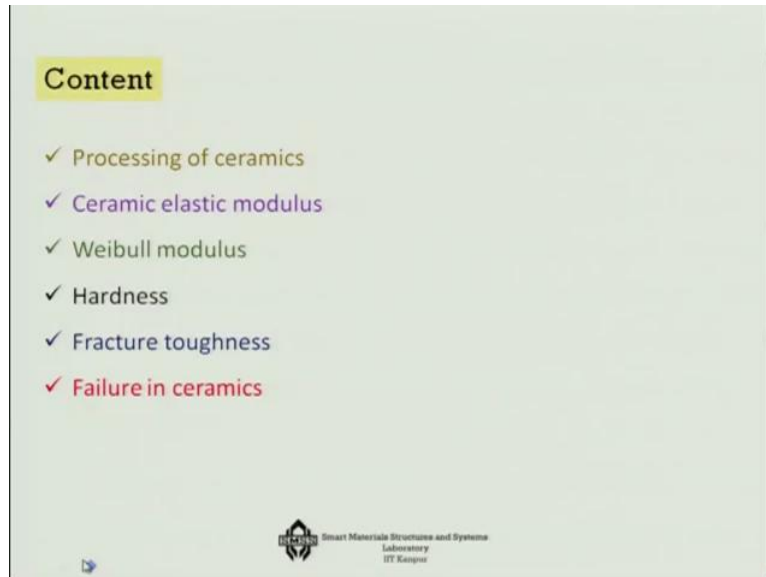


Nature and Properties of Materials
Professor Bishak Bhattacharya
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Lecture 15
Ceramics 2

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Okay, we have discussed about the basic introduction about the ceramic materials in the last lecture. Today we will see some of the more advanced topics for example, we will 1st talk about the processing of ceramics, and then we will talk about the elastic modulus of ceramics. And more importantly for ceramics because it is always a probability that is associated with the ceramics in kind of uncertainty about any value of the mechanical parameters, so the concept of Weibull modulus. And then we have to talk about the hardness, fracture toughness and failures in ceramics. So this is how we would like to cover up about more details related to the ceramics.

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Processing of Ceramics

- Unlike **metals and glasses**, which can be **cast** from the **melt** and subsequently rolled, drawn, or pressed into shape, **ceramics** are made from **powders**.
- **Ceramics powder** is consolidated and densified by **sintering**.
- **Sintering** is a process whereby **particles bond** and merge under the influence of **pressure & heat**, leading to shrinkage and reduction in porosity.
- Expensive fabrication technique due to **high cost** of mould & die.
- A similar process in **metal manufacturing** is referred to as **powder metallurgy**.

Sintering

References: Engineering Materials 2: Ashby & Jones, 4th Ed.

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References: W.D. Callister, 7 Ed.

The diagram illustrates the sintering process in three stages. The first stage shows four separate red circular powder particles. The second stage shows the particles beginning to coalesce, with necks forming between them and a central pore forming. The third stage shows the particles further merged, with the pore changing size and shape. Labels include 'Powder particles', 'Neck', 'Pore', and 'Grain boundary'.

Now 1st point is the process of ceramics because many of the properties of ceramics are actually dependent on the processing parameter and how this processing has undergone. So unlike metals and glasses, which actually can be cast from the melt and subsequently rolled down or pressed into shape, ceramics are generally made from powders. And there are actually 2 way in which we can do it, one is through a process of Sintering, which we will talk today that Sintering and pressure and heat.

