Friction and Wear of Materials: Principles and Case Studies
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Lecture – 23
Tribological Behavior of Dental Restorative Materials

Welcome back in this subsection of this particular course as part of this particular course on Friction and wear of materials. I am giving this series of lectures, particularly that last few lectures on biomaterials and bioceramic material. So what I am planning to do in this lecture is to show you some of our published research results on dental restorative materials. Before I show you the results of the dental restorative materials, it is important for you to understand that what is, how is the microstructure and properties of the natural tooth is like.

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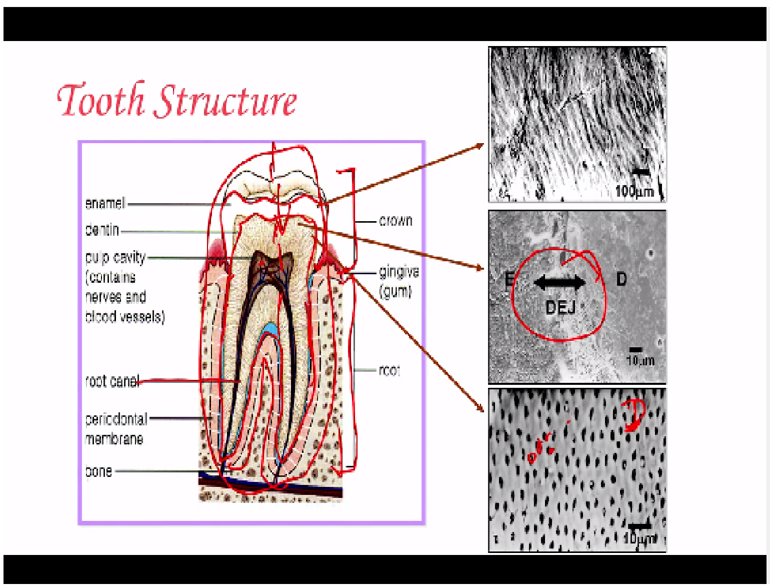

So this is the entire full tooth, whole tooth. So what you see is the top is that enamel that most of us know and then comes the dentin. So enamel and then comes the dentin which goes into the root of this dental cavity. Then you have a pulp cavity here which contains nerves and blood vessels. You have a root canal. All of you are aware of the root canal therapy. Then you have a periodontal membranes and then also this is the surrounding bone.

So certainly crown of this dental, of the natural tooth would contain enamel and part of the dentin. Whereas gingiva is the gum and the root part of this natural tooth is largely contains the pulp cavity as well as bone and the dentin. So we do not see any bone in the top part, this crown part which contains all of the enamel and some part of the dentin. Now one of my former student has done some careful analysis of the microstructure and then properties of this natural teeth.

So he took this lost tooth of several patients of different ages. So this is the microstructure of the tooth of an adult person and what you see if you take in the top or the crown, you can see some typical enamel like features. Then if you go down at the dental implant, so you have to take a section, right and if you take a section, then you go down from enamel to dentin. So you have the DEJ that is the dental enamel junction and this is the part of that dentin here.

So this microstructure shows that what is the dentinal tubules, very nicely. Now these dentinal tubules are important because all these microstructure features, they give rise to specific mechanical properties.

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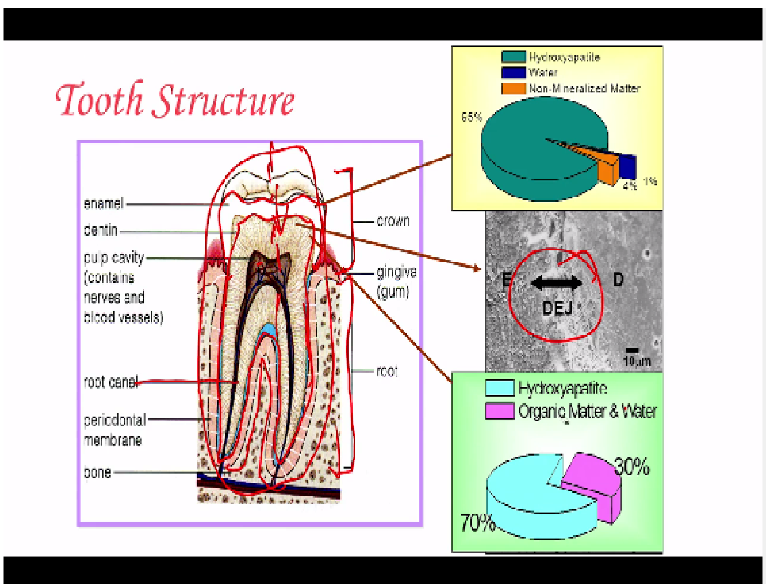

Now in terms of the content of this natural tooth. It is largely hydroxyapatite as you can see 95% is little bit of water and non-mineralized matter in case of the enamel. But in case of dentin, it is 70% hydroxyapatite and 30% is organic matter. So 30% organic matter like proteins and so on and inorganic material like hydroxyapatite is contained in dentin like up to 70%.

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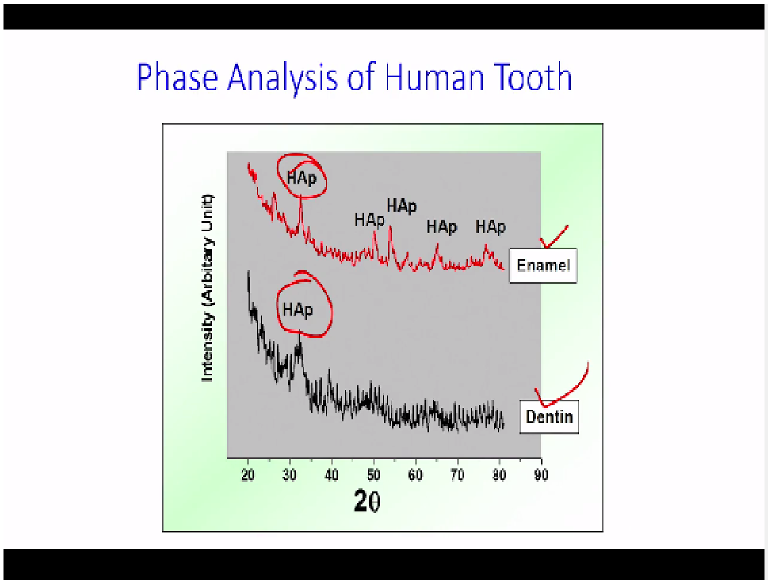

Now if you take separate part of the enamel and dentin and do x-ray diffraction, what you see here, there is a very clear indication of the hydroxyapatite phase. Now in these hydroxyapatite phase is also crystalline in nature.

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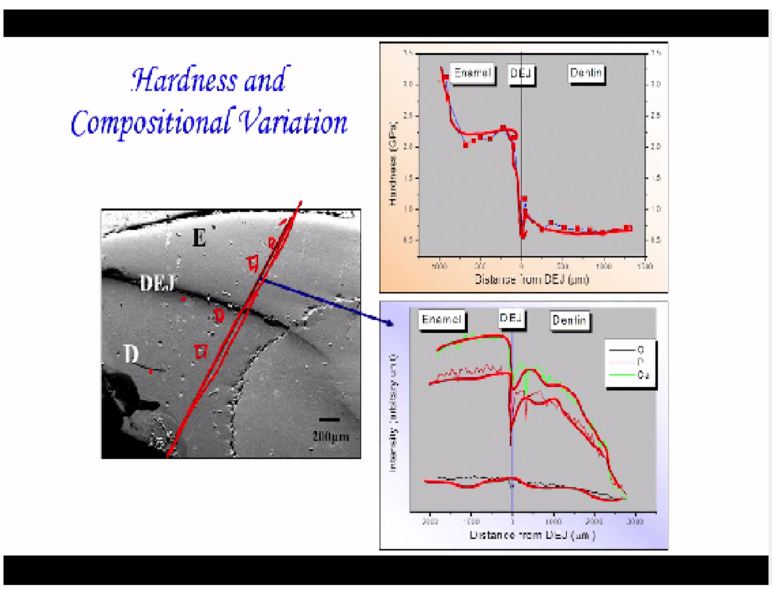

So you have a crystalline hydroxyapatite. As I said few minutes back that we have done very careful and systematic analysis by taking Vickers indentation. So this is the small Vickers indentation we have taken at different distance across this section from enamel to dentin enamel junctions to dentin and this is the result you get. So hardness typically as expected enamel part, the hardness is higher like 2 to 3 gigapascal.

