Advanced Topics in the Science and Technology of Concrete
Surendra P. Shah
Northwestern University, USA
Performance of Fiber Reinforced Materials: Historic Prospective and Glance in Future

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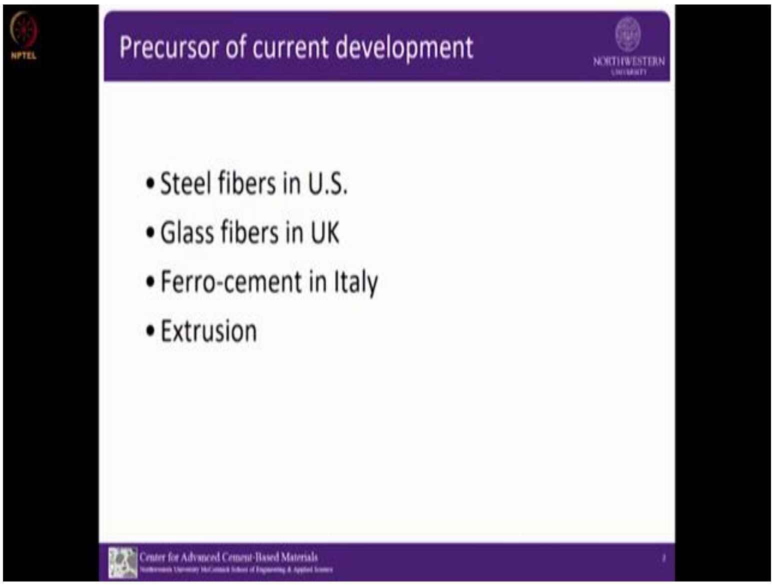

I want to talk about in general fiber reinforced cement-based composites and I want to give some historic prospective where we are today and where we could be going and after the Rilem conference, what are the basis of current development. So, it is essentially a fifty year history and why a fifty year? You will see that in my first paper on fiber reinforced concrete was published in seventy one. So, what are the precursors of current development? I have identified four started with steel fibres in US, then glass fibres in UK, primarily to replace as asbestos fibres in cement board, then Ferro cement in Italy by known architect in a near Pier

Luigi Nervi which is precursor of currently textile reinforcement and extrusion, which is can be considered precursor for 3-D printing.

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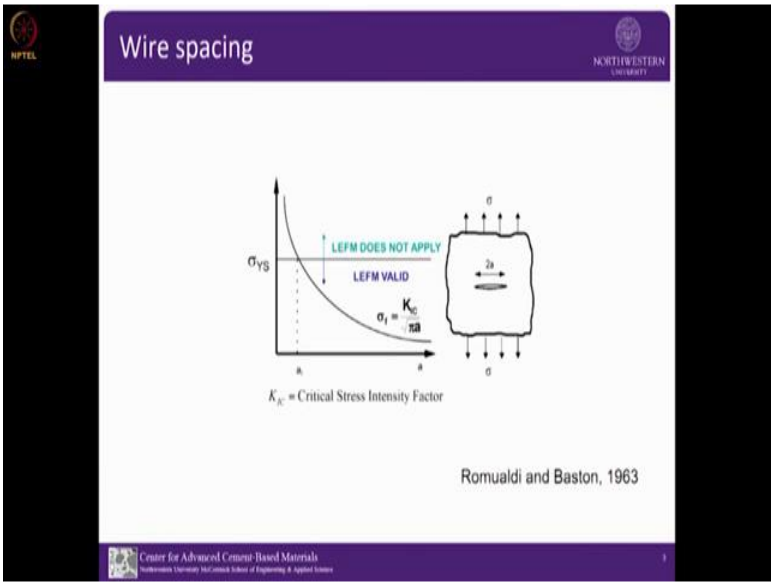

So, let me first start with the development of steel fibres in US and the concept behind the development according to Romualdi and Baston they were at Carnegie Mellon is that concrete is a brittle material, in a brittle material obey the Griffith fracture criteria that is fracture strength is inversely proportional to flaw size, smaller the flaw size considerably higher fracture size. So, they said that if you use fibres, randomly distributed fibres they can be closely spaced, you can increase the tensile strength.

So, very intriguing idea and when they published this, their paper I was doing my Ph.D. at Cornell and I was not quite interested because I had taken courses in fracture mechanics when I was in Lehigh University and I looked at the paper and it turns out in order to prove their concept. They tested reinforced concrete beams which was with wires and they were under reinforced, so, as soon as the crack happened the bar yielded. To get same volume but closer spacing, they have to use thinner and thinner, smaller diameter wire .And when I looked at it smaller the diameter of the wire higher the yield strength.

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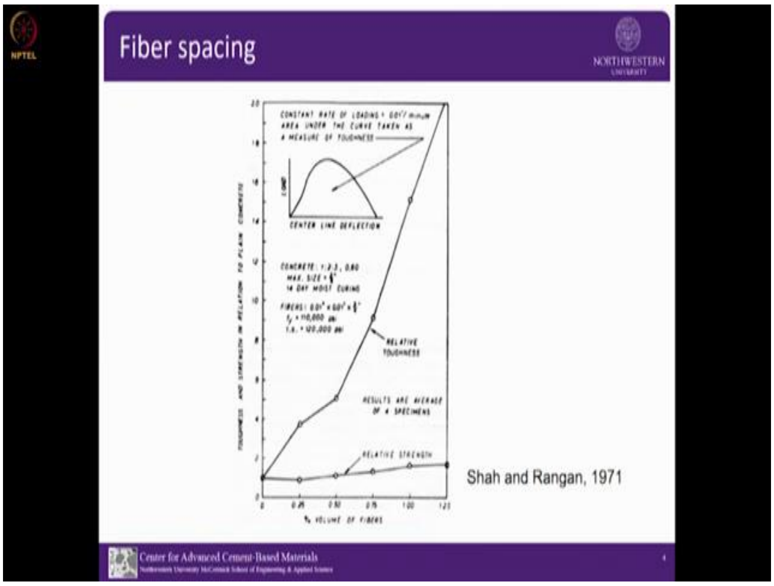

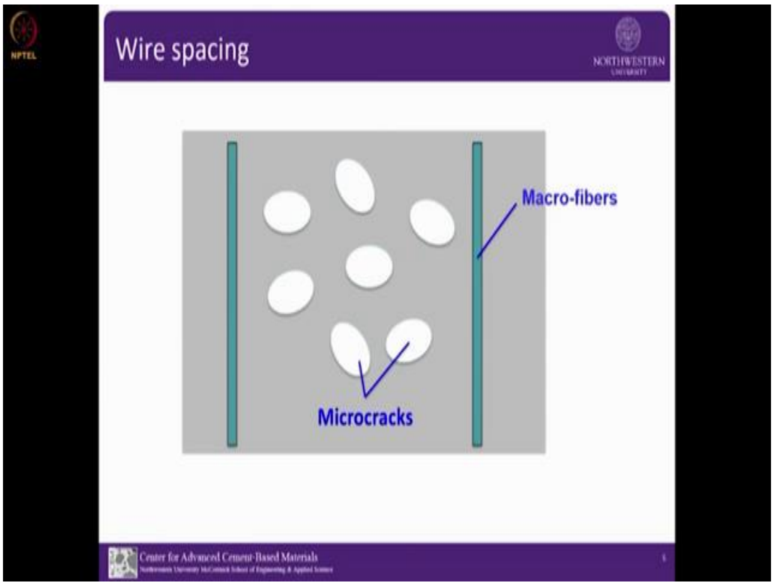

So, when I normalised the result with yield strength, it turns out there was no effect of spacing. Then that got been started and then we did some work with randomly distributed steel fibres. Here you can see I have percentage of fibres and of course more volume of fibres they would be closely spaced versus tensile strength and then what will called toughness is the area under the load reflection curve and this was the work I did with Vijay Rangan, who is now retired from Perth. And you look at it and see there is really no increase in tensile strength but there is a substantial increase in toughness and in fact if you look why is that? One of the reasons why there is no effect of spacing because at the measured scale the crack starts at the micro level. So, this fibres which are macro fibres in millimetre range and we are talking about 1 percent more or less.

So, before the fibers can interact with crack, micro cracks have already coalesced into macro crack. So, tensile strength is already formed then they will constrain crack opening or crack width and toughness. So, that is really the basic idea about the currently used fiber reinforced concrete.

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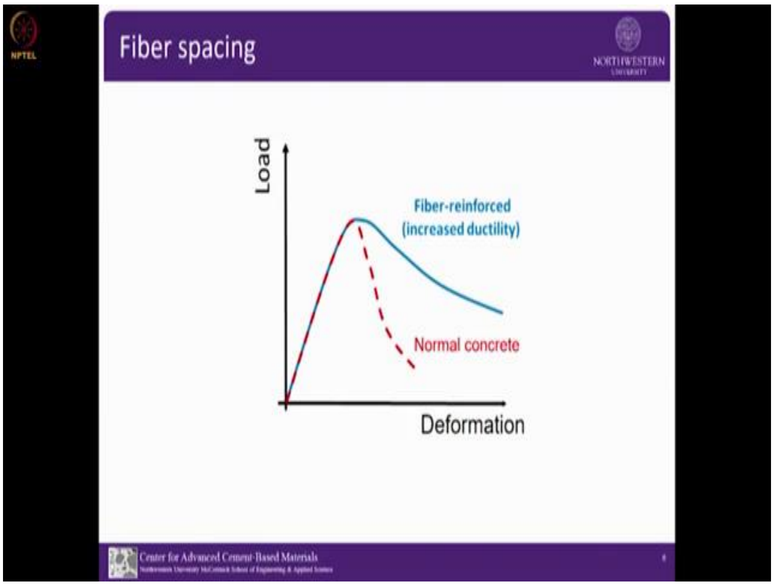

So, if you look at the fiber reinforced concrete whether it is in tension or compression, load versus deformation, we know that normal concrete in compression or tension is characterised with the strain softening, very important part in compression especially and in tension. So,when we have fibres, I am talking about a percent, it would not change the modulus, generally it doesn’t change tensile strength but it does change substantially the post peak. So, crack control is the main reason now we are using in a micro-fibres, conventional fiber reinforced concrete.