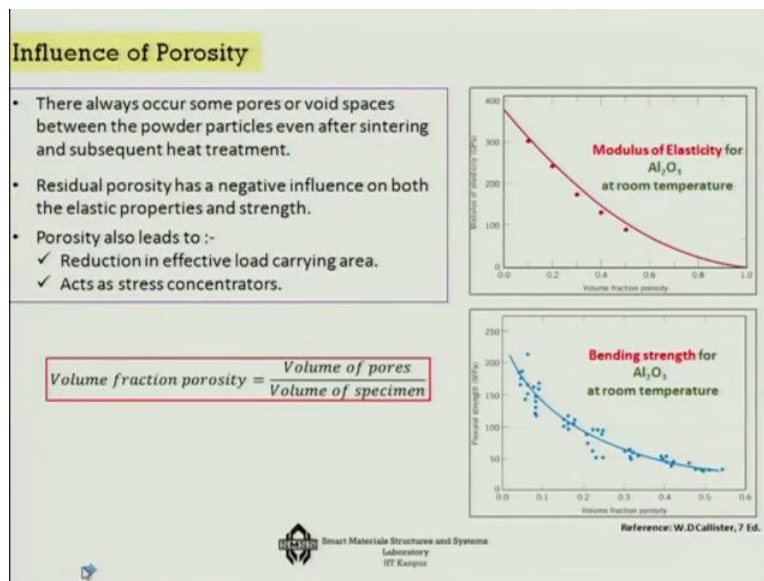
In another process which I will talk later on for piezoelectric ceramics but not right now is known as the Sol gel process. So in Sol gel processes you do not need this application of heat, but right now the more traditional one is the Sintering and that is what I will be discussing in this particular lecture. So ceramics powder is consolidated and dignified by sintering. Now, what is sintering?

This is a process whereby particles bond and merge under the influence of pressure and heat, so high pressure and high temperature. This will lead to shrinkage and reduction in porosity as well, but this is what we have tried to show you here that these are the say powder particles to begin with and then you are applying high temperature and pressure.

And what will happen is that there will be some pores and the particles are going to merge, these are the neck areas and the grain boundaries are here, particles are going to merge and as you are applying more and more pressure and temperature, this is shrinking from these to this particular you can see that it is gradually, there are pores but it is shrinking. Now, this undoubtedly actually gives the high cost of the fabrication in the ceramics.

That is one reason, and of course the generation of temperature and pressure is what actually stopped us to use ceramics for a long time for very advance high-performance ceramics type of applications. But today we can we have the hold of the entire technology. Now, a similar process in metal manufacturing also this is very similar and very analogous and this is referred as powder metallurgy okay just for your reference. Okay, so this is how the ceramics are processed and we are showing here that in this process of making the ceramics itself, the pores are inhibited. So what is the influence of the porosity?

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Because there will be always some pores or void spaces between the powder particles even after sintering and subsequent heat treatment, pores are unavoidable. The porosity has some negative impact on elastic properties and strength. For example, you look at it here that the modulus of elasticity of Al₂O₃ is at room temperature it is continuously coming down as the porosity is increasing.

Similarly, if you look at it that the volume fraction porosity as it is increasing, the strength is coming down. Of course, one interestingly you can see that much unlike the modulus of elasticity, the strengths are not like single pot you can see that there are more distinguished fashions that is what I will be talking later on that this distribution is actually probability related and that is why there is Weibull modulus that is related to the whole thing.

Now, volume fraction of porosity that plays a very important role, which is volume of pores over volume of the specimen that is what, is the volume fraction of the porosity, so higher the volume fraction you expect more degradation of the material properties particularly the

strength of system. And how do we measure the volume porosity? Well, there are several ways one is called BET technique okay by which actually we subject these ceramic materials to absorb the gas and then depending on how much of gas is absorbed, we actually find out that what the porosity of the particular ceramic sample is. Now, how do you measure the ceramic elastic modulus okay?

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How to measure Ceramic Elastic modulus?

Ceramics are not subjected to Tensile testing due to 3 important reasons:

- ✓ Difficult to prepare test specimen as required for tensile testing.
- ✓ Difficult to grip brittle materials without fracturing.
- ✓ Ceramics fail at very low strain ($\approx 0.1\%$), which requires very precise alignment on machine to avoid bending stress.

Hence, three-point bending test is used.

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Um It is like looks like a very trivial question, but it is not trivial at all. Why because when you have to make a particular sample okay for elastic modulus determination, so for example this is a steam sample. So when you have to make this sample, you have to make certain necking arrangements okay. So this is a dog bone shape of the sample.