If it goes to dental enamel junction, the hardness drops and if you go to the dentin junction, the hardness is around 0.5 somewhere between around 0.75 gigapascal. So this 0.75 gigapascal, many materials they can easily satisfy this kind of hardness requirement. However, the most important thing for you to realize here that there is a gradient in the hardness. So this gradient in the hardness is more difficult to create in a given microstructure.

I am not saying it is impossible but it requires lot of, it requires quite a bit of experiences from materials processes as well as microstructure designing which is in the domain of the material science field. What is another thing that you must realize here that it is that careful EDS analysis, energy dispersive spectroscopy analysis which can be done together with the scanning electron microscopy analysis.

And what you see in that calcium content is reduced as you go from enamel to dentin region. So is the trend for the phosphorus and oxygen content does not change. So what you are saying that hydroxyapatite content is typically reduced and what you have seen in that one of the earlier slides that enamel, the hydroxyapatite is 95% and dentin hydroxy is 70%. So one would expect that hydroxyapatite phase also reduced and that is what has been reconfirmed using that EDS based compositional analysis.

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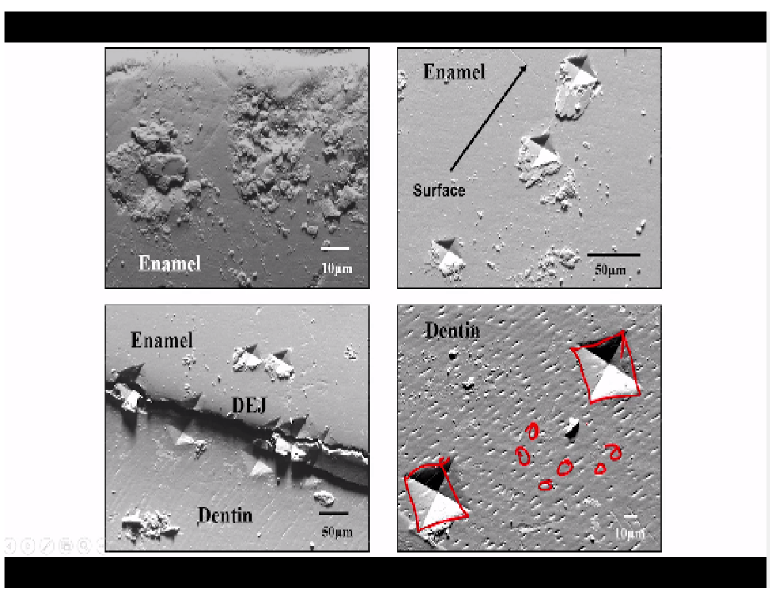

And you can see that how this indent they appear in this, at this dental enamel junction as well as the enamel part as well as the dentin part where you have this. If you remember, you have this small tubules, microtubules and you have a very clear, well distinct Vickers hardness impression in the dentin part as well.

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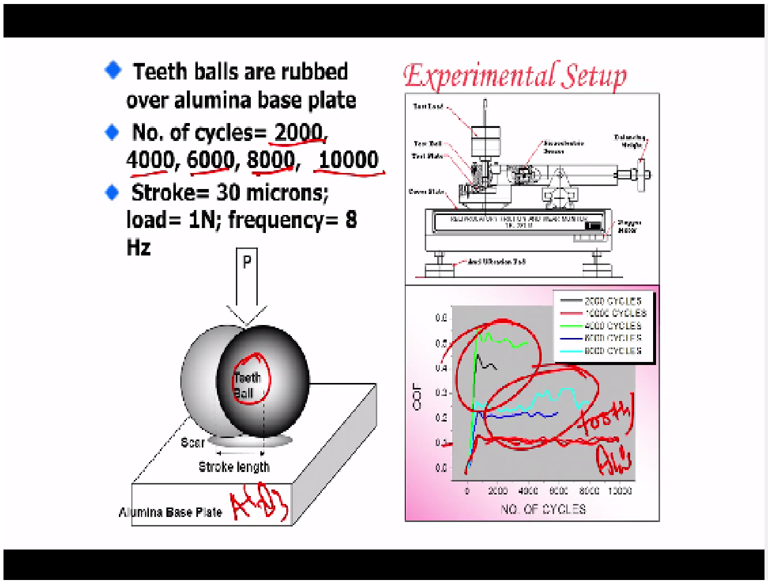

So we have not only done the mechanical property characterization microstructure but also we have done this wear study, fretting wear study on natural teeth. And what we have done? We have taken the teeth ball. What is teeth ball? Teeth ball is that we have taken this natural teeth. In some teeth, depending on which part of the mandible you have taken the teeth, then you can put this ball at one of the external surface of the tooth.

You can slightly polished and then give it a shape like a ball. And then what we have done? We have taken that as an opposite counter body, mating body and aluminize the base plate. So this is that alumina. So why alumina? Because there has been a continuous search for all ceramic dental implant and in case of all the ceramic dental implant, one of the material which has been widely investigated is, ceramic crown is the alumina or zirconia.

So alumina and zirconia, the ceramic material which can be used as a dental replacement or dental restorative materials. So from that point of view, we have investigated whether alumina, what is the friction and where of alumina against that natural teeth. So what are the cycles that we have used? Up to 10,000 cycles but different intermediate cycles also we have stopped the test just to show, just to see that how wear progresses with time?

What is the stroke length? It is fairly small, is 30 microns. The load has been taken very low, 1 Newton because we are trying to simulate the masticatory forces in the oral cavity and we have done some calculations where we have found that if you take the load of 1 Newton and if you consider the elastic modulus of the teeth ball and alumina, then we are able to simulate the typical masticatory action related stresses or equivalent stresses at the contact zone.

Now this is the frictional behavior what you see as a function of cycles. So this is like 10,000 cycles you can see very steady state behavior of around 0.1. So remember this is the mating couple tooth versus alumina, okay. Now at the larger number of cycles, there is a variation and this variation depends on we have to use the different teeth which is taken from different location and patient.

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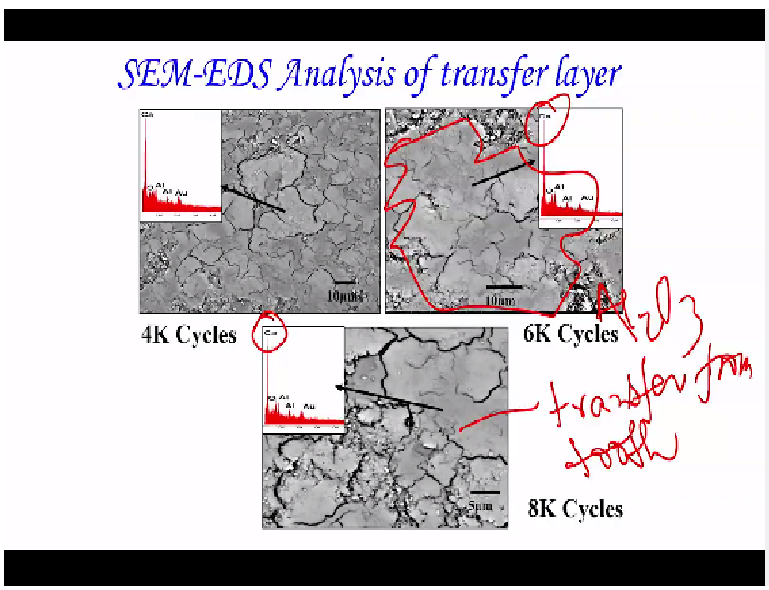

So essentially that frictional property, frictional properties indicate that it is possible to measure low coefficient of friction like 0.1 and then these tests were done in the artificial saliva solution just to simulate that maxillofacial environment. Now if you look at this transfer layer which is formed on this alumina surface, what you notice that there is a very strong peak of calcium and essentially these transfer plane what you see on that flat surface, this is the alumina surface, okay.