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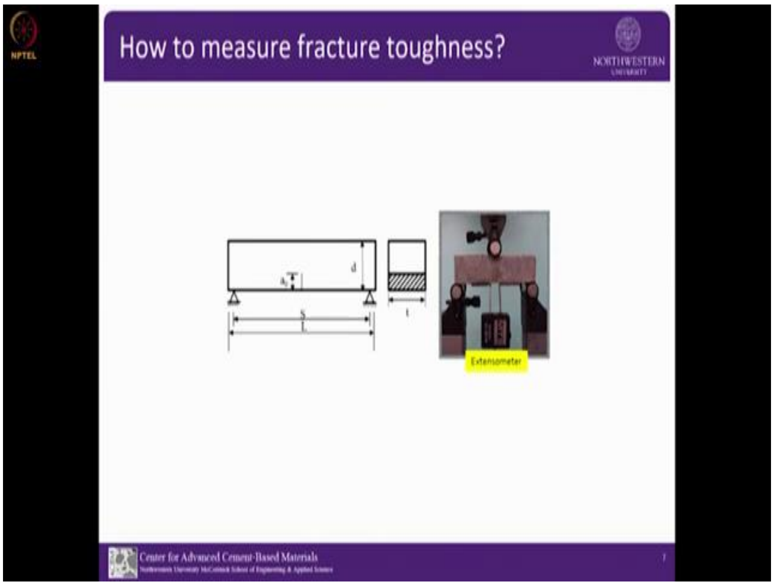

The toughness becomes important, so, how to measure fracture toughness? For linearly elastic fracture mechanics they are ASTM test, you have notched beam and then you apply the maximum load, apply the flexural load and when it reaches maximum everything is elastic and from the peak load and this crack depth, you can calculate fracture toughness. But a lot of work has been done and it turns out that fracture toughness concrete is not quite brittle, we call it quasi brittle.

So, crack propagation before the peak, there is so-called fracture process zone, you cannot use LEFM and so there is a lot of work done, I think that professor Gettu has been also involved. And now I think people conclude the best way to get experimentally the fracture toughness to have these notched beam test and you test it in a crack mouth opening displacement and when apply the load with a CMOD control rate then you get entire load deflection curve and from there you can calculate fracture toughness. So, this is a really accepted way Italian have a code based on this test.

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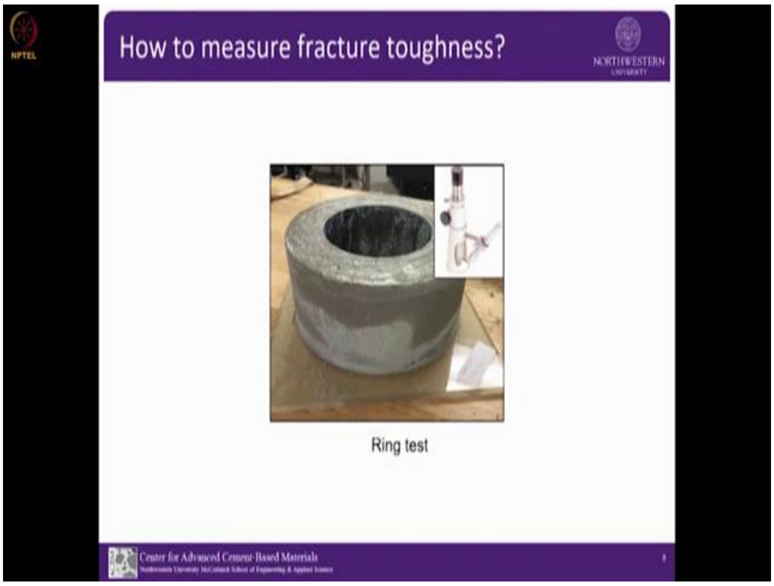

Now not too many people can have closed-loop testing, when IIT madras has it but many people don't. So, you need a method which is somewhat easier to conduct then the CMOD control. So, we were developed this so-called Ring test. In a Ring test you have a concrete here and there is a steel ring which is relatively rigid. So, after curing you let put concrete into drying, so, concrete wants to shrink still says no, so, you develop tensile stress and because of axis symmetry, it's like infinitely long tensile specimen. So, you find out when the crack starts and then using microscope how wide the cracks are. So, this test now has been standardise by both ASTM as well as highway department in US and it is a good way to have a crack potential of a material, evaluate different types of fibres, for example we showed that, when you make a highly strength concrete and when you compare it with the normal strength, high strength would have a higher tensile strength but because it is not brittle, it cracks sooner, earlier than normal concrete.

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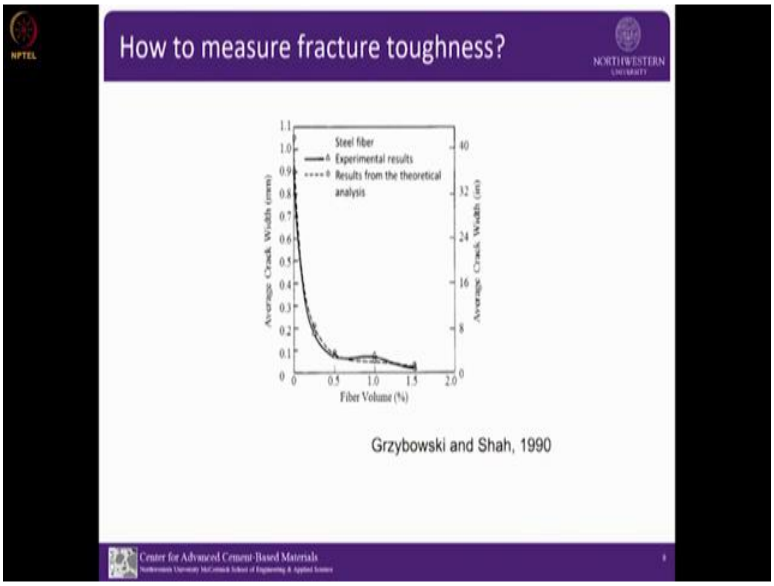

So, this is a very good test and to show how it works for example I have shown you some results with steel fibres reinforced concretes, here is the volume of fiber and there is average crack width and you can see it is small half a percent of fiber volume substantially reduces crack width and this is why we are using fibres reinforced concrete for crack control.

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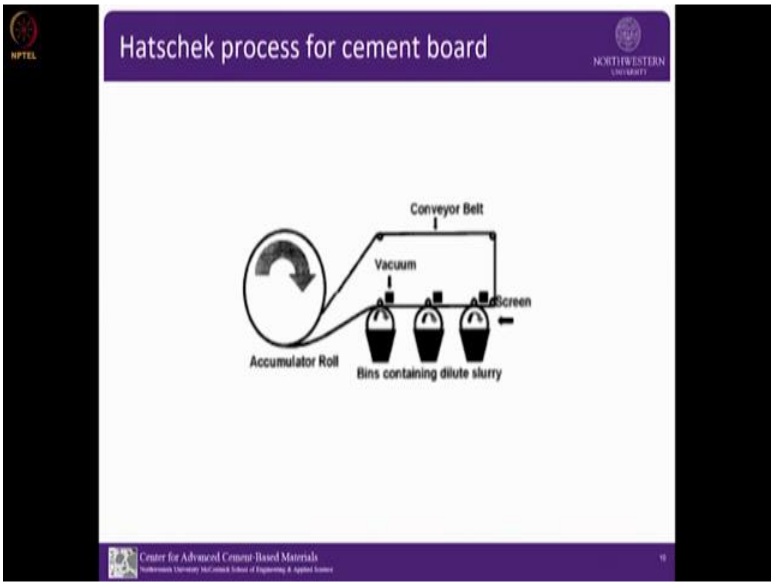

When you cast concrete, those of you have done experiment know, if you increase volume of fiber workability is the issue, if you increase the length of fibres workability of issues. So, this is one of the reason why with the cast concrete most of the application of fiber reinforced concrete, we are talking about 1 percent and aspect ratio not too much larger than hundred .So, people are looking at alternate methods of production then cast-in-place, one of them is called Hatschek process which was developed in Australia and it's widely use for so-called cement board. So, cement board is, you have very dilute slurry originally with asbestos fiber and then layer by layer it's deposited.

So, this is in fact the first example of so-called adaptive manufacturing because you have layer by layer manufacturing and you get cement board. It turns out that asbestos could cause lung cancer called as asbestosis, so many countries have allowed ban. So, people have been looking for alternate. So in England they came up with alternate of glass fibers instead of asbestos fibers, the pioneering work done by Majumdar at building researchers institute in England.

Now when we use normal glass there are issues with the Alkali reaction of E-class, so they develop adding zirconia called Alkali resistant glass fibres and it turns out that in this applications glass fibre really did not work this processing. So, now for Hatschek process people are using cellulose fibres instead of asbestos , cellulose fibres for green strength and then for reinforcing either PVA or polypropylene. But chopped glass fibre simultaneously spraying with slurry of cement and glass fibre has become quite popular and it called GFRC, glass fibre reinforced concrete.