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So you have to make this dog bone shape and this kind of dog bone shape is very difficult to make for the this making, et cetera for the ceramics, so sample preparation is the 1st important problem. The 2nd problem is that, in the tensile testing okay, we put these areas for gripping okay, but this gripping for ceramic material is an enormously difficult.

And other point is that the ceramics actually fail at a very low strain, something like 0.1%, so it is very difficult to align a machine and to avoid bending stress hence, the testing of ceramic a kind of a indirect way which is known as Three point bending that is generally used. Let us see what is this three point bending test?

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Three-point bending test

- At the point of loading
 - ✓ Specimen top surface in compression
 - ✓ Bottom surface in tension
- Stress is computed from the specimen thickness, B.M, and the M.I of the cross section.
- Maximum tensile stress at the bottom surface directly below the loading point.
- Fracture occurs on the tensile specimen face.
- Tensile strengths of ceramics are about one-tenth of their compressive strengths.

Possible cross sections

$\sigma = \text{stress} = \frac{Mc}{I}$

where M = maximum bending moment
 c = distance from center of specimen to outer fibers
 I = moment of inertia of cross section
 F = applied load

	M	c	I	σ
Rectangular	$\frac{Fl}{4}$	$\frac{d}{2}$	$\frac{bd^3}{12}$	$\frac{3Fl}{2bd^2}$
Circular	$\frac{Fl}{4}$	R	$\frac{\pi R^4}{4}$	$\frac{3Fl}{\pi R^3}$

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Reference: W.D Callister, 7 Ed.

As this name suggests itself that this is a ceramic sample as we can see here and it has actually 2 supports and exactly at the Centre the load is applied, for it is a 3 point of bending that is happening, so it is a 3 pointed supports specimen here at the top surface okay, so this part is actually subjected to compression and the bottom surface is actually subjected to tension.

Now, you can compute stress from the specimen thickness, bending moment and moment of inertia. So if it is of length L , then L by 2, L by 2, it is exactly intermediating point okay. And the sample geometry suppose B over D okay for rectangular cross section or it can be of circular cross section depending on what you wish to choose.

Now, if it is a rectangular cross-section, then with this configuration and with the force applied F , the bending moment is $F L$ by 4 okay. So this bending moment in fact if you look at it, it is done in such a manner that this $F L$ by 4 is the bending moment that if this thing is subjected to and if I know this bending moment M and if I know that where exactly this particular thing is located, then I can actually the particular fibre.

For example, if it is an extreme fibre so the distance is $N Y$ by I , so I can actually find out that what is the stress it is subjected to. So thus we can actually from the 3 point bending test and then the finding out the bending moment and the sample geometry, we can actually find out that what the compressive stress is and what is the tensile stress that the sample is subjected to.

And then we can also note down that at what stress, so corresponding to load we can find out the stress and at what stress the failure is occurring to the sample and generally the failure is in tension. Tensile strength of ceramic are about one tenth of their compressive strengths, so we expect that the failure start from here at the bottom okay because the resistance to tensile stress is much less in comparison to the compressive stress.

The same thing we can also do for, so this is the stress corresponding to rectangular cross-section, this is the one for circular cross-section. So either we consider a circular sample or we consider a rectangular surface. Next is that we can now we can find out the stress and also if you actually use some displacement sensor, you can find out the strain and you can then plot this stress strain diagram?

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Modulus of Elasticity

- The slope in the elastic region is the modulus of elasticity.
- Range of moduli of elasticity for ceramic materials is between about 40 and 1000 GPa (Diamond).
- Ceramics have compressive strength 10-15 times of their tensile strength.
- Under compression failure occur either by crushing or buckling.

Reference: W.D Callister, 7 Ed.

Material	Modulus E (GN m ⁻²)	Density ρ (Mg m ⁻³)	Specific Modulus E/ρ
Steel	210	7.8	27
Aluminum	70	2.7	26
Aluminum Al ₂ O ₃	380	3.9	100
Steel SiC ₂	400	3.8	107
Carbon	45	2.4	19

Reference: Engineering Materials 2: Ashby & Jones, 4th Ed.