So essentially this is the transfer from the tooth. So because it is a transfer from tooth, that is why this essentially tooth ball is fitted or worn away and then that is being transferred to that alumina surface leading to this transfer layer.

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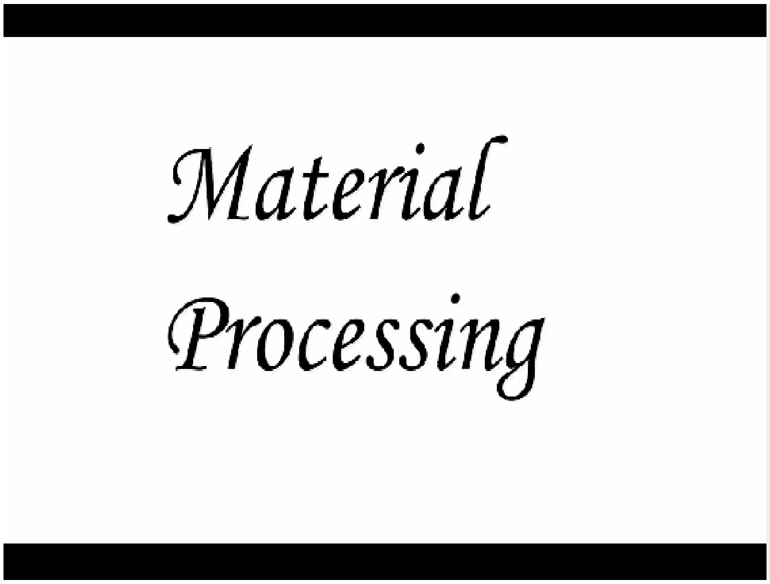

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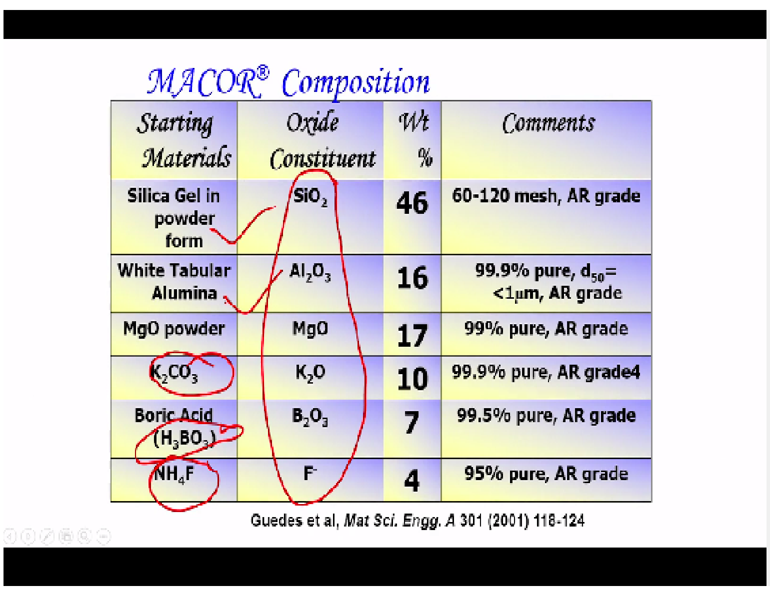

Starting Materials	Oxide Constituent	Wt %	Comments
Silica Gel in powder form	SiO_2	46	60-120 mesh, AR grade
White Tabular Alumina	Al_2O_3	16	99.9% pure, d_{50}= <1μm, AR grade
MgO powder	MgO	17	99% pure, AR grade
K_2CO_3	K_2O	10	99.9% pure, AR grade4
Boric Acid (H_3BO_3)	B_2O_3	7	99.5% pure, AR grade
NH_4F	F^-	4	95% pure, AR grade

Now in order to develop the dental restorative materials, particularly the crown materials, now I will show you some of the results that we have got by developing mica based glass ceramic that is machinable glass ceramics. So it has a typical composition and this is your different, basically oxide glasses, essentially silica based oxide glasses and it has an alumina, magnesium oxide, K2O, B2O3 and also fluorine.

Now fluorine is typically there, we used this NH4F as the precursor. For B2O3, we have used H3BO3 as a precursor, K2CO3, white tabular alumina we have used and silica gel we have used in the powder form.

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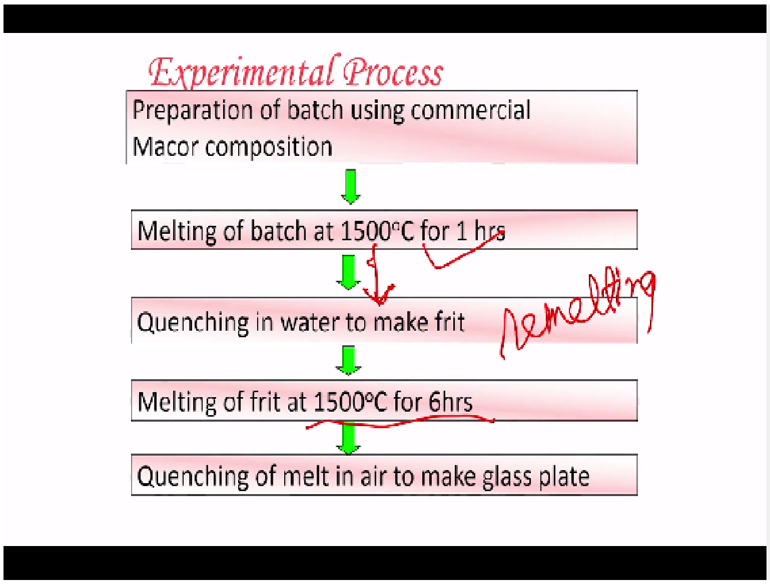

So this is the typical manufacturing protocol. So you mix it, then we melt the batch at 1500 for 1 hour in platinum crucible. Then we quench it in to water to make frit. But remember, we have used this multi-component glass materials. So in case of multi-component, one single melting is not good enough. You have to do remelting. So remelting essentially means you take the frit, again you melt the frit at 1500 for 6 hours.

So if longer holding time out, longer melting time, essentially would ensure homogenous compositional mixing in the melt state. And what is expected that after you quench the melt, then you can get the glass plate with uniform composition which is fairly important for better properties.

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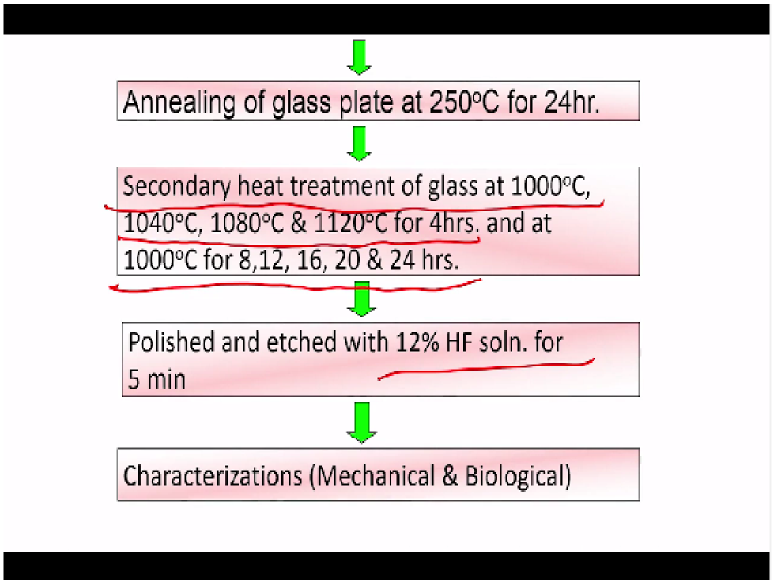

Now one can do this anneal of this glass plate at 250 degree Celsius much lower than melting temperature for 24 hours. And just to release any or just to remove any residual stresses in the glass plate, and then after that, we have done the secondary heat treatment of the glass at 1000 degrees Celsius or at different temperature, 1000 to 1120 degrees Celsius for 4 hours. This is one batch of glasses.

