Mechanical Characterization of Bituminous Materials
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Lecture 36
Modifiers for Bitumen- Part 04

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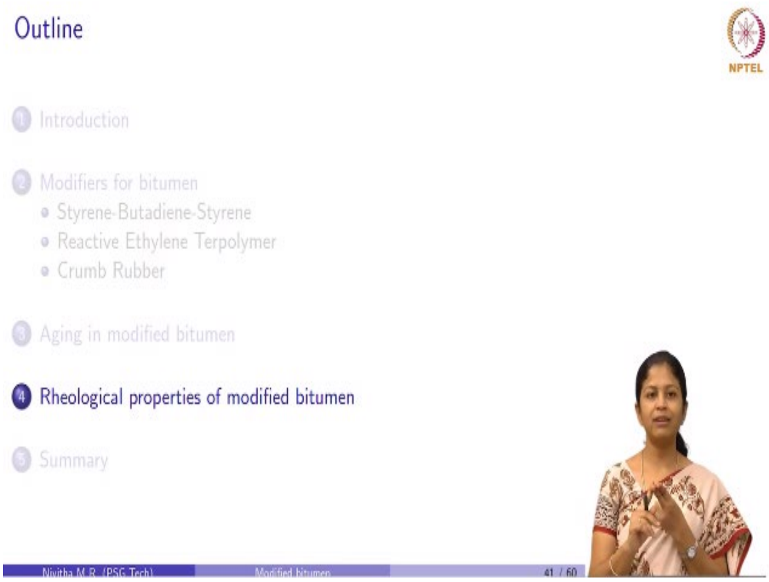

So next we are going to focus on the rheological properties of modified bitumen. So when we add a modifier to bitumen, it generally results in two implications on the base bitumen, the first one is increase in stiffness. So it can be increase in stiffness at high temperatures only or increase in stiffness at high temperatures and reduction in stiffness at low temperatures. It can be any of these depending upon the type of modifier.

The second most important and desired property of modified bitumen is the elastic recovery, the addition of modifiers is set to improve the elastic recovery of base bitumen. So we need to quantify these two effects of the polymer modified bitumen.

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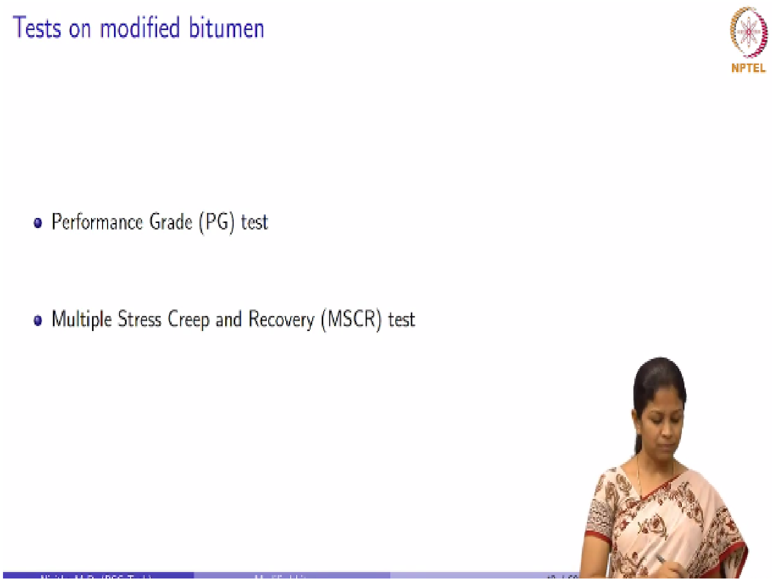

So for this universally there are two tests which are used; one is a performance grade test. So we know what is a PG test. It is used for unmodified bitumen but it is used for modified bitumen as well. The second is an MSCR test which is a multiple stress creep and recovery test. So, I will tell you why an MSCR test is needed in addition in the case of modified bitumen whereas only a performance graded PG test is sufficient for an unmodified bitumen.