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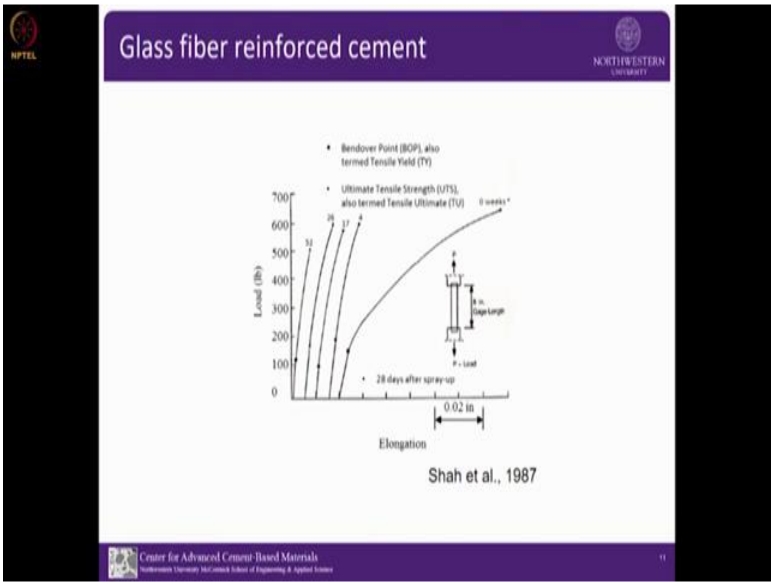

And let me show you some work we did with the GFRC. So, when the alkali resistant glass fiber was developed to test thatit doesn't corrode in the high PH, they do a test with a glass strength surrounded by, higher calcium hydroxide solution and then measure it to see whether the tensile strength with accelerators work. So, it is called SIC and they showed that alkali resistance glass fibre in this test works very well but when you make composite out of it with the glass fibre chopped, it turns out that because calcium hydroxide can go into the filament and make the glass fibre brittle even alkali resistant glass fibre with the right environment can become brittle.

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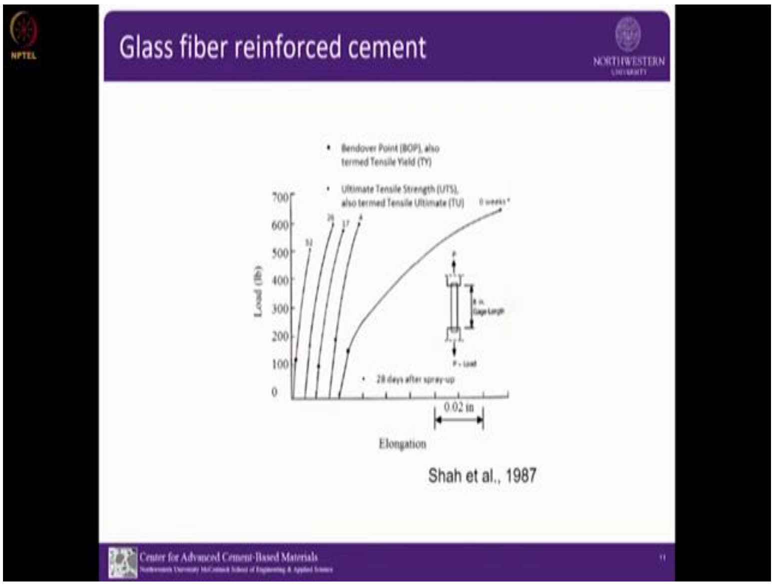

So to show that we did this work and I think this was sponsored by Saint Gobain which also you are working with. So, if you look at the twenty eight day, this is sprayed up glass fiber reinforced, you have a linear behaviour, people often call this bend over point and then you are this nice multiple cracking and strain hardening response and this is you know four to five percent of glass fiber. So, with this technique you can use longer glass fibres and higher volume, a great response. Then we subject that to this accelerated, I think about 60 or 65° in limewater and you can see that there is a substantial degradation.

So, the important point, even though with single SIC test, there was no degradation in the composite there was substantial and because I said that glass fibers, which is made with different filaments calcium hydroxide goes in and becomes embrittle. Then there are other ways to solve this you can use a pozzolanic material, so that there is less lime but this is important to know I know that I had talked with one of you who is working on textile and I mention that he must also test the composite to see whether you have durable results.

Now it is different with a continuous fiber rather than chopped because when the chopped fiber are inclined then they can break, so you might have a different mechanism with that. The two alternate that, then cast-in-place that I mention, one was the hatschek process and the second was this GFRC where you spray glass fiber and cement and both would allow longer fibres and higher volumes, so you can get this strain urging response.

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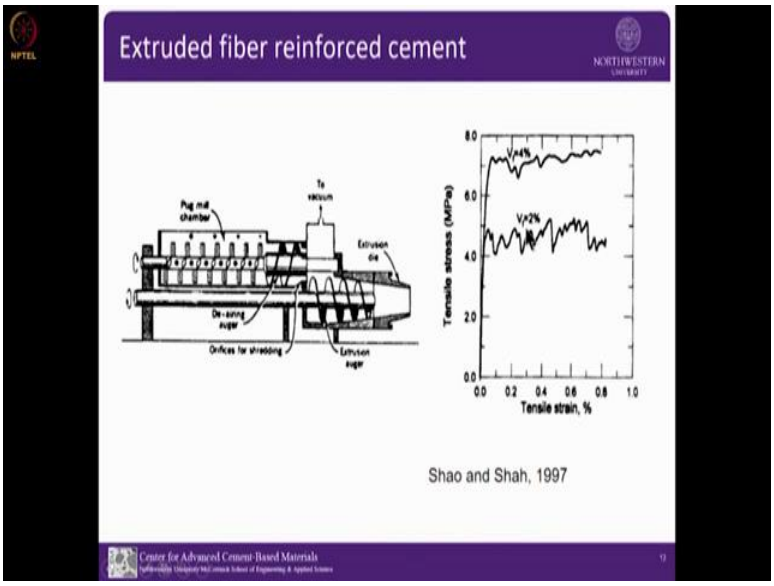

Well third way which is extrusion and I talked about extrusion because it is precursor to 3-D printing and we developed this in 1997, so this is a screw type extrusion where you have fiber reinforcement material looks like dough and you push it through a dye and depending upon the shape of the dye. And the fibres we used here are I called micro fibres, so they were PVA fibres, 14 micron in diameter and rather short, 2 mm long and you can see that we got 2percent and 4percent ,we got a higher tensile strength because fibres can now interact with micro-crack and if you design it right you get a strain hardening response because it is essentially like a ductile.

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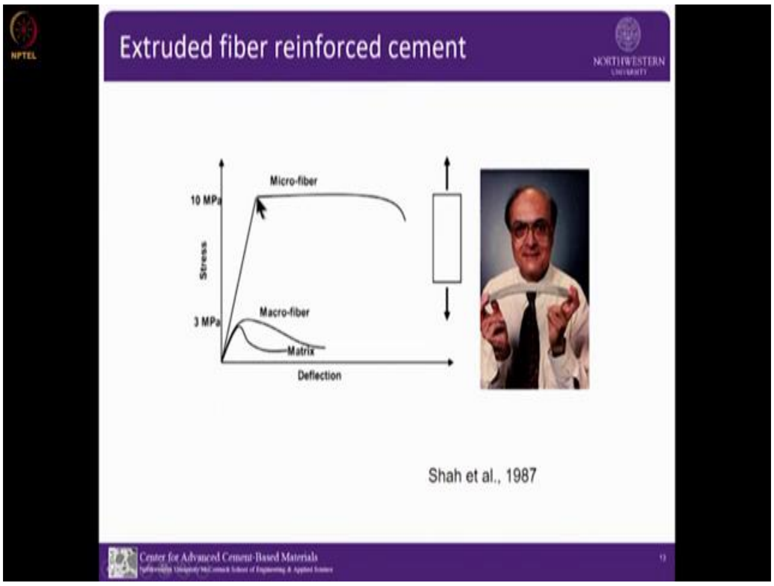

So, this to show that I often during that time I show this to my class, you can see from the hair it was a long time ago 1987, so, this is currently where we are in fiber reinforced comparison. So, most as I said some of might macro fibre is still the most commonly used commercially but people have been working with this so-called strain hardening cement composite and here is for example one of the that what we did which is a 10 Megapascal and strain hardening response.

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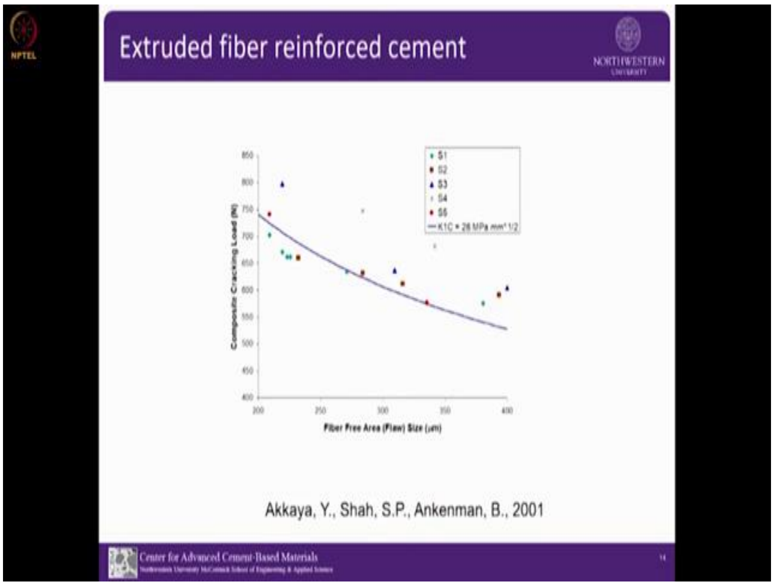

What we found out if you recall I told you that with the effect of closer spacing did not work with the macrofiber but with the small fibers we have now, so what we did, we had these all the material had the same volume of PVA fibres but because of dispersion other reason, we got different results and so we cut the specimen and using SCM calculated what is the fiber free area and assuming fiber free area is the flow size. So, here is we calculated the fracture composite versus fiber flow size and we did get somewhat similar to fracture in Griffith fracture criteria. But the flow size has to be in a small enough range and fibres have to be in the range size that they can interact with the cracks.