Another batch of glasses we have done with increasing time, like 8 hours, 12 hours, 16 hours, 20 hours, 24 hours but at fixed temperature 1000 degrees Celsius. What I am trying to point out here that this glass ceramic materials, they are extremely interesting material because like metallic materials, you can schedule different heat treatment conditions to get a variety of microstructures.

And often those microstructures are really fascinating in nature. And we are going to see some examples of the fascinating microstructures what one can obtain by changing this heat treatment conditions, okay. So to summarize, we have done 2 type of heat treatment. One is for different temperatures but constant heat treatment time of 4 hours. Another one is the constant temperature but different heat treatment time.

So by different heat treatment time, with increasing heat treatment time, one can expect that some microstructure characteristic phase would course in and that is, that may happen. Then you can polish and etch with 12% HF solution just before the microstructures characterization, okay.

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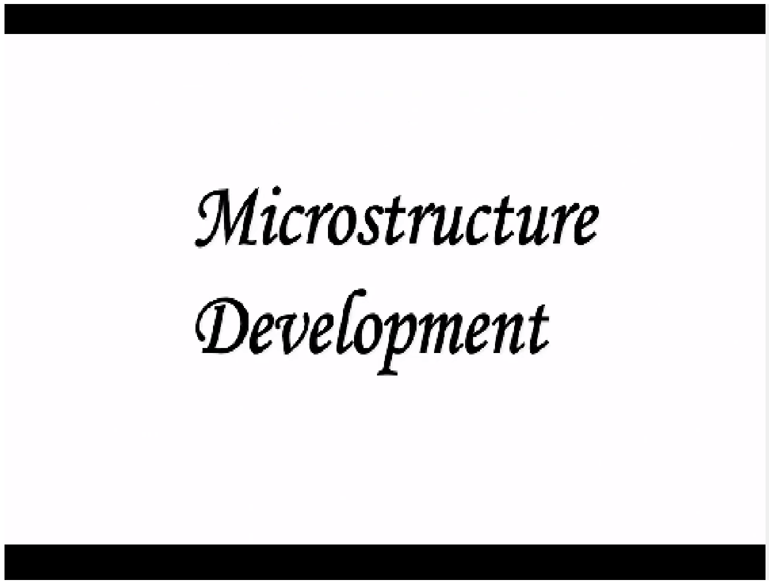

So that we have done.

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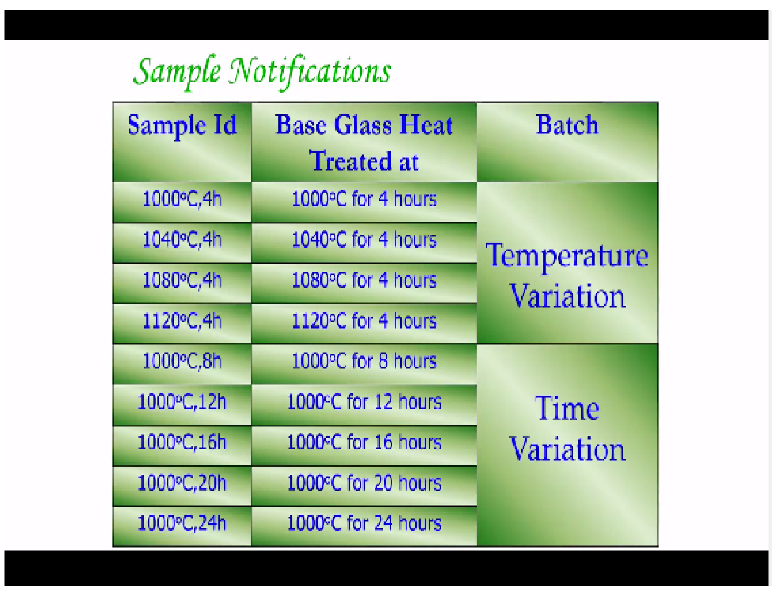

Sample Notifications

Sample Id	Base Glass Heat Treated at	Batch
1000°C,4h	1000°C for 4 hours	Temperature Variation
1040°C,4h	1040°C for 4 hours	
1080°C,4h	1080°C for 4 hours	
1120°C,4h	1120°C for 4 hours	
1000°C,8h	1000°C for 8 hours	Time Variation
1000°C,12h	1000°C for 12 hours	
1000°C,16h	1000°C for 16 hours	
1000°C,20h	1000°C for 20 hours	
1000°C,24h	1000°C for 24 hours	

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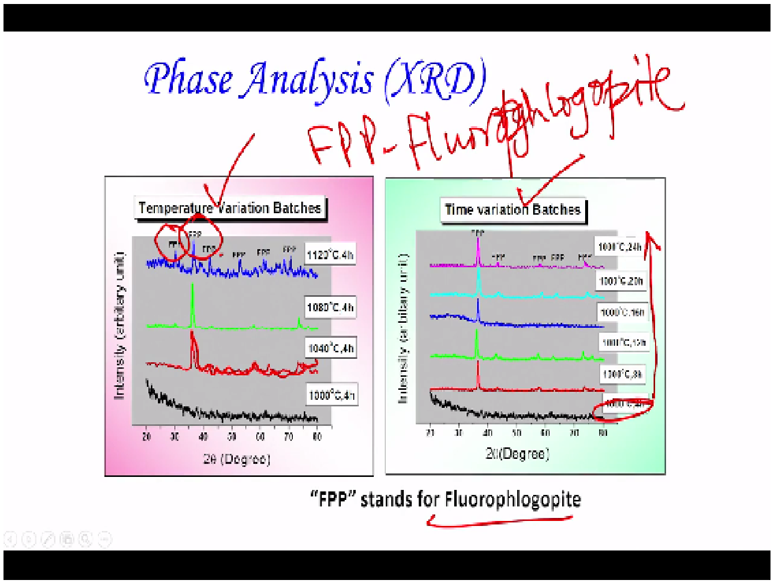

So now let us see that what are the phases that are formed, phase composition. In the temperature variation batches, we have got this fluorphlogopite that FPP. FPP, this is that one of the important phase, fluorphlogopite. So fluorphlogopite is, this FPP stands for fluorphlogopite and you see that it is a very crystalline peaks at 1000 degrees 4 hours, it is an amorphous glass. There is no distinct peak, okay.

So at 1000 degrees Celsius, 4 hours, you start seeing that some clear distinct peak of fluorphlogopite. 1120 degrees Celsius, you see that most of the crystalline peaks actually appear. What it shows that now this glass ceramic materials which is heat treated at 1120 degrees Celsius for 4 hours, it contains phase fluorphlogopite. In the glass ceramic materials, these are the 2 different glasses of materials, typically glasses means it is a completely amorphous phase.

There is no crystalline phase. Glass ceramics means glasses which contain dispersed crystalline ceramic phases. So when you are dispersing crystalline phases in an otherwise amorphous matrix, one could easily perceive that glass ceramic materials must have better mechanical properties in terms of hardness, strength and wear resistance. And we are going to see all these in the next few slides.

So this is for the temperature variation batches and this is for time variation batches. Again this is the XRD patterns at 1000 degrees 4 hours. It is all amorphous. And as we increase the holding time, you can see some of the peak intensity or some of the peaks they appear. But here you do not see that much difference what you have seen in the temperature batches. But mostly fluorphlogopite phase, they appear as we increase the holding time.