Silicon carbide under compression
Reference: www.ceramics-uk.org.uk

Comparison under Tension & Compression

Stress $\sigma = F/A_0$

Slope $E = \sigma/\epsilon$

Strain $\epsilon = \Delta L/L$

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What you will see is that the stress strain diagram unlike here metallic samples is remarkably different here. The stress strain diagram you will gradually generally you will find them to be a simple straight line as you will see here for Aluminium oxide or glass and a sharp point of failure okay. So there is no plastic region that is observed and this is of course the modulus of elasticity when you test them in tension.

Interestingly, if you test them in compression then you would see that this kind of a failure than that will be happening, so instead of a single direct failure like tension it will happen in stage by stage by stage in compression. So in fact, this kind of a step-by-step failure you can see that Silicon carbide under compression, so how with respect to time you can see that this kind of failure is happening to the system. So that is the point that we will see the taking on thing is that you will see that the elastic modulus is generally quite high, but the failure strength is very low and the strength is not very high because of the presence of this porosity.

then at a particular applied stress value, I can find out that how many samples have failed at a stress lower than this particular stress value, so that will give you the P_f .

So N_x is actually that the number of samples that have failed below a particular stress value, so N_x over $N + 1$ then will give us the P_f . Now if I know P_f and I know the probability of success is actually the reverse of P_f that is it is $1 - P_f$ okay. So I know the P_s and if I know P_s okay and I know the average strength Σ_0 that is known to me okay and I also know what is the applied stress value at which I am calculating this.

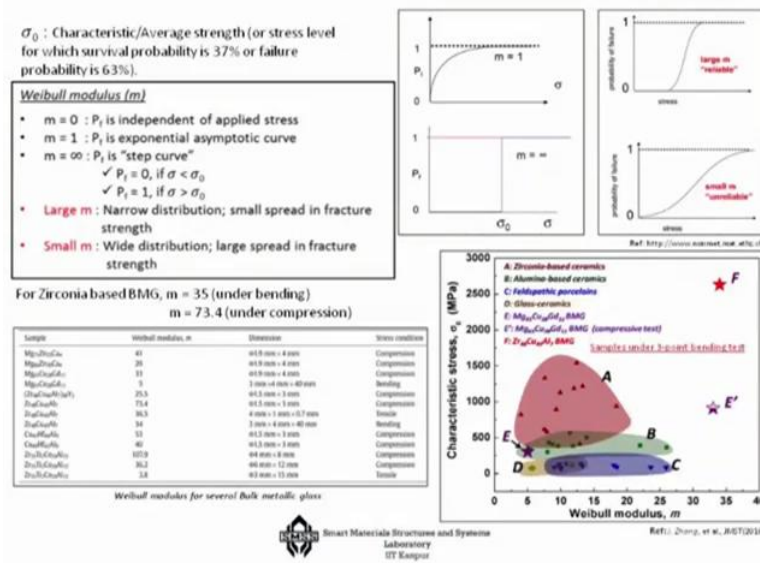
So by a simple label of plot then I should be able to find out that what is the slope of this line that means I should be able to find out what is the value of M okay. In this case, what we have tried to show is that this value of the N if you look at it the slope of 0.2% Carbon hot rolled steel okay, we will see that the value of the N is very high, it is like a 90 degree, whereas this slope here is it has a finite flow in this particular case.

Not only that, the same material if you actually reinforce with Alumina for example, with a controlled particle size because the particle size variation actually makes this M to be here and there, so if you do that you would see that the value of M will be higher 4.7, so in fact it is the slope is actually increasing. Not of course as I have the hot rolled steel, but it is better than the slope that you would see in this particular case.

So this tells us that the Weibull modulus is a very important factor because that gives us an idea of the variation of strength or such mechanical properties in ceramic materials. And secondly, the manufacturing process should be guided in such a manner that we will get Weibull modulus so that the ceramics can actually compete with the metallic counterparts because nobody would like to design a product where the strength can be anything, the strength can be varying between suppose 100MPa to 100MPa, you would not like to design a simple like that.