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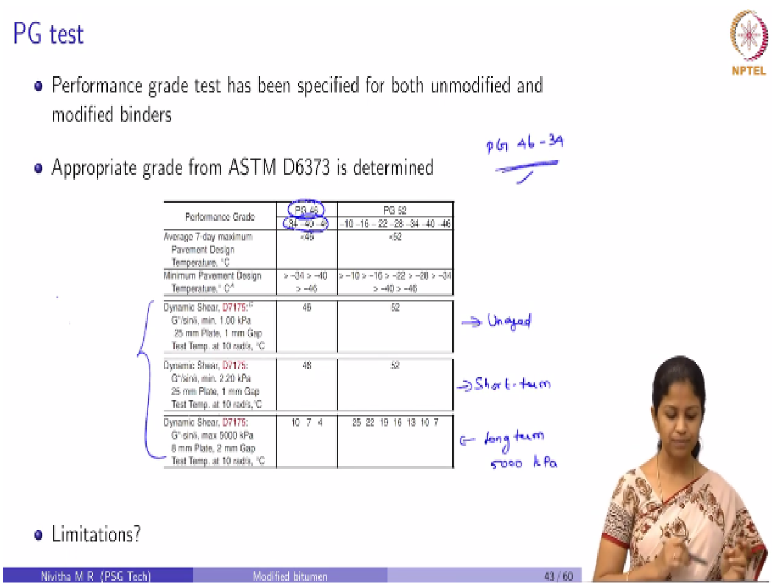

So how does this performance grade test work? So we know it is based on ASTM D6373 specifications. So we have a PG grade which is specified in terms of its high temperature grade, and we also have its low temperature grade here and we know there are at least three tests that needs to be done on this material; one is a dynamic shear where we measure |G*|/sinδ, it has to be a minimum of 1 kilopascal at the test temperature.

Similarly on short term, so this is on unaged material, on short term aged material again we measure the same |G*|/sinδ, but it has to be a minimum of 2.2 kilopascals and on long-term aged materials, we measure G*|×sinδ and it has to be a maximum of 5000 kilopascals. So we know all these tests how it is done and why it is done. So all these introduction is given to you when the performance grade lecture.

So we do the same test procedures and we evaluate what is the performance grade of modified bitumen, but this will give us only the influence of the modifier on the improvement in stiffness properties, whether it has increased the high temperature grade of the modified bitumen and whether it has lowered the low temperature grade of the modified bitumen, but we will not get any information on the elastic recovery properties of the modified bitumen.

We can have, say for example, PG 46 -34 material right and it can have different elastic recovery properties, say for example, we use an elastomer and a plastomer to get this grade of bitumen. The elastomer is expected to have more elastic recovery properties compared to the plastomer but more, more both of them can match PG 46 -34 grade, right? So we need to quantify the elastic recovery properties of this material separately.

And that is why we have a multiple stress creep and recovery test designed specifically or specified specifically for modified bitumen. So what is this MSCR test? It is used to capture the elastic response in the case of modified bitumen.

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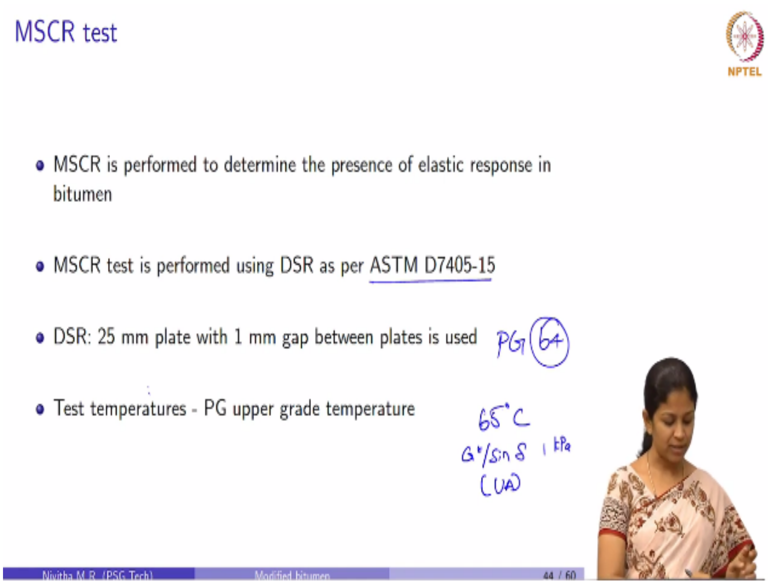

So this MSCR test is performed using a DSR, so we are using a 25 mm parallel plate geometry for this and the gap between the plates is specified as 1 mm here and the test is performed as per ASTM D7405 the 2015 version and the test temperature for this particular test is the PG upper grade temperature, so before we do an MSCR test, we have to do the PG test, determine the PG temperature grade of the material.