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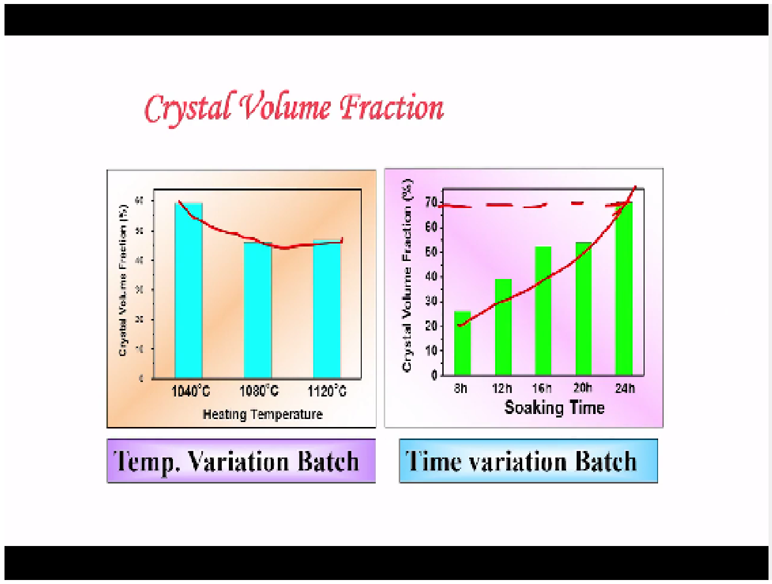

It is not only fluorphlogopite phase but also the volume fraction of fluorphlogopite also increases. For example, from 8 hours to 24 hours, you can see at 24 hours, fluorphlogopite phase is close to 70%. So 70% crystalline phase, 30% the glass amorphous. What you see a crystal volume fraction, it is 60% frictional volume present at 1040 degrees Celsius but when you heat treat at 1120 degrees, it reduces but it is still close to around 45 volume percent of the crystal phase.

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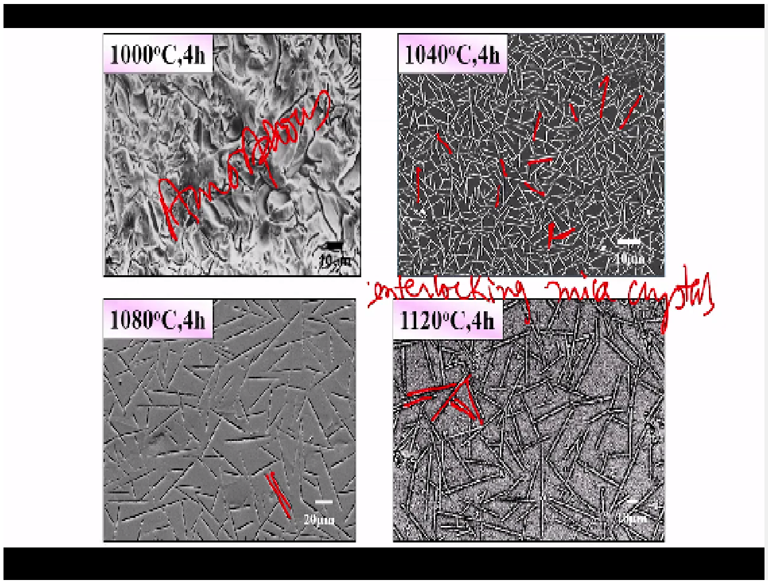

Now as I said few minutes back that you change the heat treatment conditions, you can expect different microstructures, that can evolve. So this is the clear case of the amorphous glass test. So you do not see any crystalline phases, okay. Now this is what in the typical mica crystals that form. Now what is interesting features of the mica crystals what you see here?

Now if you look at this, although this mica number is changed, so this mica number, again this mica crystals has changed. So this mica crystals, they are randomly distributed. There is no aligned growth of the mica crystals and often they are interlocked. Interlocking means, these mica crystals, they grow in this direction. This mica crystal grow in this direction. The moment this guy heats on to this growing mica plate, then their growth is stopped.

So there is kind of interlocking mica crystals, that is the best word. So interlocking mica crystals that develops in this particular microstructure. And one can also see that mica crystal can coarsen, that plate, mica crystal plate, that plate width can increase with increase in the heat treatment temperature. But while the holding time is the same.

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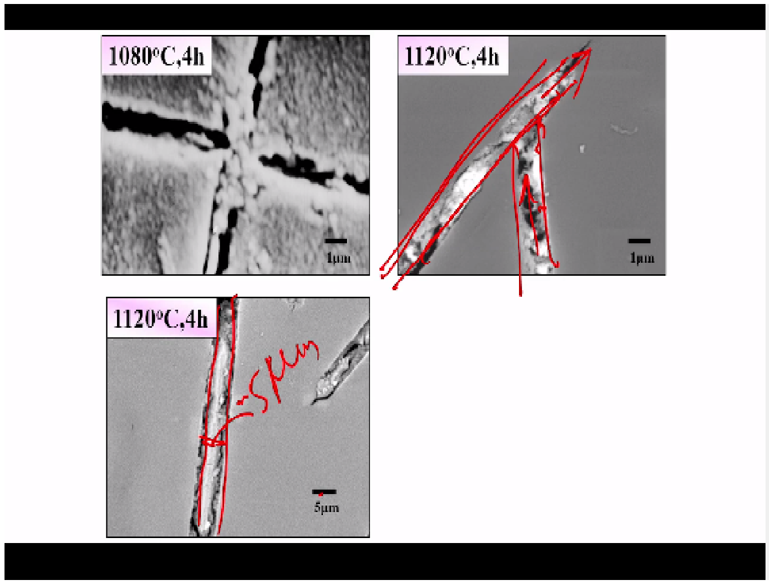

And you can see here, this is the typical, this is the 5 micron. So essentially this is also roughly around 5 micron is the typical mica crystal width. And this is the signs of this interlocking microstructures. You can see that these guy particularly is growing in this direction, this guy is particularly growing in this direction. The moment it heats, then this growth of this particular guy is stopped. And that is what is being shown in this particular SN images, okay.

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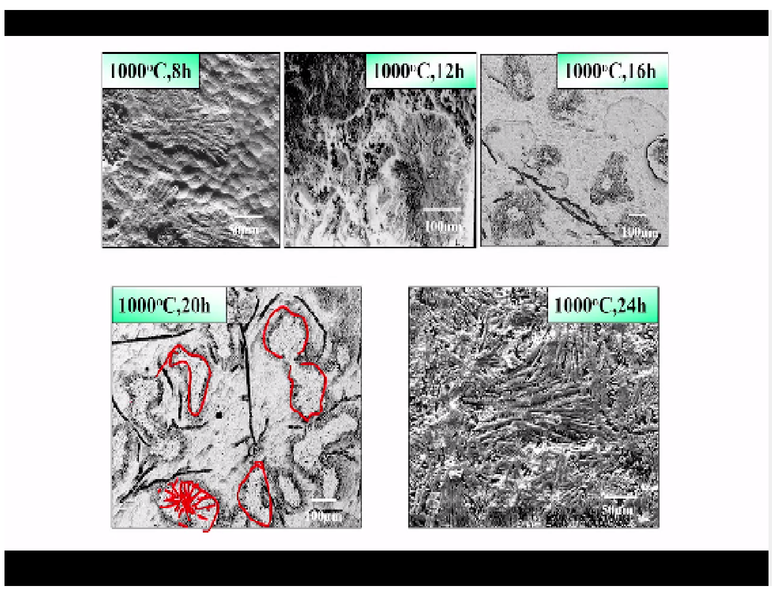

So by changing the heat treatment time but keeping the heat treatment temperature at 1000 degrees Celsius, what you see here that a different microstructures develops. What different microstructures? What you see that this is a butterfly type of microstructures. I will show you some more features. So there is a central nucleating points and these crystals then grow. There is a multidirectional growth and this multidirectional growth leads to a butterfly kind of structure.

