformation is there, then this energy we defined as G c. So, this is the relation that in the last class we derived.

And there are three prominent mechanism how you can do that to increase this energy needed to extend the crack. The first one is crack deflection. So, crack deflection automatically occur in a fine-grain ceramics with lot of grain boundaries.

So, as you can see once the crack propagates, and it try to tries to propagate from the grain boundary region because grain boundaries are defective region, it is a planet defect so, they are relatively softer. So, the crack tries to follow that. So, you see the crack is not going in the straight kind of fashion it is following the grain boundary. So, therefore, we call that this is a deflection kind of thing. So, this part is for a polycrystalline ceramics.

Similar effect also you can get when the ceramic is dispersed with harder this rod shaped dispersant inside the matrix, there also similar phenomena occurs the crack deflects. So, this is having an angle, some angle here so, if I define this angle, if you draw a straight line of crack propagation, it is getting deflected so, roughly say this deflected this angle theta which is around 45 degree, then you know the applied stress is basically intensified due to the presence of a crack so, that c and rho parameter is involved.

So, here what is happening this applied stress due to the crack bending like this, it is intensity is a bit less. So, this tip stress intensity that is reduced because of this deflection part and you remember in the plane stress condition, we defined this relation and the actually, the fracture toughness is the critical stress intensity factor is a balance between the creation of the surface and the elastic store energy released.

So, if you assume theta is roughly 45 degree, then you can work out that the fracture toughness of otherwise straight kind of fracture, straight kind of crack propagation this is enhanced by about 25 percent. You can work it out and you will see that the fracture toughness value is increased to 25 percent and therefore, this crack deflection that enhances the toughness of the material.

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The second concept which is important is known as crack bridging and this is applicable to a fibre reinforced ceramic composite. So, you can see these fibres are just confronting the crack propagation and the first picture is here, no interfacial debonding is taking place because what happens when crack propagates, then this intensity at the crack tip that is transferred to somewhere as so, it is doing additional work.

So, one of this work is debonding of the reinforced fibre from the matrix so, energy spent. So, the first case here it is not exactly debonding, but it is taking the thrust of the stress, of the crack, crack tip intensity, stress intensity and in this case, it is actually getting debonding from the structure so, from the matrix it is getting debonded. So, it consumes a lot of energy and therefore, the crack propagation is hindered to some extent.

So, I can define the fracture toughness in this case by this empirical relation. So, here the c component means the composite. So, Y c means the composite elastic modulus. G m is the toughness of the unreinforced matrix. So, matrix material is defined by m and the reinforced in the form of fibre is given by f.

So, you can see this relation and this relation tells that the fracture toughness will increase when the volume fraction of the reinforcing agent will increase of course, from this relation, you can see that and also the ratio of the elastic modulus of composite and elastic modulus of the fibre.

If that ratio goes up so, you will have to select your composite I mean the fibrous reinforcement in that way so, that ratio also enhances and also the interface, this gamma f upon gamma i is the ratio of the fracture energy of the fibre and reinforced matrix interface. So, that interfacial energy between the fibre and the interface, if it is increases, then also the fracture toughness will increase.

So, you can consider that as if this fibrous reinforcement is putting a small elastic strain because normally the energy to fracture of this fibrous material, it is many fold increase to with respect to the matrix material we will see in the next lecture. So, as if a spring is attached and once the crack is propagated, then it is elongating the fictitious spring and that actually spent all the energy in doing so, the fracture toughness value is increased.

So, that is the concept of the crack bridging. And in the process, you know that the intensity is lost here. So, as if this crack opening, this opening is compressed so, it is bridging as if it is bridging the crack this reinforcement and as a result, your fracture toughness is increased.

> **Toughening of Ceramics Materials Crack bridging** Composite K_{Ic} (MPa \sqrt{m}) 10 Aluming .
Mullit Glass 10_{iii} 20 30 40 SiC whisker content (vol %) Toughening is associated with crack SiC whisker increases the toughness of composites using different matrices bridging and grain pullout of elongated matrix grains in Si₂N₂

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So, a typical example I have shown that silicon carbide whiskers are used, they are very defect free fibres of silicon carbide is having very high fracture stress, it is elastically deform, it has elastic modulus which is much larger than the matrix and we have defined three different matrix aluminium, mullite and glass.