So for example, we have a temperature of 65 degree Celsius at which $|G^*|/\sin\delta$, in the unaged condition is 1 kilopascal right? So then we associate the PG grade as PG 64 because you know that we need to come to the lower grade in case of the material PG grade determination. So if the temperature is 65 at which $|G^*|/\sin\delta$ is 1 kilopascal, we associate a PG grade of PG 64, so at 64 degrees Celsius we have to perform this MSCR test. So this test can be done only after doing the PG test.

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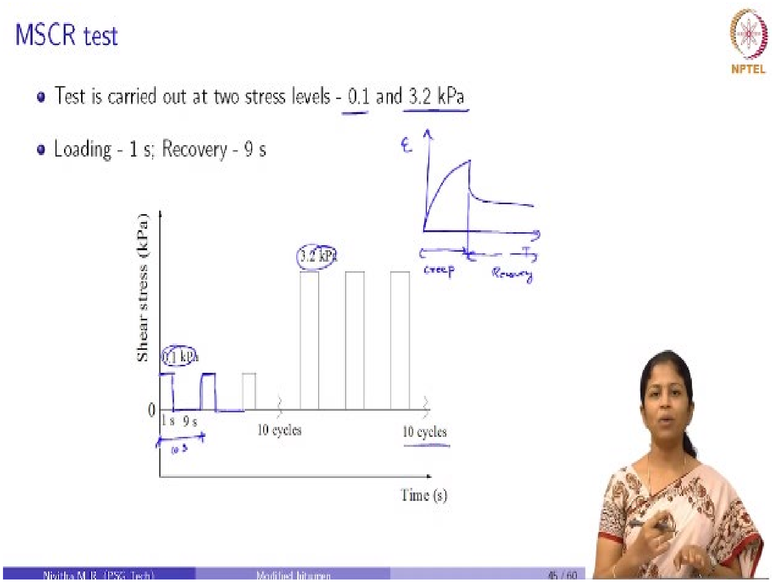

So how was this test carried out? this test is carried at two stress levels 0.1 and 3.2 kilopascals, so we can see that one is a lowest stress level and the other one is relatively higher stress levels, so there is a significance behind having two stress levels and evaluating the property of modified bitumen. So if you test the material at 0.1 kilopascal stress, we might not be able to see the beneficial effects of modifier, only at high test levels the modifiers will outperform compared to the base bitumen.

So to quantify the beneficial effects, we need to test the material at higher stress levels. Also, 0.1 kilopascal will mostly test the material in linear regime, so we know what a linear regime is and the difference between a linear regime and a non-linear regime. So we want to see how the material behaves in the linear regime as well as in the non-linear regime, so it should be able to recover relatively in the similar manner in both the linear regime and in the non-linear regime.

So we have two stress levels for this particular purpose, 0.1 kilopascal mostly to capture in linear regime and 3.2 kilopascal mostly to capture the effect in non-linear regime. So, they are the two stress levels which are used here and we know that this is a multiple stress creep and recovery test. So we know what a creep and recovery test is right? So what we do here is we apply some amount of stress, right?

This is creep, we give some amount of time for the strains to recover, right? So this is a creep portion and this is a recovery portion, so this is a creep and recovery test. So, we have multiple stress creep and recovery test. So this is like 1 loading 1 cycle. We are going to give 10 cycles of this creep and recovery test at one stress level and 10 cycles of this creep and recovery test at a higher stress level of 3.2 kilopascal.