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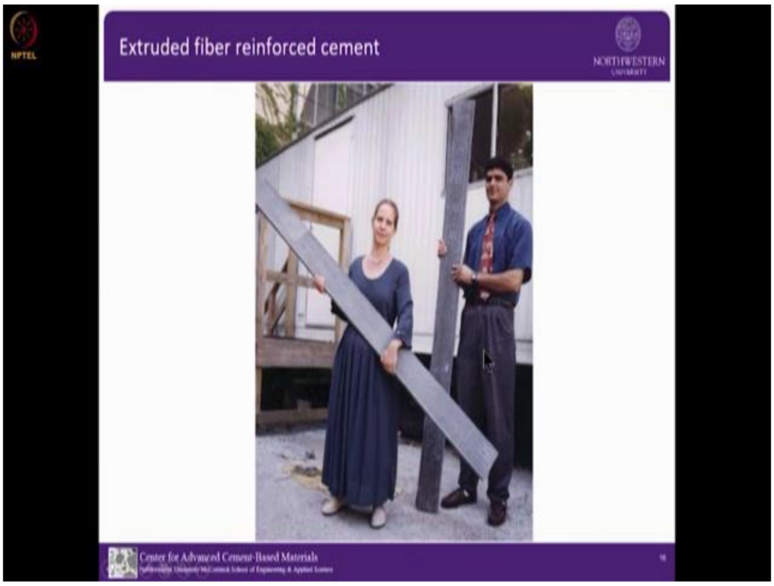

So, with extruded cement I showed you this, depending upon the dye you can manufacture sheets and as I mentioned so-called cement board with different fibres have been extruded in Japan, you can even extrude pipes or the cellular wall. And to show you that how light that can be, you might many of you know Alva Plight she is a professor, now at Israel but she was the postdoc with me and this is quite almost 2 meter tall cellular section and it is light enough that Alva can lift it and Elmas who is now a professor in Istanbul.

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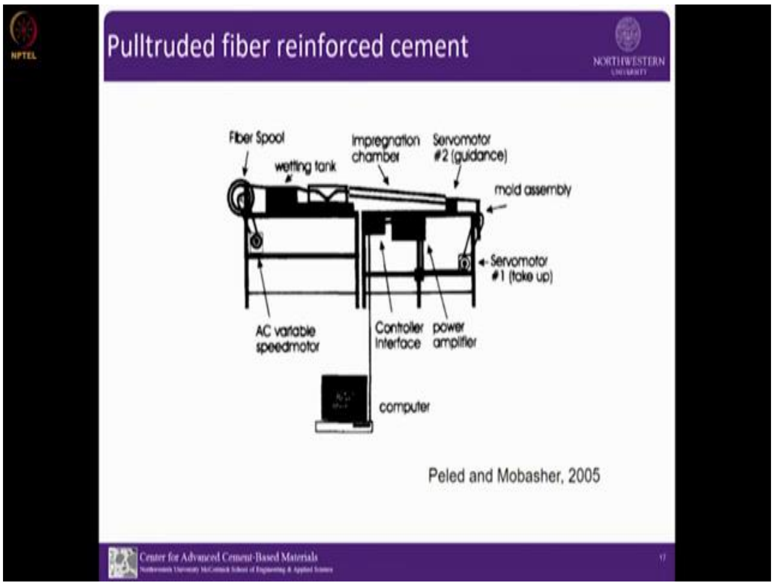

So, then the other ways to manufacture and one of the Alva and Barzin Mobasher are at Arizona state, there using this pulltrusion. Now pulltrusion is quite common for resin and epoxy type of composite, this is the first time the cement, you have continuous strength, it goes through the cement slurry and then you develop composites.

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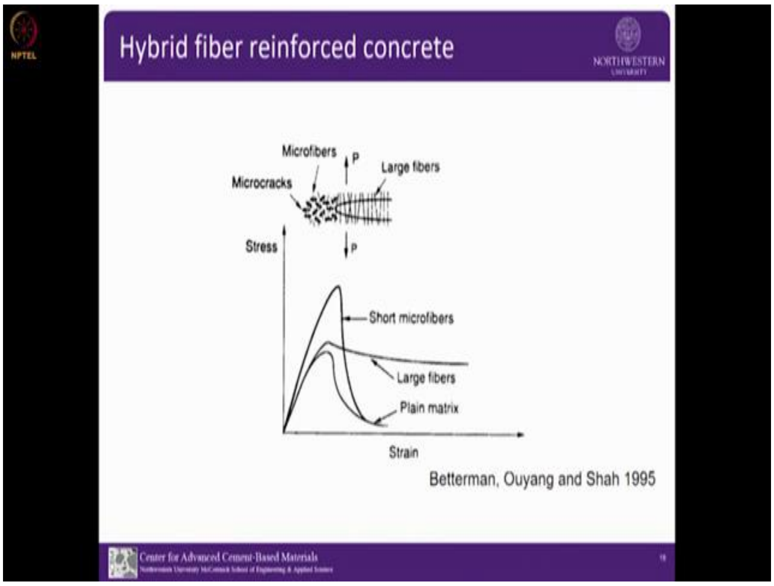

So, another thing which is people have studied quite a bit is so-called hybrid composite.In the hybrid composite, we know that short fiber can increase the fracture strength but they are short, so once the crack develop they do not have as much on a long-range crack controlling property. Large fiber can do it but they do not really increase the tensile strength. So, you can combine the two and produce the results which you have a very very fine crack width and also post peak ductility.

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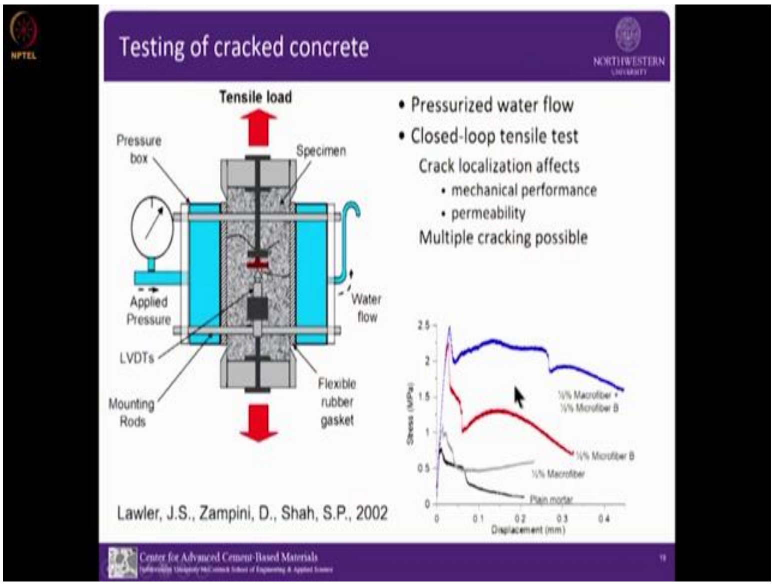

And to show you an example of this, we did a test, mortar specimen reinforced with fibres and subjected it to tension. So, there will be crack and we wanted to see what is the flow property because most of the time when we use fiber let us say as a layer, you want it to be protective of a rebar, so you want to make it as in permeable as possible.

Now if you use big fibres as I mention they will control crack but the crack should be wider, finer fibres the crack should be much finer and it also works with the mechanical property. Here is the stress displacement of plain mortar which is shown here and here is this half a percent of steel macrofibre and as I showed you it doesn’t really change much but a post peak. Now this is half a percent of micro steel fibres, we ask Bekett to make for us just the microfibre, I think it was 30 micron in diameter and then of course when you combine them you can get both high peak strength and strain hardening response.

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So, that is what I wanted to talk about hybrid. Now I want to talk about the precursor of textile reinforcement I know you have another people are working on textile. So, original idea was by Pier Luigi Nervy and he use wire mesh, several layers of wire mesh and intuitively he said that wire mesh will homogenize crack, so crack should be so fine that you can use the material in that range. And there are many examples of Nervy design, one of the most famous one is the opera house in Sydney, there were thin roofy material made out of ferro cement.