So, as you can see with the increase of the volume content of this reinforced material progressively, you see the fracture toughness is increased and due to this crack bridging phenomena and this is an example of a SEM picture. You see that when the crack propagates, this energy is spent to pull out the fibrous material from the grain matrix and it is shown for silicon nitride and that leads to the increment of your fracture toughness value.

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The third mechanism which is especially important for zirconia base ceramics. This is quite interesting. You see that zirconia in room temperature, it is monoclinic and then, at a specific temperature, it undergoes a polymorphic phase transformation to tetragonal structure and at higher temperature, it transformed into a cubic structure.

So, when the cooling is controlled while making this zirconia powder, then it is possible that at room temperature, this tetragonal phase, they that is because otherwise it should be monoclinic, but the tetragonal phase is stabilised at room temperature and it is constrained in an otherwise monoclinic matrix.

So, you can see that this type of metastable tetragonal zirconia phase, they are dispersed in a monoclinic matrix and when the crack propagates, then this constraint is removed. So, once the constraint is removed, then tetragonal phase will transform into monoclinic phase.

So, if you calculate the volume expansion because when it transformed from tetragonal to monoclinic, then the volume is expanded, and you can calculate it is about 4 percent volume expansion. So, just in front of the crack tip, there is a compressive stress filled zone. So, that it must overcome before it can propagate.

So, remember in during the toughening of the glass, the surface we put intentionally a compressive field zone and ceramics are strong in compression and weak intension, so, when the crack propagates it will have to overcome that compressive zone before it can propagate.

So, we will find that whenever the crack propagates, this energy is spent to transform this otherwise tetragonal phase to monoclinic phase. So, this is shown by this kind of laminar structure, a monoclinic structure. So, this compressive zone all the energies, most of the energies of the crack tip that is being spent in this phase transformation that is why we call it is a transformation toughening. So, due to the transformation, you can get this kind of effect.

And the same thing, you can have this when this metastable tetragonal phase is exposed to the surface of a ceramic and you are doing some work like polishing or putting a scratch on the surface, they are also this kind of phase transformation takes place and it generates a compressive field zone at the surface and basically it is strengthening the material.

So, this is one good effect that if you put a scratch on top of a zirconia ceramics in fact, its fracture toughness will get enhanced. So, that is the basic concept of the transformation toughening.

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So, as I have said that initially at room temperature, the phase is monoclinic, then at 1170 degree Celsius, it transform into a tetragonal phase and 2370 degree Celsius, it transform into a cubic phase followed by a liquid phase at 2680 degree Celsius. So, this case in transformation toughening case at room temperature, you have some tetragonal phase which is constrained in the monoclinic matrix and that leads to the transformation toughening.

So, you have three different types of transformation toughened zirconia ceramics, the first one is partial stabilized zirconia, when by the addition of a magnesia, calcia or atrium oxide at room temperature, you can stabilize the cubic phase, then you heat treated, then coherent tetragonal phase is precipitated in the cubic matrix and it is small enough so, they do not spontaneously transform, but when crack is there, they transform and they gives the transformation toughening effect.

Otherwise, you can make tetragonal zirconia polycrystal which are very high bending strength, the MOR value is quite large 2 giga pascal around that is quite the strength wise it is a large value and also you can have zirconia toughened ceramics where tetragonal zirconia particle you can finally, dispersed in a matrix like alumina, matrix like mullite or spinel and then, it can toughen those composites ceramics. So, these things are done for the zirconia toughened material.

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So, when you have a toughened material, it exhibits a weird behaviour, we call it is an Rcurve behaviour. In normal ceramic material, you have no dependence of the fracture toughness value with the flaw size, with the crack length. But here in this case, the fracture toughness increases as the flaw size is increase.

So, of course, the underlying mechanism will be operative, something similar to crack bridging or transformation toughening that means, a closer force is imposed by bridging the ligaments or the transform zone. So, therefore, this fracture toughness in fact, is increases, but not to a undefined limit so, it is having a plateau so, they are after that it more or less remains constant so, it is not a everlasting phenomena. So, it reaches a steady state.