So you would like to design a product with a material, which gives a very higher value of Weibull modulus. So to do that we have to keep track of that how this Weibull modulus is going to change as I am introducing various reinforcements, composites, et cetera or changing the parameters like the porosity making it more uniform porosity, etc and increase the value of the Weibull modulus.

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You can see it here for example that this is the Weibull modulus N equals to 1, so N equals to 0, P_i is independent of applied stress, N equal to 1, P_f is exponential asymptotic curve, M equals to infinity, P_f is the step curve, so that is M equals to infinity that is the step curve okay. So large M essentially would mean that there is a narrow distribution of the property that means the particular property is we call it as very Crisp, so it is we will call it as a Crisp probability that means the material property variation range is very small.

On the other hand, the smaller M means it is a wider distribution, larger spread in fracture strength. So here the Weibull modulus as you can see here as the Weibull modulus is increasing, you can see that these points are coming F or E prime, etc, okay. So these are like bulk metallic glasses okay. And then as you are somewhere around A , B , C s, you can see at Zirconia based ceramics, Alumina based ceramics, okay or (18:25).

And when you talk about usual glass ceramics, etc, here somewhere give the very low Weibull modulus something like glass ceramic okay. So that is how for various ceramic materials, the Weibull modulus varies. Now, then we talk about some of the other properties like the hardness, which is very important because I told you earlier also that ceramics have one of the good applications of ceramics is in terms of shaping or grinding or giving a smooth texture to metallic tools, et cetera.

So ceramics are hard and brittle due to the presence of ionic and covalent bonds, which have the much higher bond energy in comparison to the weak bonds like hydrogen bonds, et cetera. And this property is utilized when we are making abrasive or grinding action,

ceramics having Knoop hardness of about 1000 or greater are utilized generally for their abrasive characteristics.

Suppose, this one is a Tungsten carbide wheel or a Diamond abrasive, these are generally used and you can use it for actually sharpening metals okay. And their Knoop hardness if you look at it, Diamond about 7000, Boron carbide 2800, and anything greater than 1000 that means up to Alumina, you can actually use them for abrasive or grinding purposes.

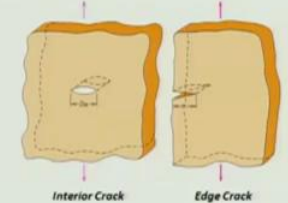
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Fracture toughness of Ceramics

- ✓ Ceramics – highly brittle in nature, thus *low fracture toughness*.
- ✓ Fracture toughness – a material property
 - Ability to resist crack propagation.
 - Measurement of the energy required to grow a thin crack
 - Unit = $\text{MPa}\sqrt{m}$
- ✓ Low Fracture toughness = Brittle failure
- ✓ High fracture toughness = Ductile failure

Fracture toughness, $K_c = Y\sigma_c\sqrt{\pi a}$

Where,
Y = dimensionless parameter
 σ_c = critical stress for crack propagation
a = crack length



Interior Crack Edge Crack

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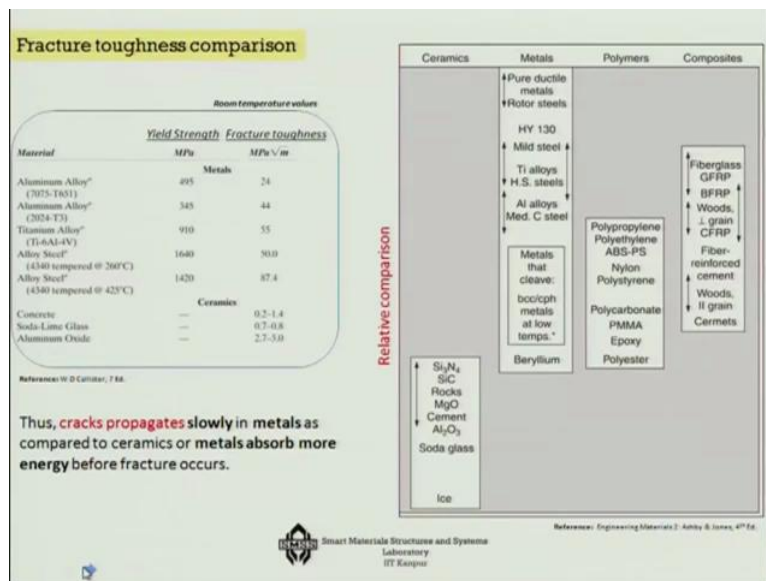
Reference: W.D Callister, 7 Ed.