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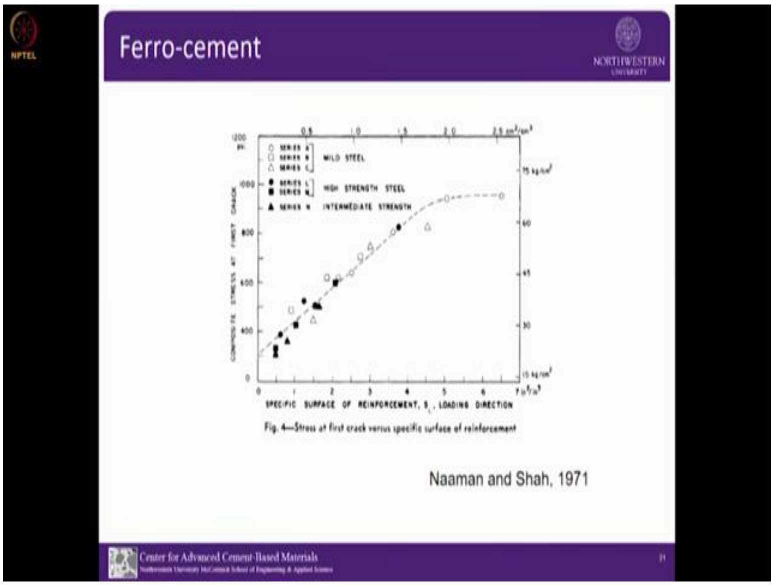

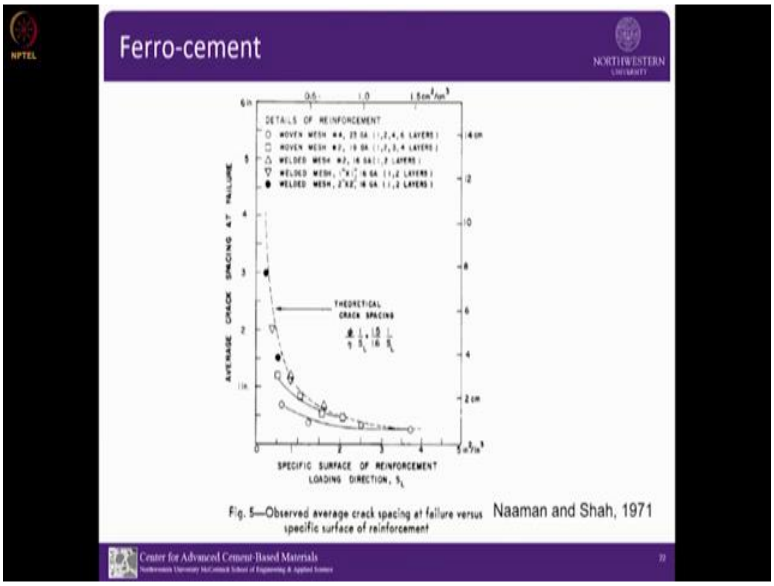

So, later on I was visiting professor at MIT and Tony Naaman he started doing his graduate work, I was in the materials group and Tony wanted to something with reinforced concrete, so we said okay let us look at ferro cement and see we can give some engineering qualitative reason why cracks are so fine in ferro cement. This was a paper published in 1971 and what here is plotted is the composite stress versus surface area per unit volume.

So, higher the surface area, higher the bond, you increase the time for first crack and that is important and then you also decrease the crack width. Here we should average crack spacing versus specific surface of reinforcement and you have more and more multiple cracks and more and more cracks for the same displacement obviously finer crack. So, that is the basis of ferro cement and now of course as you know instead of steel wire people have been using glass fibre cloth, synthetic fiber cloth as well as carbon fiber and quite a bit of work has been done in Germany with the textile reinforce with glass fiber.

So, this is the basic idea that with the textile you can have multiple cracking, strain hardening response and to the bond becomes a key point, even now with textile, let’s say with carbon fiber, bond is an issue. With a glass fiber the bond can be issue because of the coating.So, people have been as I found out at the conference in Dresden working on how to improve the bond with the surface area. And one of the reason I mentioned to somebody, we could check whether dipping the glass fibre cloth in a slurry of nano silica would have improve the bond because nano silica might but with the coating, I am not sure but it is many ways to do it .

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So, now with the development of self-consolidated concrete, we can use longer fibres and higher volume because with self-consolidating concrete material flows like water. So, that has changed the previous thinking that we cannot add more than half a percent or 1percent.So, we can add higher volume of fibres, longer fibres and that is led to what we called ultra-high performance concrete. So, ultra-high performance concrete has no aggregate, primary silica sand and compressive strength can be as high as 200 Megapascal, in fact ACI, American Concrete Institute defines the UHPC as minimum 145 Megapascal and sufficient ductility, it is very important because ultra-high performance will be very brittle almost like glass if you don't have fibres.

As a result of the development of fiber reinforce concrete and SCC, we have now UHPC and I just want to show you one, some very recent work I was involved with, this was at Hong Kong Polytechnic in Hong Kong. Where we use high-density polyethylene fiber, higher density, there were higher modulus and a longer, we can use longer length and finer diameter.

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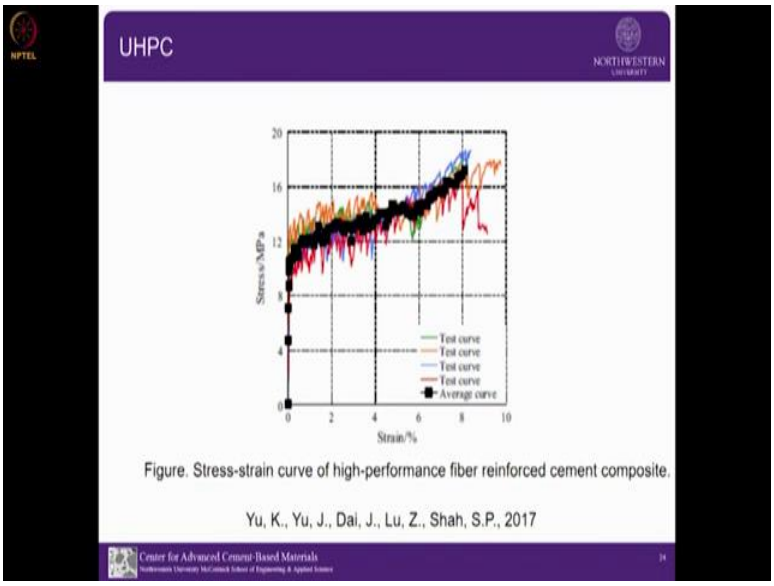

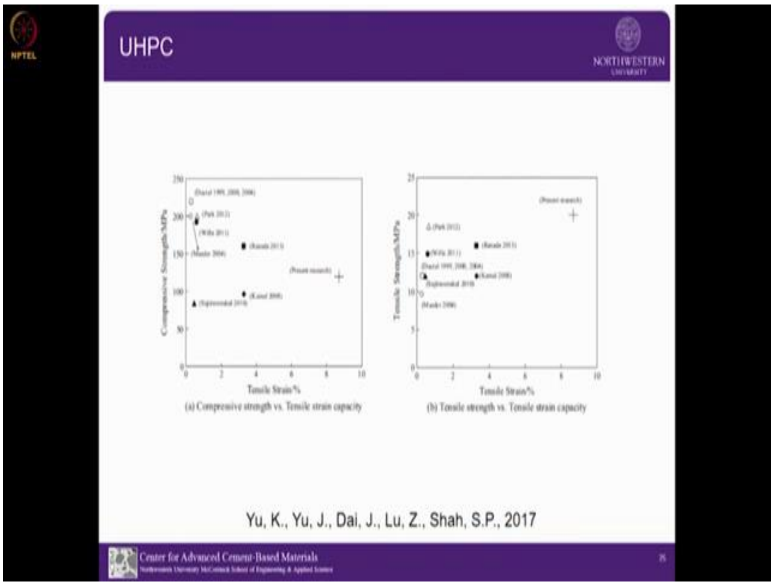

And you can see the interesting thing is that the strain here is 8percent, you know you do not except multiple cracking to have such high and a peak , this is in tension, peak tensile strength is as much as 16 Megapascal. Because of now we can control rheology, we can add 2percent volume of the fibers and I thought would be interesting, we also put that in paper. Here I have shown the UHPC compressive strength versus tensile strain because, we want high compressive strength and we want high ductility, so the work we did, we were not able to obtain 150 but it is more like a 120 Megapascal but considerably higher strain. And if you look at the same results, same a tensile strength versus tensile strain in tension because this is considerably higher compared to what has been done.

So, that is I pointed this out, that now really with the development of rheology and fibres, we can produce both strain hardening composite with relatively large strain and high compressive strength.

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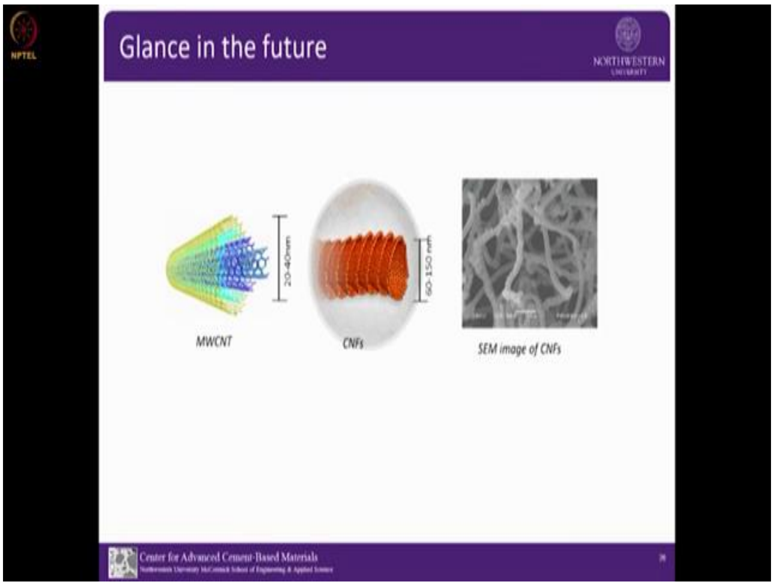

So, this is where we are as far as I would say let us say micro fibres and macro fibres, we have a convention fibre reinforce concrete, for example lot of work has been done, being done here with what happens in a crack concrete with time in creep and UHPC which quite a bit of work being done very high-strength concrete up to 200 Megapascal and 3 to 4percent of fibers, for example Lafarge has developed product they called Duque Taal, which is the same idea but now UHPC has been using in many applications. But the conventional reinforce concrete, reinforcing bar or prestressing they help structural ductility.