This is schematically shown here, so we have 1 second of loading. So the load here is 0.1 kilopascal the shear stress, which is given here is 0.1 kilopascal and then we allow this strain to recover for 9 seconds. Then after 9 seconds we give again a loading of 0.1 kilopascals right and then allow it to recover for 9 seconds. So 1 cycle here composes of 10 seconds right? 1 second loading and 9 seconds recovery, so we have 10 seconds for a cycle.

So similarly we give 10 cycles that 0.1 kilopascal stress level then we move on to the next stress level which is 3.2 kilopascal. So again at 3.2 kilopascal we give 10 cycles, each cycle has 1 second of loading and 9 seconds of recovery. So like that we again give 10 cycles at 3.2 kilopascal stress level. So this is the MSCR test that is performed here. So we need to say under repeated loading how much the material can recover

And this in a way is related to how the strains recover in field because in field we see there is a vehicle passing. So we have a point here, there is a vehicle passing. So at that time the material is loaded and once the vehicle leaves this influence zone, then the strain starts to recover. So we have loading-recovery then another vehicle tries to enter the influence zone, again there is loading on this material then once it leaves the point there is some amount of time for the strains to recover until the next vehicle comes.

So this test is designed in this specific manner to capture the relative phenomena what happens in the field.

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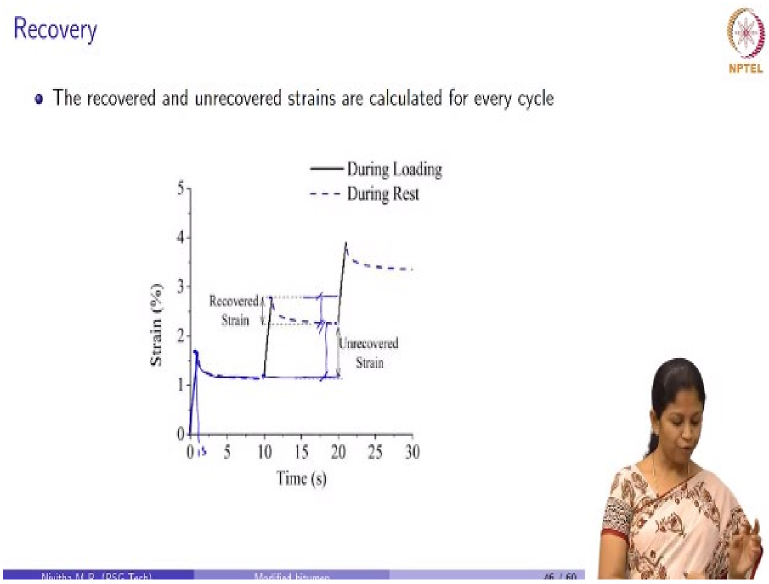

Now, this is the strain which is captured in the MSCR test. So like we said, this is the loading period which is shown as a solid line and this dotted line corresponds to the recovery period. So, during the loading period, so this is at the end of 1 seconds of loading. So we have a peak strain which is given here and then the strain start to recover, right? So this is the recovered strain, the difference between this peak strain and the strain at the end of 10 seconds, right?

So this is called as a recovered strain, so this much of strain the material is able to recover and this one right? So the strain in the initial period to the strain at the end of 10 seconds, so this portion is the unrecovered strain. So we can quantify what is the amount of recovered strain and unrecovered strain for every cycle. So this information is used to calculate a number of parameters and that is used to form specifications.

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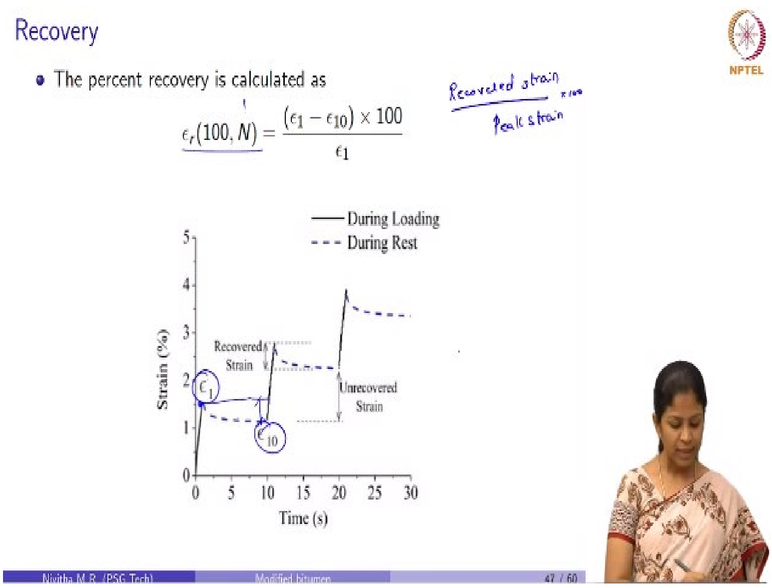