So, once you have this R-curve behaviour, as you can see the degradation strength in normal ceramics, if you increase the flaw size, the strength is reduced, but with this kind of flaw size for a R-curve material, you have much higher strength so, strength wise, it is good, the toughened material will show higher strength.

And of course, reliability of the ceramics that I will be describing in my forthcoming classes because the scattering in the fracture strength, fracture stress is a major concern in ceramic material. But the toughened ceramics, you have more or less uniform fracture stress of this material and we will discuss it in terms of verbal distribution and other statistical parameters in my forthcoming classes.

But the disadvantage is that that I will be describing it. The reliability of the ceramics is increased, but it is more prone to fatigue. When there is a fatigue failure, you have a toughened ceramics, then fatigue strength is relatively less and it is shown that the thermal expansion coefficient in terms of the thermal shock resistance I should say that is increased for this R-curve material.

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So, now, we will talk about certain important properties in ceramic and other nonmetallic materials which is time dependent. So, fatigue is one of them. The material fail due to cyclic load for a period of time and you can see different types of cyclic load you can apply.

So, first one is a positive sinusoidal cycle which is quite irregular. The second one is a perfect sinusoidal circle cycle of the load, this tension and compression and third one is it is in the second one is only in the positive direction, it is not going into the negative direction.

So, the stress amplitude is the average of the minimum and maximum stress, but in the third case, it is tension and compression both are applicable. So, it is going up and down. So, usually, dislocation movement in the ceramics is limited. So, normally ceramics are not very susceptible to fatigue damage, but as I said in case of toughened ceramics, the case is different, and it undergoes the fatigue failure.

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So, if you see a typical static; typical fatigue plot, this is the stress amplitude I just define is plotted with the cycle, number of cycles. So, it is in that way it is can be converted to the time and we can define a load ratio term which is the minimum stress divided by maximum stress so, that gives you load ratio. So, when it is in tension plus compression mode so, load ratio is negative.

So, for smooth crack, smooth material where it is crack free, this will continue until it fails. So, the S, N curve will be something similar to this when the material does not have large crack density. But when the material is having crack density in it so, the stress intensity factor difference when the stress maximum and minimum you take sigma max and sigma min so, the delta of this stress intensity factor is given by this relation and this zeta is a geometric factor and it is having a order of unity.

So, you can see there is a plot here where the crack growth rate per cycle so, that is crack length is c so, this is dc by d N in log scale is plotted with log of this delta K 1. So, III region you can identify, there is a threshold value of the so, called stress intensity factor so, below that the crack will not grow.

So, this is termed as region I. So, cycling loading is safe in that region. In region II, this is a linear. You see that this value dc by d N is linear with delta of stress intensity factor of mode 1 and finally, at higher stress intensity rate, it will fail suddenly. So, there are three, this prominent regions here.

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So, if I define the fatigue mechanism, there is no single mechanism which can successfully explain all the fatigue data. But this is the fatigue in ceramics is fundamentally different from that on the metal. In case of metal, usually crack propagation, results, a dislocation activity at the crack tip because of the plastic deformation.

Because the material is ductile in nature and in ceramics which exhibits the R-curve behaviour, they are more susceptible to fatigue that disintegrates indicates that the cyclic nature of the loading is somehow diminishing the effect of crack tip shielding mechanism described earlier as far as the toughening of ceramic is concerned.

So, in case of fibre or whisker, reinforced ceramics which i will be describing in my next class, unloading induces fracture and that is due to the buckling of this fibres from the matrix and that in turns reduces the shielding effect.

So, if on the other hand, it is induced by bridging, then unloading cycle that causes cracking and crushing of the matrix and that reduces the frictional sliding and or at the bridging interface and that leads to fatigue. So, this is the normal mechanism which is actually defined to explain the fatigue behaviour of ceramic material.

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Creep is another time dependent degradation behaviour and this is a gradual elongation of the materials so, the strain is involved, and it is a function of time, when the load is constant. So, at ambient temperature, creep of polymer is more extensive, but metal and ceramics usually, they exhibit creep at elevated temperature.