So, after the crack is formed reinforcing takes over and we get substantially higher load and ductility, with fibres the main emphasis is in crack control and if you have a UHPC then in some applications with very thin member, very high-strength you may not need conventional reinforcement. However we know cracks really start at the nano scale, the most brittle component is cement and the voids in cement capillary pores and wherein gel pores are at nano scales. So, people say why not also consider reinforcing it at nano scale and the idea is that if you really want super impermeable concrete. So, you have a ladder reinforcement, you reinforce, conventional reinforcement, micro reinforcement and reinforcement at nano level. so many people have been working on nano fibres primary carbon nano tube and carbon nano fiber.. People also have been working with graphite and graphene and graphene oxide.

We started working with carbon nano tube, many years ago. When I had a great student Zuvi Matax and she is said I have heard about nano and I want to work on nano fibres. I said okay. So, we started looking at, you know that time many other people, we were thinking of conventional reinforcement. So, we started adding 1percent or half a percent of carbon nano tube. First of all we found there very very difficult to make because we are talking about nano scale with huge surface area and results were not promising. It turned out that Zuvi by accident made a mistake and instead of 1percent she added 0.1percent and she founded at the 0.1percent at the best results compared to 1percent.

So, then sort of lights starts can be said, if you want nano material well dispersed then we have to use small amount. Then of course now most people are using very small amount and dispersion is still a big issue. We are using surfactant and ultra-sonication but we find out that we still don’thave really good handle on dispersion. We measured dispersion using UV spectroscopy but the slight change in superplasticizer, even with the same company in a brand-name could change the dispersion and so, we are trying to see whether we can have a better control but we know that we can disperse it well.

So, I want to show some results of well dispersed carbon nano tubes, we have been also using carbon nano fibre. Carbon nano fiber somewhat more courser and the way they are made, we have not checked it but we think the bond could be somewhat better.

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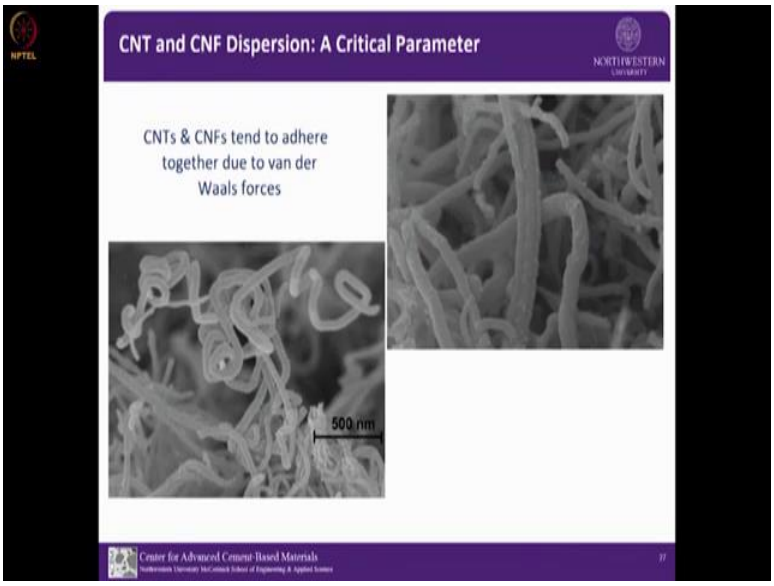

So, here to show you an example of what happens if they are not well dispersed fibres, they agglomerate it but then well dispersed, then we have individual in this case multi-walled carbon nano tube. This is with the SCM and there are really not very good way to find dispersion of carbon nano tube in hardened material because you do not really see them very well because they are at nano scale in SCM, in aqueous solution, you can with this UV spectroscopy. So, the idea with carbon nano tubes reinforcement is not like conventional reinforcement you have an aqueous solution, where you in the factory you have ultra-sonication and surfactant and then you just use that and similar to superplasticizer.

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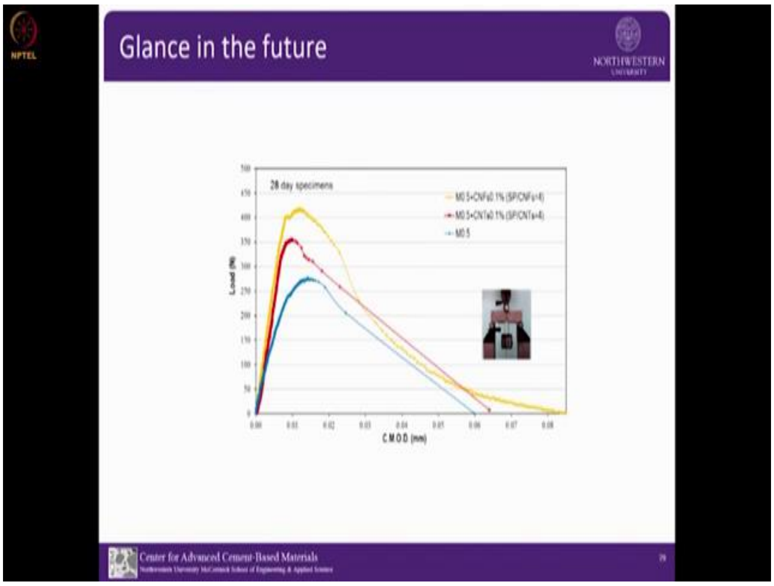

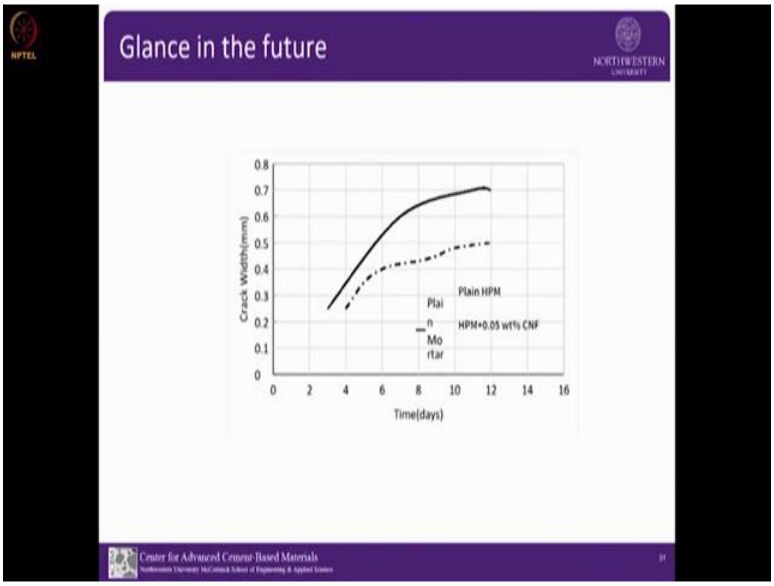

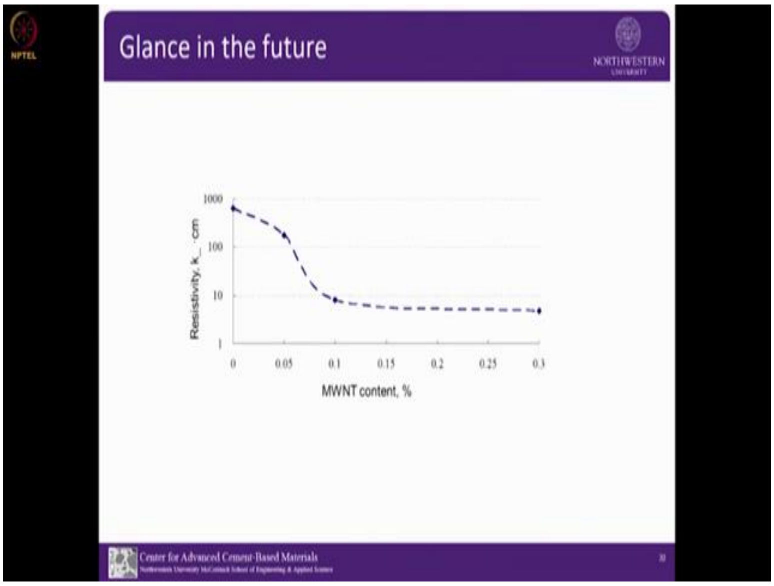

So, that is a different way of processing. So, I want to show you some results with the carbon nano tube, so these are fracture tests and these are mortar specimen with water to cement ratio 0.5 and these are 0.1percent .If you look at carbon nano fiber for example, we have substantially improved almost double the flexure strength but also maintaining very good post peak response.

Now we know that carbon nano tube 0.1percent they are well dispersed and why is it working so well with such a small amount? And that time we did not, we had and might be but Northwestern was saying no, we cannot use it because one of the student had spilled some mercury. So we said we could measure it, what else, what property can be substantially different because of change in nano pores? So measured autogenous shrinkage and we found out the substantial difference.

So, this is a water to cement ratio 0.3 and you measured it after time of setting and so, this is a plain cement paste and this is with very small amount 0.048percent. So, substantial reduction in autogenous shrinkage and we are still trying to understand why this is so. Then we use this ring chest and autogenous shrinkage, measured the shrinkage when the crack starts and how wide they are. So, this is a plain mortar and this one with 0.05percent carbon nano fiber. So, crack starts later in the ring and once it start, it is a lower crack width.