The first parameter which is calculated here is percent recovery. So which is very simple as the name says how much percentage is the percentage of the strain the material is able to recover. So I have to denotions here, E1 represents to the strain at the end of 1 second, which is also nothing but the peak strain. Similarly I have another denotion E10, and which is this strain at the end of 10 seconds. So this is for the first cycle.

So the first parameter that is calculated here is percentage recovery. So as the name says, it says how much of strains the material is able to recover? So that is what is quantified by this parameter. So let us take two denotions epsilon 1, which is the peak strain here and the strain which corresponds to the end of 1 second. So at the end of 1 second, what is the strain in this material that is represented by epsilon 1 and we have another parameter epsilon 10.

So this epsilon 10 says how much is the strain in the material at the end of 10 seconds that is after the end of recovery period. So the percentage recovery is captured calculated as epsilon r and this is for 100 Pascal stress and for nth cycle. So here we are going to calculate it for the first cycle. So it is given as epsilon 1 - epsilon 10 that is nothing but this difference here. Epsilon 1 - epsilon 10, which is nothing but the recovered strain by the peak strain.

So I can just write it simply as the recovered strain by peak strain into 100, right? So we calculate this percentage recovery for first cycle. So similarly, we calculate it for all the other 9 cycles and we take an average value, so this is the percentage recovery at a 0.1 kilopascal or 100 Pascals. Similarly we can calculate the percentage recovery for 3.2 kilopascals also.

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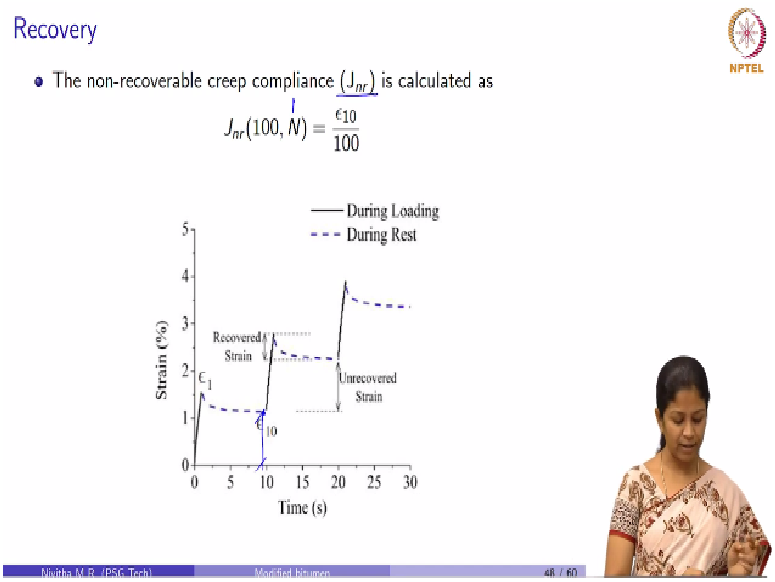
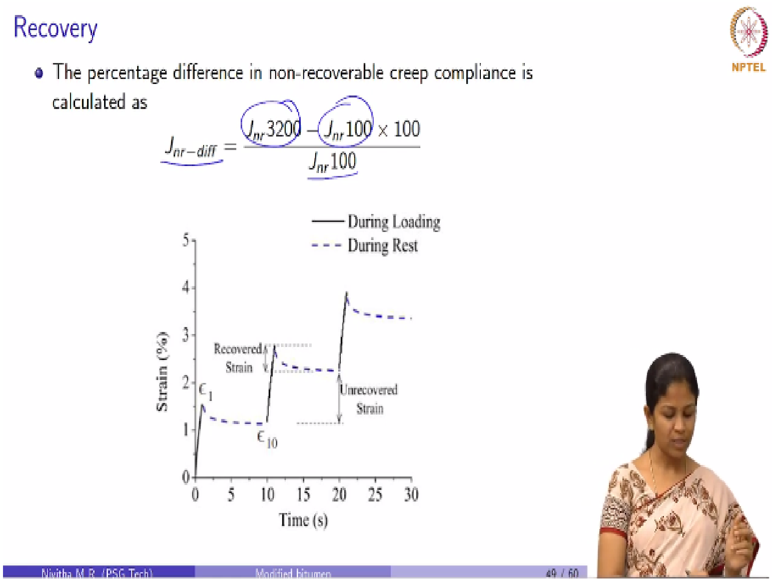