The next important thing in the ceramics is the fracture toughness of the ceramics okay. Now ceramics they being highly brittle in nature, they have low fracture toughness. And fracture toughness, I have not talked about it earlier; it is a material property which talks about the ability to resist the crack propagation. And it also talks about the energy that is required to grow a thin crack before the failure occurs and the unit of it is megapascal times square root of meter.

Now low fracture toughness means brittle failure just like earlier I showed you brittle failure of the chalk samples and the high fracture toughness, it means ductile failure. I showed you also ductile failure of the mild steel samples. Fracture toughness is actually defined in terms of the product of these 3, where Y is dimensionless parameter, Sigma c is the critical stress that is needed for crack propagation and A is the crack length okay.

So there can be various types of cracks like interior cracks as you can see it here or edge cracks okay. Also there are various ways in which we can apply the stress and depending on the failure occurs, I will talk about it at the later stage.

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Now, fracture toughness is being a very important parameter because we would always like to have a sample to have higher fracture toughness, so this is an Ashby chart which gives a relative comparison of fracture toughness. And as you can see here that the fracture toughness of ceramics here somewhere at this point, this is for the ceramics okay. And this is where we have the fracture toughness of the metals okay.

This is where we have the metallic fracture toughness, then polymers and then the composites. So if you compare it, you can see that Aluminium alloy, what is the fracture toughness that is about 24 megapascal square root meter. And if you compare that with Alumina for example, okay very low, 2.7 to 5.0.

So fracture toughness of ceramics are we can see here that it is qualitatively it is lower in comparison to the metals, polymers and composites, one can say that just by shutting eyes itself okay. And among the metals of course there are variations like the mild steel has higher fracture toughness than Aluminum alloys okay.

And among the polymers even though they can sustain a much of strain that their modulus of elasticity and also the yield strength being low so you will not get a fracture toughness beyond a certain value. And composites you will you will get kind of a mixing of these possibilities okay.


So because you can have a metal ceramic composites, where metals will have the high fracture toughness because of the ceramics it will come down a little bit and you can have again the polymeric composites, ceramic polymer composites, where it will be once again

polymers will have slightly higher fracture toughness and ceramics will bring it down and you get some kind of an average value for the composites. So that is what is the fracture toughness comparison of various types of materials. This is particularly important whenever we are considering the reliability of a particular product.

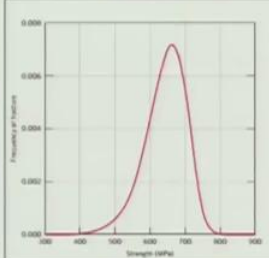
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Failures in Ceramics

- Ceramics have **negligible plastic deformation** before fracture (**no warning**).
- The brittle fracture of ceramics limits its applications.
- Occurs due to the **unavoidable** presence of **microscopic flaws** (micro-cracks, internal pores, and atmospheric contaminants) that result during fabrication.
- The **inherent flaws** leads to crack formation and propagation.
- Ceramics are good structural materials under compression as difficult for crack to propagate.
- The **flaws cannot** be closely **controlled in manufacturing**; this leads to a **large variability** (scatter) in the **fracture strength** of ceramic materials.

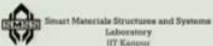


Brittle failure



Variability in fracture strength for Silicon nitride

Reference: W.D Callister, 7 Ed.



Now, failures in ceramics there are certain territory signs of it. One is that it has negligible plastic deformation meaning thereby that it will almost give you no warning okay, so in fact you see the collapsing of the bridges for example, we will hear about that quite after. Now, many of these bridges are made of concrete structure okay, so concrete structure is having predominantly ceramic materials in it also, it is having some reinforcement like mild steels.