So, this combines in a way the advantage of both fibres and shrinkage reducing admixture because of reduce shrinkage as you can see and you reduce crack width. We many people are been talking about so-called smart material. So, you have take advantage of piezoresistive property of earning carbon nano tube, you know piezoresistive means you apply stress and the resistance change proportionately and repeatably, so like a sensor.

So, we did some work where we measure the resistivity. This is the work was done at Hohai University in Nanjing and multiwall carbon nano tube, what is resistivity without any and you can see at 0.1 percent conductivity has double, I mean two orders of magnitude not double and all resistance has gone down.

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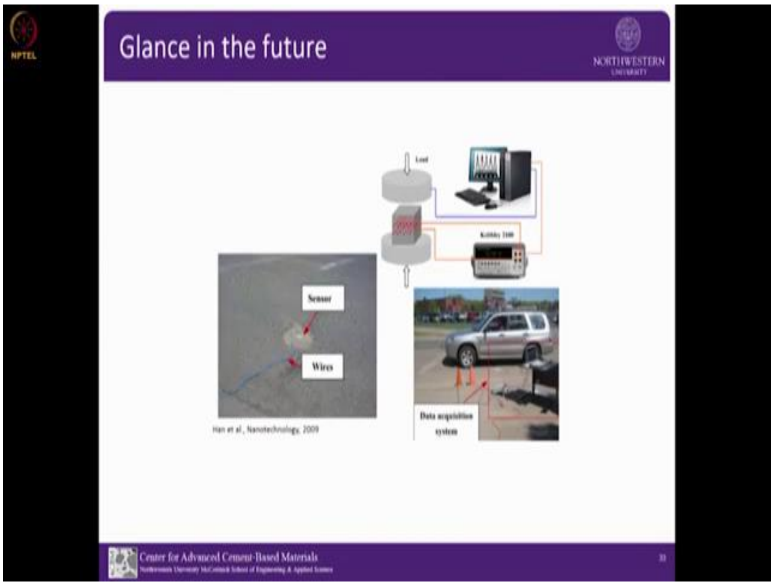

So, that is very effective for using this material as a sensor and many people have done work, this is not our work but somebody in highway department in Minnesota. So, this is the cement in 0.1percent carbon nano tube and you apply compressive load repeatedly and remotely measure, calibrate the change in resistance. So, when you embed this as a sensor, when a car goes through the change in resistance and second car and you can measure for example the speed of the car remotely and there are many other ways that people can use this as a sensor.

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So, that is another property of carbon nano tube. I want to conclude with some very interesting direction, so last year about January, the local big ready-made company, I think called Prairie before, when you were there it was material services, now one of the largest, so they call a workshop and they said that the tall building designers. They are asking that they want a double the modulus of concrete, we are talking about 55 Giga pascal and the reason.

So, there are people from material suppliers, ready-mades, contractors and they said currently to satisfy the requirement of modulus, they are increasing compressive strength instead of, let us say they would be happy with. So, the tall building with hundred, they are using 140 just because of the modulus and that is a more cement, not sustainable and also material is more brittle.

So, after that I realised that most of our work was on cement and carbon nano tube because we are talking about nano skill but we had seen increase in modulus. So, we said let us see what happens whether we can increase also, what is the increase when you have mortar, sand and aggregate. Now you realise if you as aggregate then carbon nano tube is not doing anything to aggregate, so, proportionately increase in modulus would be less.

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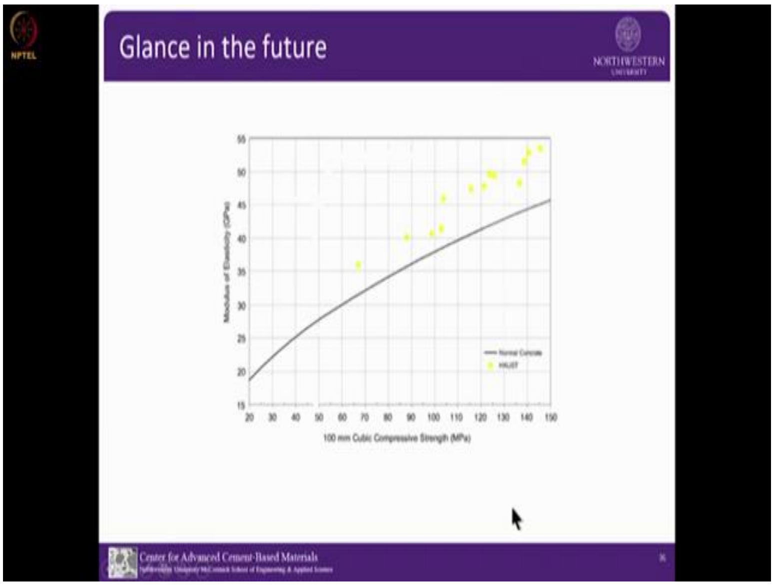

So, currently this is a, you know for example from the code in Hong Kong, modulus versus compressive strength and so if you want, let us say 50 Giga pascal modulus, we are talking about 150 compressive strength, so, keep this curve in mind and people are trying to do better than this curve but still you need high compressive strength to get that modulus.

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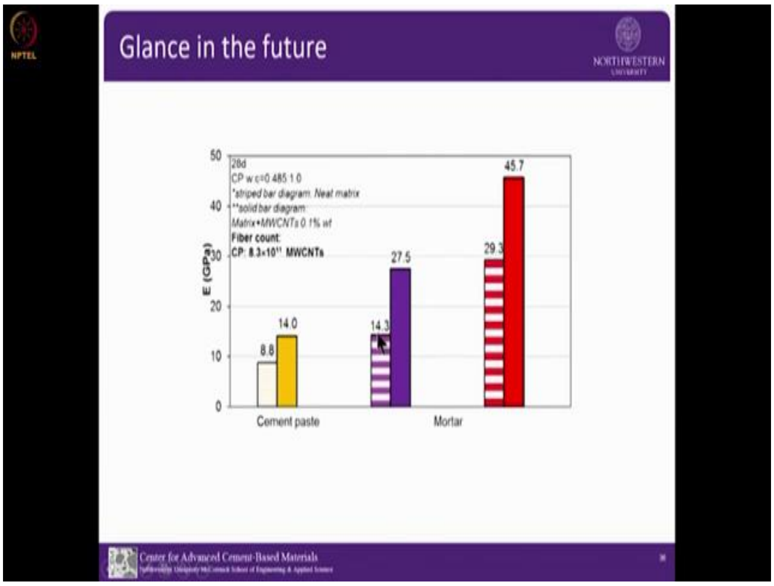

So, we when did this with cement paste mortar and concrete. So, look at this (above side), this is a modulus in gigapascal, cement paste and un-reinforce and 0.1percent but look at the same amount of carbon nano tube, the increase in mortar is even higher. You cannot model this with composite material approach, same thing more or less with concrete and I will show you why this is but so we thought that this is quite interesting.

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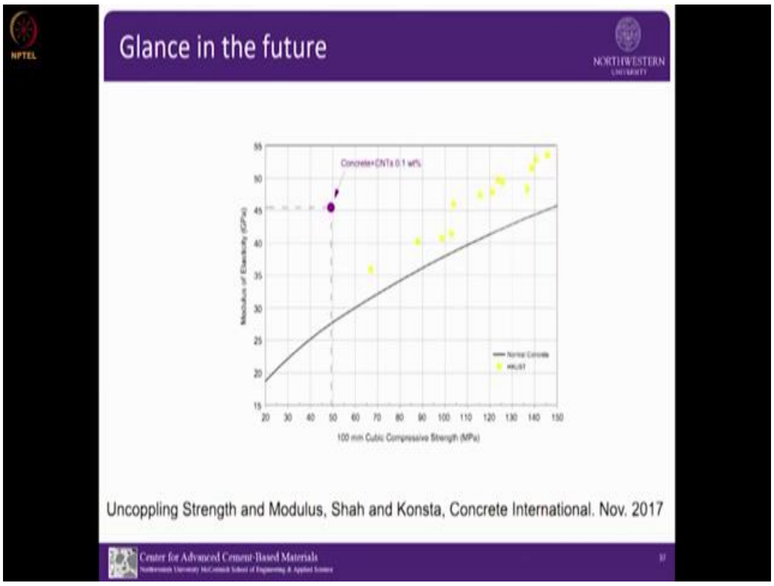

So, we plotted this our results with concrete cylinder and 0.1percent. So, this is the curve I showed you before and we did not really change much compressive strength but substantial increase in modulus and if you want that modulus with conventional approach then you need that much compressive strength. So, this is I think exciting development. You know recently in concrete international we had published.

So, designers seem to be quite a bit interested in this and we will see, of course the challenge is, we have only done this in the lab menu and to do it for concrete, we are going large, even though 0.1percent for ready mixture, so, you need a larger facility to ultrasonicate and so scaling up is always big problem. So, we will see how it goes but I thought this is very interesting.