The next parameter which is calculated here is the non recoverable creep compliance, which is called as Jnr, right? J subscript n r. So, this parameter is what is used in specifications. So what is this parameter? Jnr again for 100 Pascals shear stress and for nth cycle for us again, the first is cycle is nothing but epsilon 10 by 100. So what is this epsilon 10? This epsilon 10 gives us the unrecovered strain. So this quantifies the unrecovered strain as a percentage.

Now, we want more amount of recovery of strains to reduce the permanent deformation because if we have accumulation of strains, it is going to lead to permanent deformation. So we want to minimize this accumulation of strain. So if we want to minimize this accumulation of strain, we want Jnr value to be as low as possible. If this Jnr value is high what it means is that the amount of unrecovered strain is high.

So which is not desirable, so we want a minimum value for this Jnr and when you see the specification, you will see how this Jnr value is specified for different classes of binders. **(Refer Slide Time: 13:25)**



The next parameter which is given here is Jnr difference, which is nothing but Jnr at 3200 Pascals - Jnr at 100 Pascals as a ratio of Jnr at 100 Pascals. So it says how much is this Jnr value at a higher stress level relatively to that at a lower stress level? So this means that at higher stress levels, we do not want the strain recovery to be very low. So there can be some materials whose strain recovery can be very good at low stress levels, but as the stress level increases may be the recovery properties are very poor.

So we do not want such kind of materials because under heavy traffic loads we want the strain recovery to be as high as possible. So to quantify that they have given this parameter Jnr difference which is defined as Jnr at 3200 - Jnr at 100 as a ratio of Jnr at 100, right? So these are the parameters which are used in specifications.

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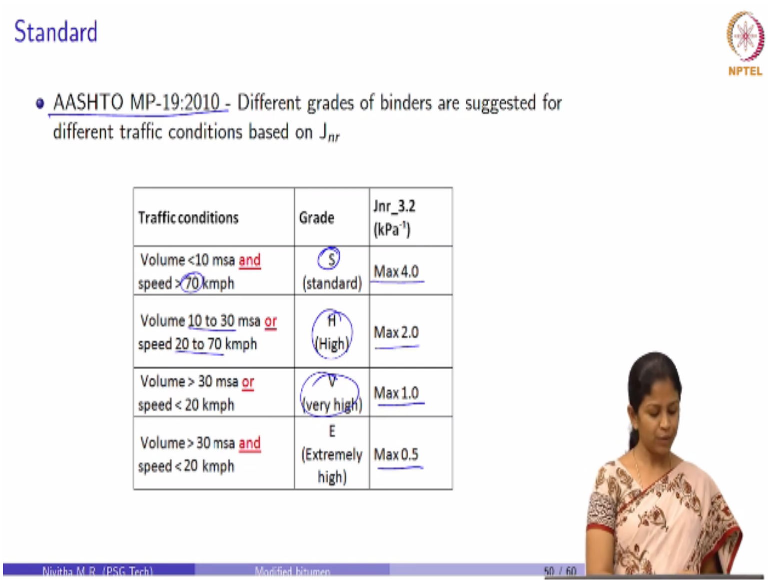

So let us now see how this Jnr value is used to specify different grade of binders for different traffic conditions. So this is specified in the AASHTO MP-19: 2010 specification. So here traffic is represented using two parameters, one is the volume of traffic, the other one is the speed of traffic. So by this time we know what is the effect of the number of repetitions on the distresses? The second one is the speed.