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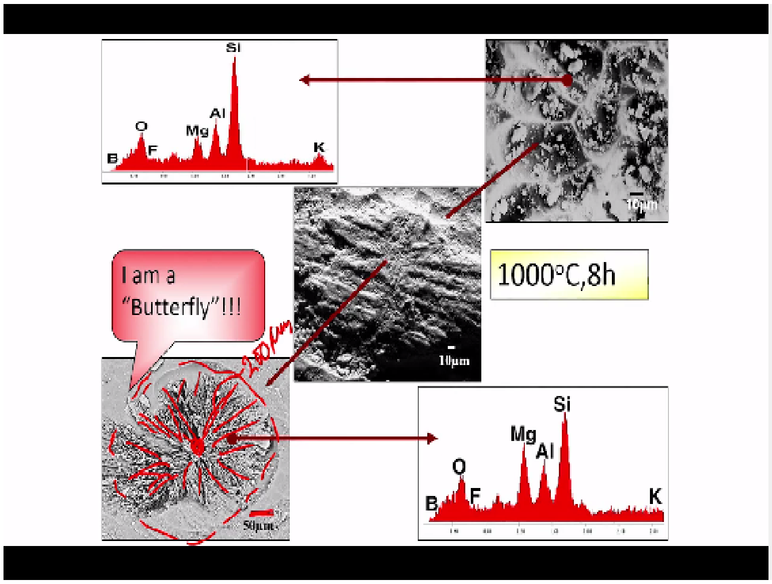

So this is one phase and this is also one of the butterfly crystals that forms at different heat treatment time, okay. Now what you notice here that this is the much more clear picture. This is 50 micron. So essentially if you see that this butterfly crystals, they have somewhere around 150 to 200 micron is the radius of this crystal. And this is a spherical kind of a crystal phase that develops.

And the spherical crystal that is the center. And from center, this different characteristic features of the butterfly that expand towards the edges of the butterfly crystal. This is kind of very fascinating microstructure features. People have reported different other kind of features like cauliflower type of features, crystal morphology in different kind of glass ceramics. But these butterfly kind of crystal structure that was something new when we reported in this particular glass ceramic systems a few years back, okay.

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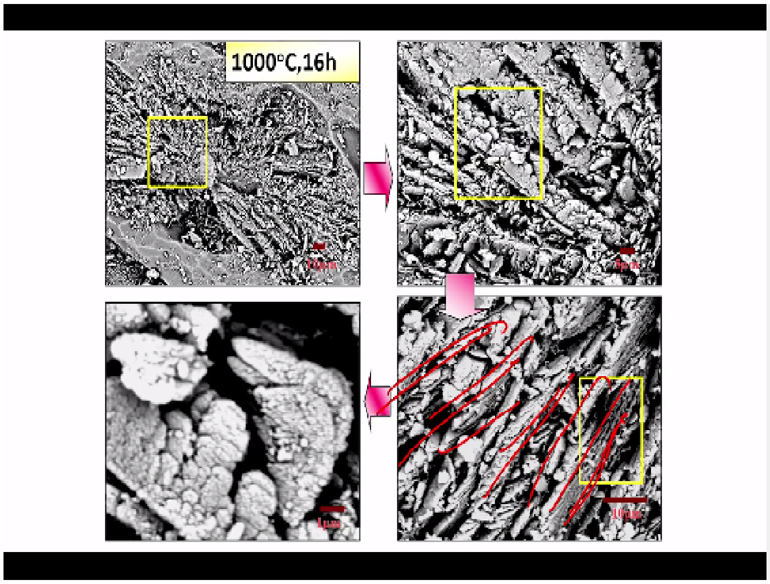

You will see the different kind of area of this butterfly crystal if we zoom it. You will see all these different features of the butterfly crystals. This is one of the features that you can see that how this crystal phases, they grow in different directions, okay.

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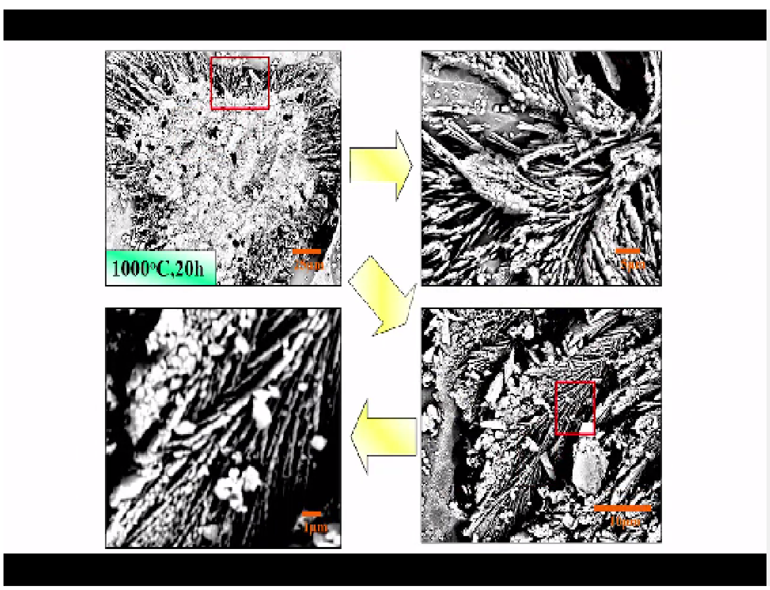

So these are like more observations that how these butterfly crystals, they grow when their glass ceramics is heat treated at 1000 degrees Celsius for 20 hours.

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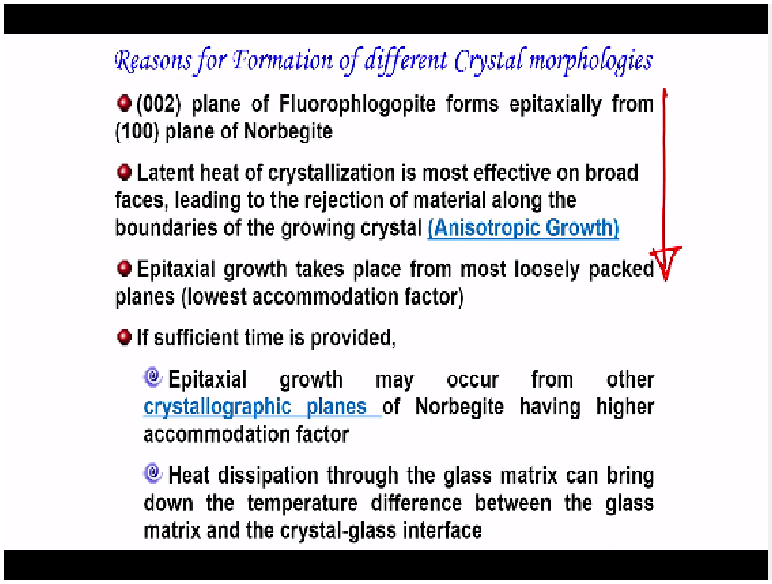

Now these are some of the underlying scientific reasons that how this microstructure develops. However, I think for this particular NPTEL course, this may not be very relevant. But just for you to remember is that if you change the heat treatment conditions, you can essentially develop that different butterfly crystal morphology or different type of morphology in this particular glass ceramic materials.

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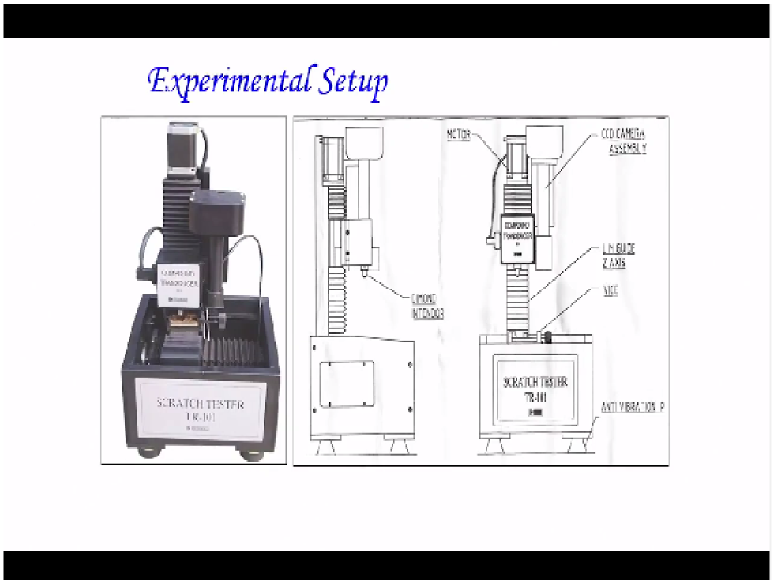

Now since this materials are potential dental glass restorative materials; therefore, we have also done not only fretting wear test but also scratch tester. So the scratch tester just to show that what is the scratch resistance in this material.