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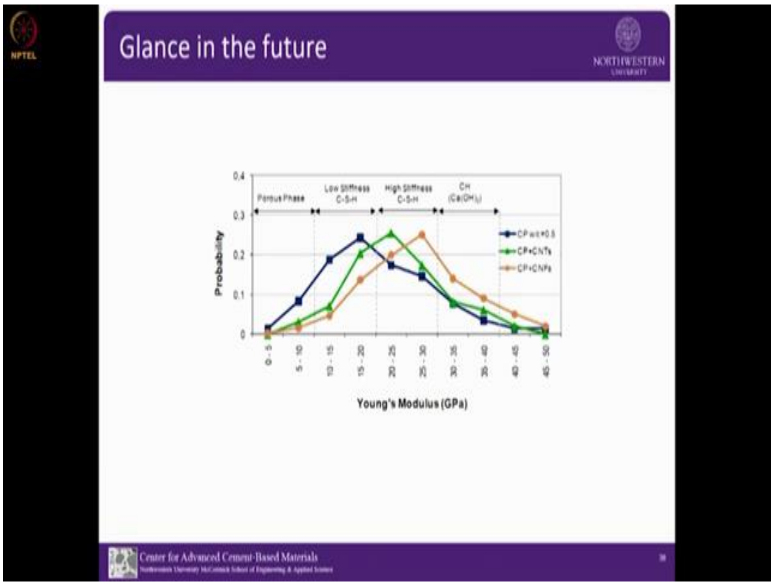

So, now why is small amount of carbon nano tube increase the modulus? So, first let us look at cement paste, for cement paste we did this nano indentation, some of you are familiar with it, you measure at the nano scale by using the indentation from load deflection, unloading, loading and using contact mechanics who calculate, what is the modulus of nano phase. So, this is an essentially the modulus of CSH and so this is cement paste with water to cement ratio 0.5 and this for example is cement paste with carbon nano fiber.

So, you can see that we have moved CSH modulus to the right, so, carbon nano tube has improve the stiffness of calcium silicate hydrate, so this is one of the reason why, even with a small amount 0.5percent of carbon nano tube, you can see the increase in the modulus of the composite. To see what is happening with the aggregate, we decided to measure the modulus at the nano scale of the interface.

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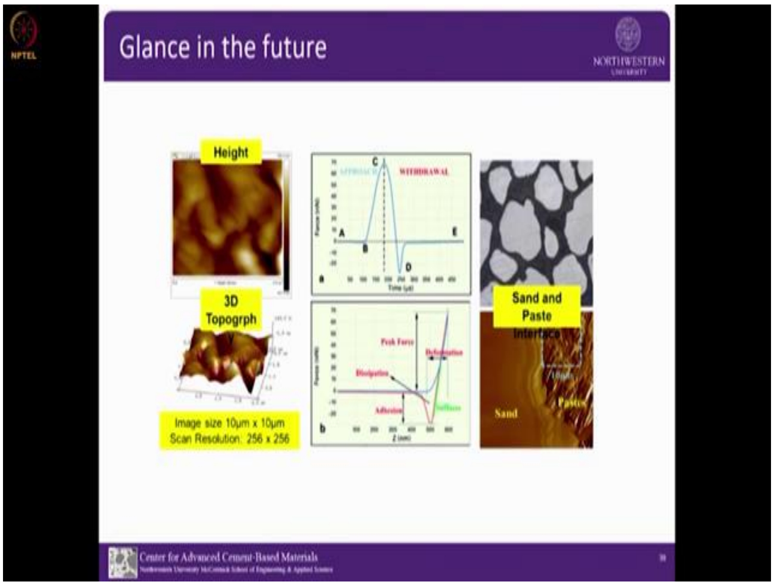

Now you know the interface is 10 to 15 micron, so, you cannot use conventional nano indentation because indent itself is few micron. So, we are using atomic force microscopy needle and it is applied in the cyclic loading, so you essentially measure as dynamic frequency. Also the force is very small and we can measure the spacing is much smaller than with conventional in so, measure doing this I want to show you some average valuation, of course there is a histogram.

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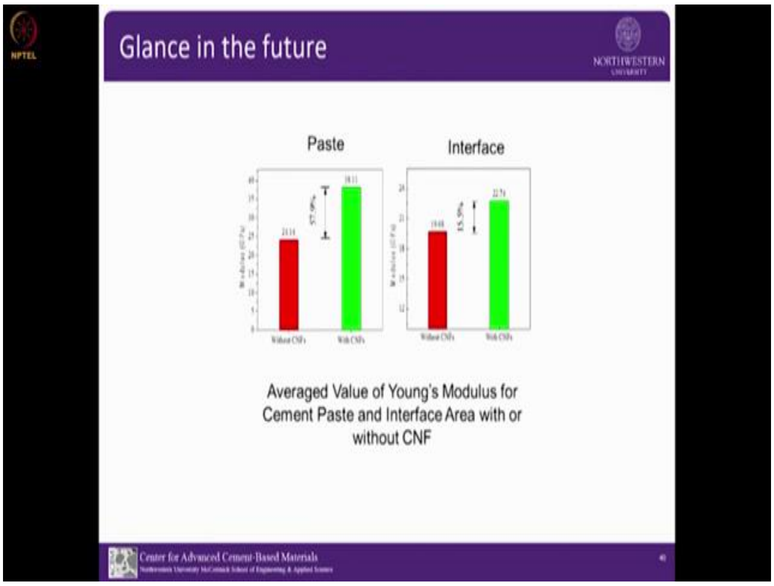

So, this is average increase with addition of carbon nano fiber of cement paste and interface, so, one of the reasons why mortal and concrete were proportionately more effective is that the interface modulus increases, so, you only, not only in case paste but also interface but we have to realise when you have a, let us say if you have a silica fume, it also densifies the interface but silica fume increases the compressive strength and but makes material more brittle. Here we do not really increases much compressive strength and as I showed you before material is not more brittle.

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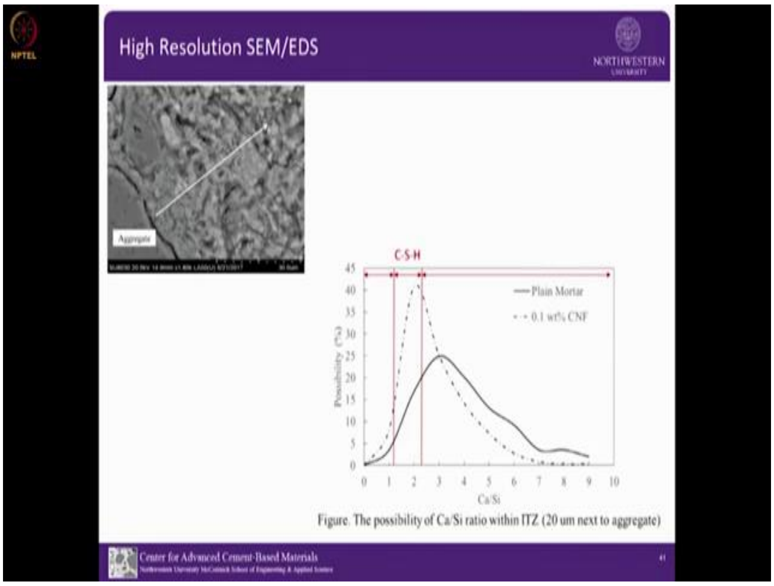

Then another interesting thing we found, we did with the SEM and EDS and this is the work done by Yuan Guan, the current Ph.D. student that she took several lines and measured using calcium to silica ratio, so these are average results and what we found that at the interface, the calcium to silica ratio is lower when we have carbon nano tubes. We know lower the calcium to silica ratio higher is the modulus of CSH and I quite do not understand it. I presented this at one of the conference where Karen was their and she said, she thinks that the for us the carbon nanotube may influence the crystallisation of CSH. So, that may be the reason but we do not know, but we are just looking but it is very interesting that if carbon nano tube also changes the morphology of calcium silicon hydrate. So, it is that really the modification at the nano scale, so that is really a good direction to do it.

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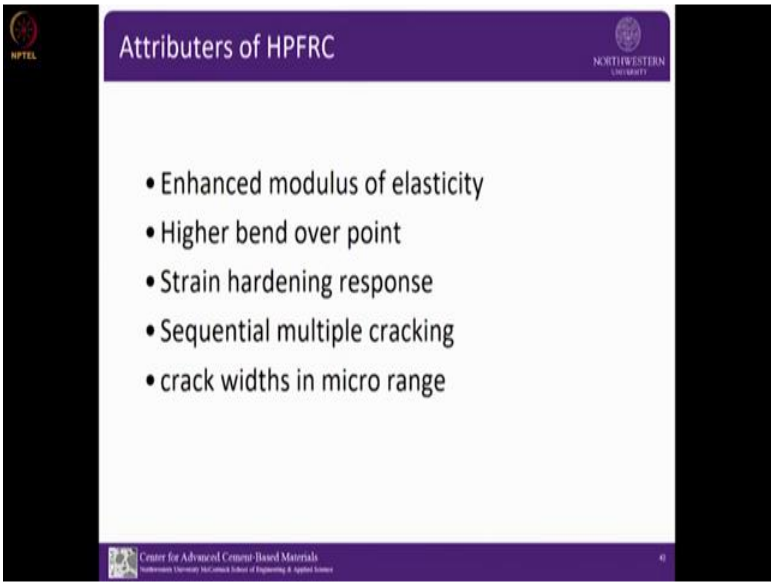

So, just to summarise what do we want in ultra-high performance fiber reinforced concrete, well I think we want modulus of elasticity, clearly that is very useful in tall building design, we want about at put higher band over point that is higher elastic, so we want noticeable crack to start as late as possible, very important we want strain hardening response and , during strain hardening we want sequential multiple cracking, that first cracks certain.

So, during the sequential multiple cracking, cracks do not open because there is no noticeable de-bonding at the interface, only after the cracks saturation, the crack will become open. So that is important, so many application you want crack width in micron range, we have shown and other people have shown that, let us say for a cover concrete 2 inch or 50 mm thick of cracks 50 micron or less, the material is impermeable.

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So, that is one way to control it. So I hope that I have given you some idea about what is the current development and what are the precursors? How we reach there? That is very important for us to know. So, that is my last slide. Thank you.