But if this reinforcement is below a particular level okay, so below a critical level then it becomes actually the governing in the failure is governed by the ceramics. So it is a ceramic governed failure that comes into picture if reinforcement is given below a particular level due to poor quality maintenance, corruptions, et cetera.


Now, if that happens then this kind of structures will be susceptible to sudden failures, we may see that bridges collapsing suddenly why because the steel that is supposed to be going there is not given or it got corroded or something and hence it is no longer a metallic driven failure or you will get warning for quite some time because it does not fall of a sudden. On the other hand, if it is a ceramic governed failure, it will be a brittle fracture and it will happen all of a sudden.

So this is what is something that we see many a times okay. And this occurs due to the unavoidable presence of microscopic flaws or micro cracks in the ceramics. And these cracks are unavoidable because these are inherent these are the inherent flaws that are there in the system. So in order to have ceramics with high strength you need to control these flaws okay during the manufacturing.

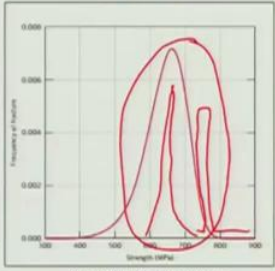
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Failures in Ceramics

- Ceramics have negligible plastic deformation before fracture (no warning).
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- Occurs due to the unavoidable presence of microscopic flaws (micro-cracks, internal pores, and atmospheric contaminants) that result during fabrication.
- The inherent flaws leads to crack formation and propagation.
- Ceramics are good structural materials under compression as difficult for crack to propagate.
- The flaws cannot be closely controlled in manufacturing; this leads to a large variability (scatter) in the fracture strength of ceramic materials.



Brittle failure



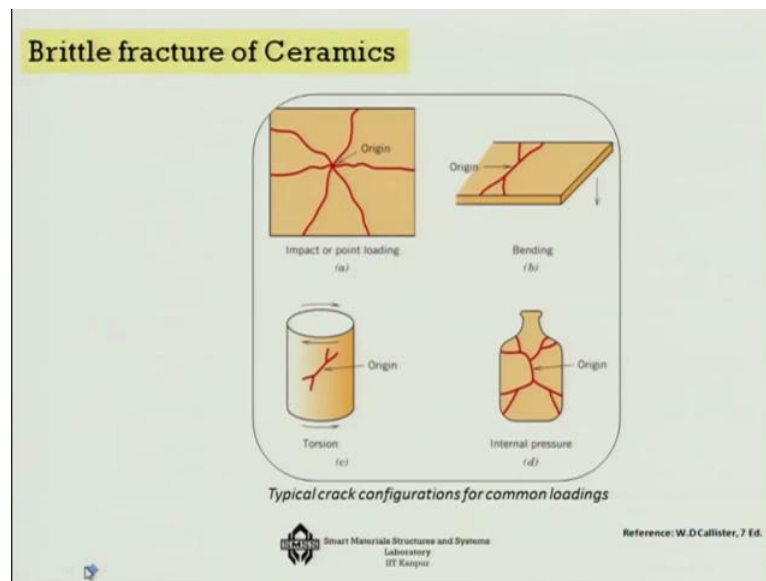
Variability in fracture strength for Silicon nitride

Reference: W.D Callister, 7 Ed.

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And if you if you cannot control it, what you will get is this kind of a strength variation okay, so this will become sharper and sharper as you can control it more and more. In fact, for metals you will get it almost like you call it a Crisp probability I told that a straight line scenario. So that is what that is why the manufacturing process is so important in the ceramics.