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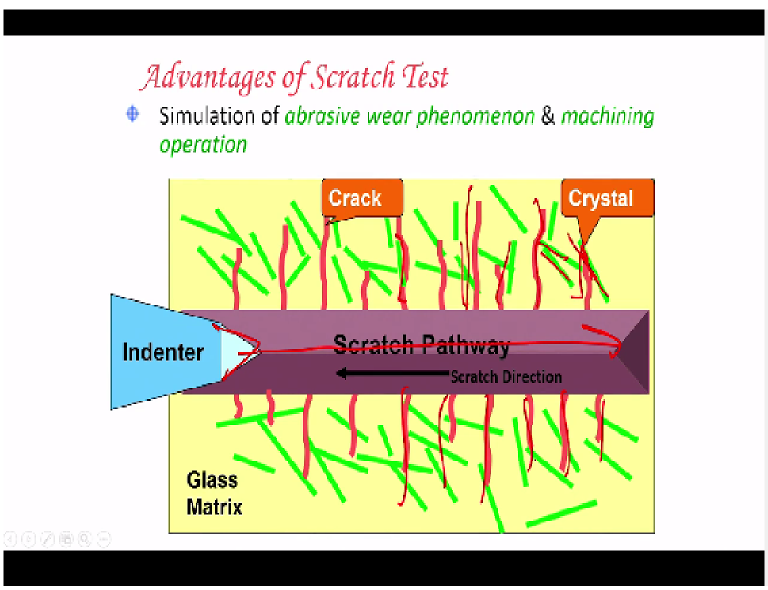

So you have an indenter and this indenter that will scratch pathway on this material. And there will be cracks that will be generated and you have the crystals also, the green color is the crystals and the red one is the cracks that is, what will be generated from the side of the scratch.

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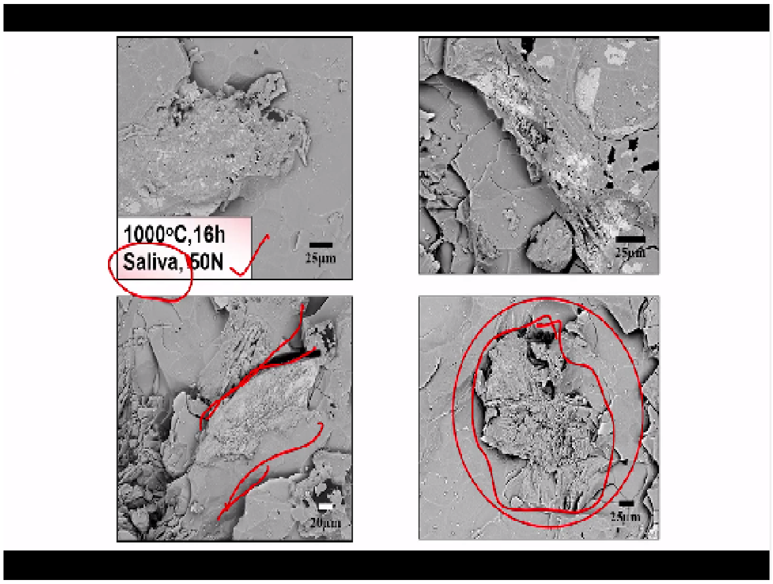

So essentially it simulates abrasive wear phenomenon and also to some extent machining operation. We have done the scratch test, not only in ambient conditions but also in artificial saliva. Why artificial saliva? As I said that we are trying to simulate the maxillofacial or aural environments.

So aural catalytic contents saliva but we have used an artificial saliva which we prepare in the laboratory and this is the case for 1000 degrees Celsius 16 hours sample and at 15 Newton is the load on the scratch indenter and then you can see very significant cracking which is generated in this glass ceramic materials, leads to spalling of this blur butterfly crystals in this microstructure.

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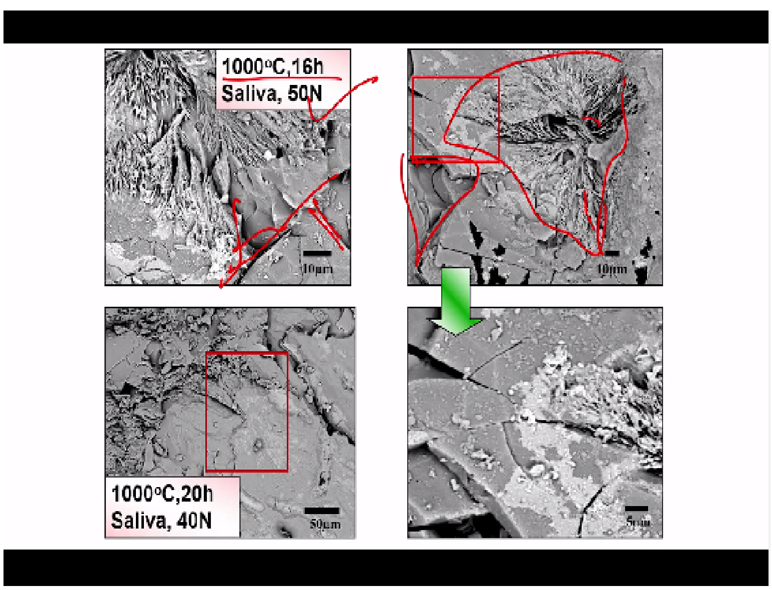

The same thing we have seen that very deep cracks which is formed along the several, around different locations in the butterfly crystals when we have done the scratch test on different materials. And you can see that there are already some materials spalling off and there is a deep cracking which can potentially lift off or spall off these particular butterfly microstructure. You can see very clear evidences of cracking in most of the microstructures regions whereever I am showing in this particular slide.

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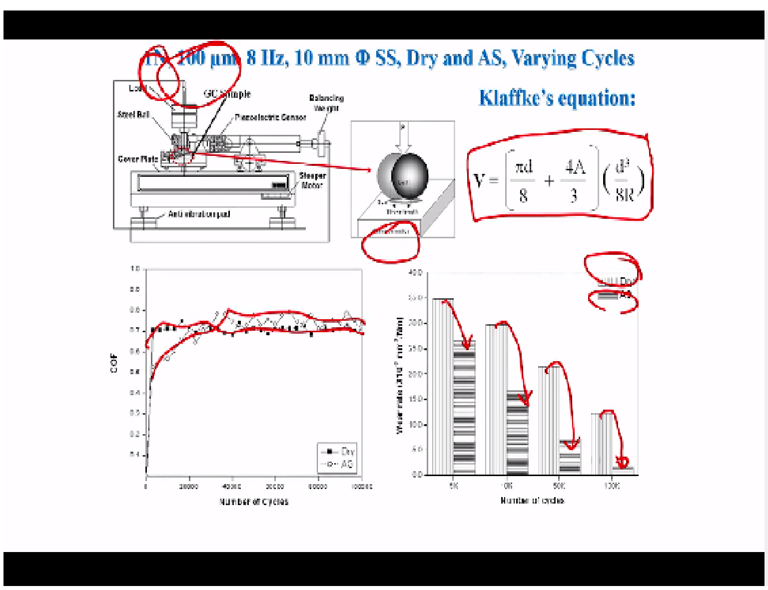

Now coming to tribological studies, like in the case studies we have shown before, we have used that fretting wear tester. And then in this particular case, we have used the glass ceramic as the flat material and stainless steel as the ball material and it is 10 mm stainless steel. In order to simulate the masticatory action related loading conditions, we have used that 1 Newton as the normal load and 100 micron is the reciprocatory sliding amplitude and 8 Hz is the frequency.

And what you see here that one can calculate that what is the volume, wear volume by this particular equations. So in terms of the dry and artificial saliva, we did not find much difference in terms of the frictional properties. So the frictional properties are very fairly comparable. In case of the dry, it is slightly lower but this difference is within the window of the standard error, standard deviation.