So, the typical strain versus time curve in case of this non-metallic material is something similar to this. So, we can define a primary stage. So, you can see the creep rate is continually the slope is decreasing so, it is related to the dislocation movement and usually, it is more for ceramic and metal samples and in case of polymer, it is the chain which passes to each other. So, you know that the polymer is having the crystalline, semi-crystalline material and the chain movement is responsible for this primary creep.

So, for the rubber and polymer material, it can be defined by this relation, the strain is proportional to B, beta is a constant and t to the power m, m is also a constant and as I said in case of polymer, it is uncoiling or tangled polymer chains proceed at much lower slit at as the energetically easiest rearrangement giveaway to the reorganisation involving cross linking and the crystalline movement ah.

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In case of a secondary creep that is the linear portion so, this relation is valid here, it is a linear relation and the slope of strain versus time curve is given by this empirical relation.

So, here you see that the dislocation which are present in the sample that is actually responsible, the dislocation glide is thought to be the most important process for this secondary creep and as the temperature is further increased, then you will find that dislocation claim is important and dislocation movement control the creep rate, it is therefore, called dislocation creep or power law creep where it is a power law dependence is there.

So, we can define this term that D is diffusion coefficient of the rate limiting spaces where it is the dislocation movement, G is the shear modulus, b is the burger vector because it is associated with the dislocation, N is the number of the dislocation, sigma is the stress which is being applied, n is an exponent which is having distinct value around 5 to 6 in case of metals and 2 for plastic.

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So, at higher temperature this secondary creep, this mechanism changes as compared to the last one. So, diffusion here is thought to be the most important phenomena which is taking place and depending on the diffusion, you can see that this diffusion could be the atomic movement or as we have learned, it can be considered in an opposite movement of the vacancy.

So, when this diffusion is occurring along with the grain boundary, then we call this is a coble creep. So, atom or vacancy they diffuses along the grain boundary surface and as you can see if I apply a tensile load here so, it will be under compressive stress perpendicular to it. So, this movement is parallel to the compressive stress axis. So, this is the empirical relation.

Here also, this vacancy concentration is important, grain boundary diffusion coefficient of the vacancy is important, delta which is the thickness of the grain boundary width that is important and diameter of the grains that is important.

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So, again if you raise the temperature to still a little bit higher, then this empirical relation changes and not only that, then the atomic movement is through the lattice. So, diffuses within the grain either atom or you can consider the vacancy and this mechanism is called Herring-Nabarro creep.

And here, the value of A is a constant and your omega is the vacancy volume. Now, D here is not the surface diffusion, but the bulk diffusion coefficient and d here is the diameter of the constituent grain of the ceramic material. So, rate of diffusion creep increases rapidly as the grain size is reduced.

So, therefore, creep of the thin film on nano-structured material, they are surprisingly large as compared to the normal bulk phase material and as I have said you can consider it either a atomic movement or the movement of the vacancy along with either the grain boundary or the lattice dimension.

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So, temperature dependence as a creep is a straightforward Arrhenius relation and here, you can consider either temperature is increasing, or stress is increasing. So, if the sequence is when the stress is remain constant, then this progressively higher temperature will actually dominate the strain effect and when the temperatures are constant, these three temperatures, they are remain constant, then it is the stress value in progressively higher order will control the creep behaviour.

So, in tertiary stage, it is defined as a very rapid increase of the strain and leads to the creep rupture and in fact, voids are created in the region of the fracture and there is a considerable grain boundary movement and this part of the curve is very fast so, you cannot model it with known empirical relation as we have done for primary and tertiary creeps.

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So, the reference to study this material are mostly taken from the book by Tilley and Barsoum, the reference is given here.

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And then, this particular lecture I talked about the toughening of ceramics mostly three different types of toughening was covered crack deflection, bridging and transformation toughening. Then, toughened zirconia ceramics is a classification that was explained. Rcurve behaviour of the ceramics which is basic very special for ceramics for toughened ceramics that was described and fatigue in ceramics, little bit fatigue micro-mechanism and as well as creep in the non-metallic material has been described.

Thank you for your attention.