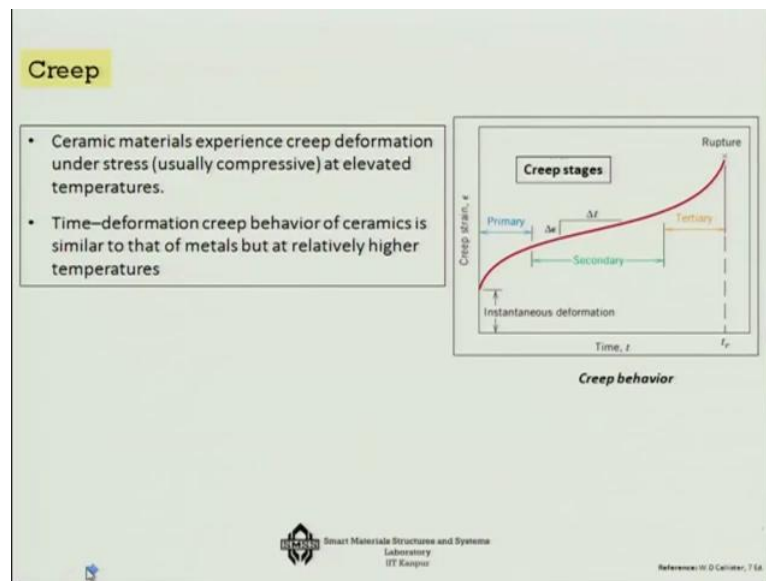
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Now this is some of the pictures that show various types of brittle fractures in ceramics like if you have the impact or point loading, one of the ceramic tiles says, you will see that this is the way the cracks are propagating. Or if it is bending, you would see that this is the way the cracks are propagating. If it is a torsion, you will see it to the 45 degree angle okay, which you will see many strength of materials this thing that white is 45 degree angle occurs because that is the principal plain where the failure is occurring in tension.

And then if it is due to internal pressure, you would see this type of a failure. So by looking at the failure, you can say the fact that the crack can actually give us idea of whether this has happened due to impact, due to bending, torsion or internal pressure (())(27:33) that is very important. The next important thing is the creep because ceramic materials do experience lot of creep under stress and at elevated temperature.

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If you look at the creep, there are actually 3 parts of it. 1st of all is the primary creep, where it is quite predominant that the creeping strain is actually increasing with respect to time, then it gets stabilized that is the secondary part and once again it increases at a sharp rate at tertiary region okay, so as the time continues this happens at a sharper rate towards the end, so that is what is the nature of creep in the ceramic material.

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How to make Ceramics Conductive?

Two ways to make ceramics electrically conductive.

- ✓ At sufficiently high temperatures point defects such as oxygen vacancies can arise, leading to ionic conductivity.
Example: Zirconia
- ✓ Introduction of certain **transition-metal** elements (such as iron, copper, manganese, or cobalt), **lanthanide elements** (such as cerium), or **actinide elements** (such as uranium) can produce special electronic states in which mobile electrons or electron holes arise.
Example: Copper-based superconductors are a good example of conductive transition-metal oxide ceramics—in this case, conductivity arising at extremely low temperatures.

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Now, how do we make ceramic conductive because ceramics are generally insulating in nature, so there are 2 ways in which you can make the ceramics electrically conductive. One is that you raise the temperature because at sufficiently high temperature, the point defects

such as this oxygen vacancies can arise and this will lead to ionic conductivity, so that is something that happens in the Zirconium.

The other is that; introduce transition metal elements such as Iron, Copper, Manganese or Cobalt or lanthanide elements such as Cerium or actinide elements such as Uranium. If you use this, this can produce a special electronic state in which the mobile electrons or electron holes will increase. So this is like copper-based superconductors, this is one of the best examples that ceramics in general are not conductive they are highly insulating, but the superconductors are made by using ceramic along with one of this transition metals and then you increase the conductivity.

Interestingly, this conductivity is arising in extremely low temperature, so in the one hand we have this high-temperature raised conductivity that is in the Zirconia and on the other hand, this is at extremely low temperature superconductivity, where you are actually adding transition metal elements to introduce conductivity into the ceramics, of course you can make ceramic composites to do the similar kind of a thing.

So this is where we will come to an end in this lecture, in the next lecture now we have discussed about metals, we have discussed about ceramic, so now it is the time to discuss on the polymers and the classification of the polymers, Thank you.