But we do notice reduction in wear rate when you conduct this test in the artificial saliva. So what you see here at 5000 cycles, we see there is a drop in the wear rate. At 50,000 cycles, this is a further drop in the wear rate and 1000,000 cycles, again it is the further drop in the wear rate, okay.

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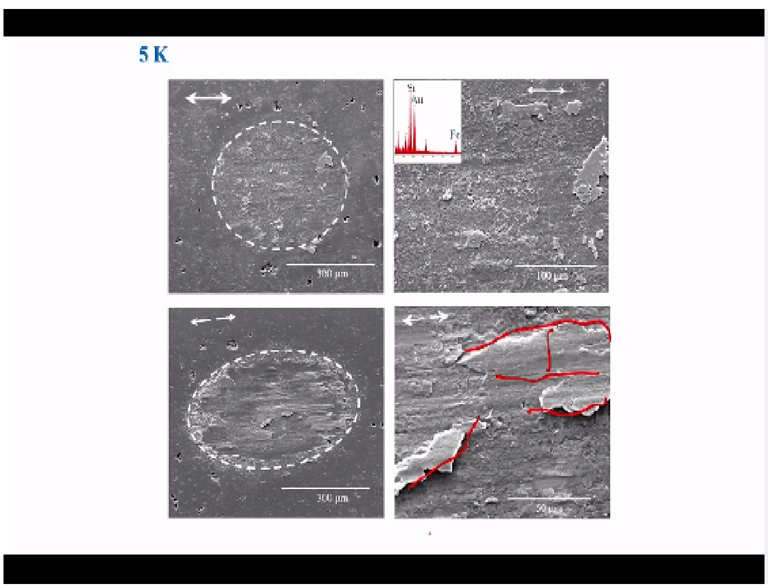

Now since establishing the wear mechanism is one of the kind of, one of the major motivation for many material scientists. We have done this particular, this is the scanning electron microscopic images of these materials or these worn surfaces. And what you notice here that even after the test is done for 5000 cycles, there is very clear signs of the delamination, subsurface cracking as well as the cracking on the top surfaces, top worn surfaces. All these feature are very clear after the 5000 cycles.

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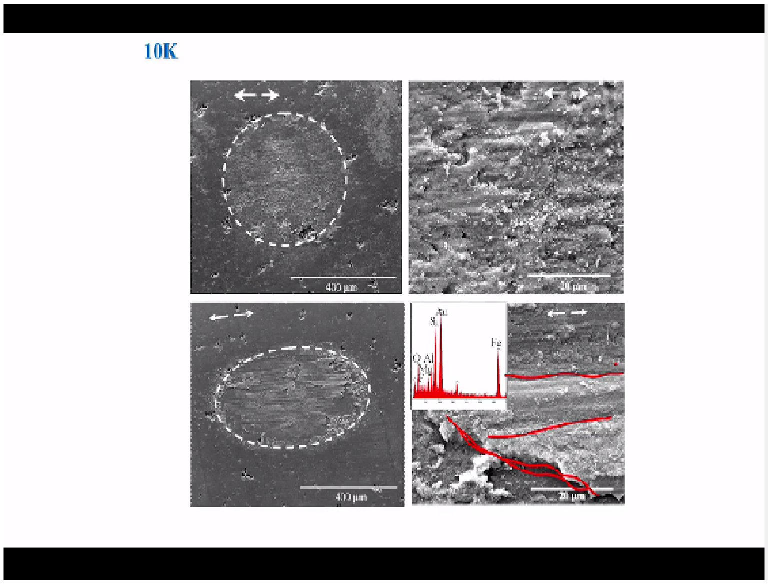

And as we go, increase in the sliding conditions, sliding duration, you will see that severity of this cracking is even more. So this is the deep subsurface cracks which is clearly visible and there is a deeper abrasive scratches also one can clearly notice along the fretting directions. So all these features essentially contribute to the severe wear rate of these materials.

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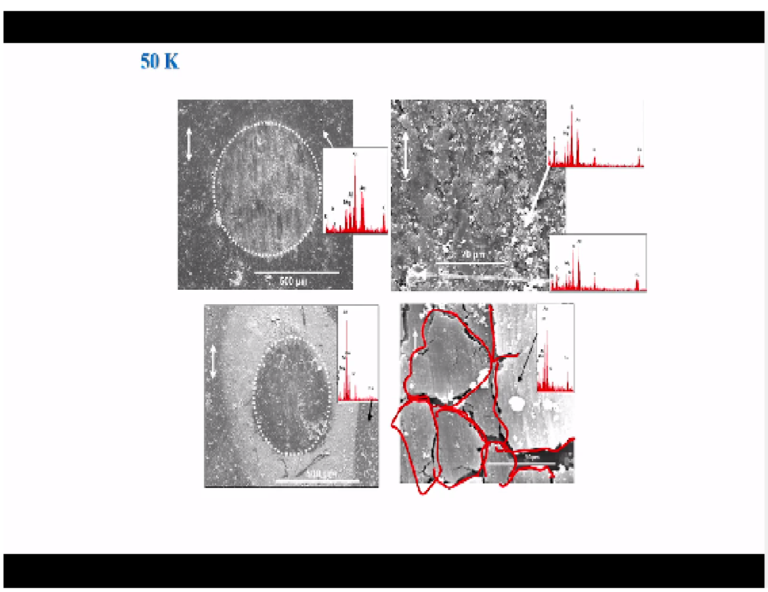

In case of 50,000 cycles, you will see that this wear tribological, wear worn surface, or worn surface also contains very significant cracking but here there is a very clear signatures of the tribochemical wear and these tribochemical layer, it is cracked very significantly and then of different parts of the tribochemical layer is kind of on off.

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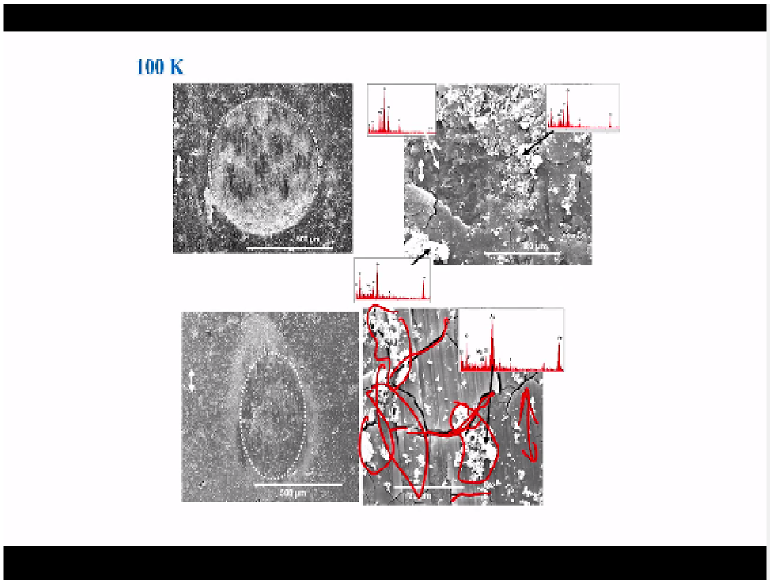

At 100,000 cycles, you do see that wear debris particles in different contrast and these wear debris particles, these can lead to the three-body wear situations also. And we can also see very deep cracking, mostly it is in the perpendicular to the fretting directions and these deep cracks, they are kind of responsible for causing extensive spalling of this tribolayer.

So overall what I have observed, what I have demonstrated that this glass ceramic materials which are being developed for dental restorative applications, these glass ceramic materials can undergo wear in artificial saliva environment to a much lower extent than what one can measure in the ambient conditions. So these glass ceramic materials therefore can be very potential dental restorative materials.

However, what I have not shown but we have published that results is that these glass ceramic materials also support the cell adhesion and proliferation of the osteoblast cells. Osteoblast means that is the bone forming cells and also they have good antibacterial properties. That means they have bacteria acetal properties.

Bacteria acetal properties again, some of the pathogenic strains like Staphylococcus aureus, Staphylococcus epidermidis and so on. So all these properties together make this glass ceramic materials, machinable glass ceramic materials as a material of choice for dental restorative applications. Thank you.