So we know that at high speed the time of loading is very less, so the material will exhibit a viscoelastic solid like behavior and the deformation is very low. Whereas at low speeds the time of loading is very high and the material will try to deform. There will be more strains on the material. So based on these two parameters, the Jnr value is specified for different conditions.

The first case is a standard case where in the traffic volume is less than 10 MSA relatively lesser number of repetitions and the speed is greater than 70 kilometres per hour. So less number of traffic repetitions and most of them travelling at high speed. So this is defined as a standard case and the Jnr value here at 3.2 kilopascal shear stress is given as a maximum of 4.

The next case is the volume is between 10 to 30 MSA here or the speed is between 20 to 70 kilometres per hour. So this defines a case where in either the traffic volume is high or the traffic speed is relatively lower. This is said as a high case and the Jnr value here for this case is maximum of 2. The third case is when the volume is greater than 30 MSA or the speed is less than 20 kmph.

The difference between the second and the third case is that the second case is for speeds between 20 to 70 kilometre per but the third case is for speed less than 20 kilometre per hour. So this is said as a very high traffic case and the Jnr value here is a maximum of 1. The fourth cases where the volume is greater than 30 MSA and the speed is less than 20 kilometres per hour, the third case is either one of these variables are critical, either volume is greater than 30 MSA or the speed is less than 20 kilometres per hour.

Whereas in the fourth case both the volume is high and the speed is relatively lower. So this case is observed mostly near the toll plazas where the vehicles are travelling relatively slower and there are more number of vehicles queued up. So this is an extremely high case and the

Jnr value here is a maximum of 0.5. Now for all these four cases we can see how the Jnr value varies.

For a standard case, the Jnr value can go up to a maximum of 4, whereas in an extremely high case, it can go only up to a maximum of 0.5, so like we defined before, a higher Jnr value is not desirable under extremely high traffic conditions because there is more amount of unrecovered strain.

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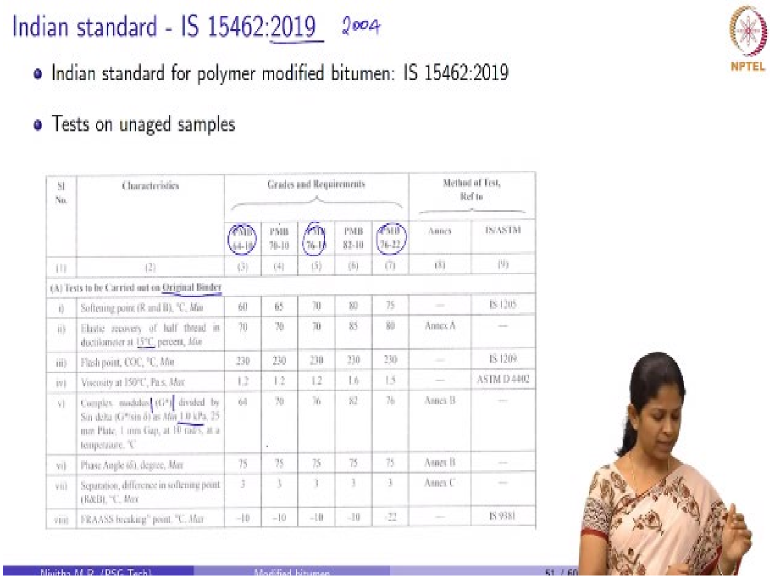

Next we move on to the Indian standard for modified bitumen. So this standard is IS 15462. The resent to version is 2019 there was a previous version which was published in 2004. So this 2004 covered all the modifiers in the same code, so we had one table for elastomeric modified bitumen, one table for plastomeric modified bitumen and two tables for crumb rubber modified bitumen.

One for natural rubber and another one for crumb rubber, but in this recent revision, it separated into two codes. The first code 15462 focuses on all type of modified bitumen, whether we prepare it using an elastomer or plastomer or any other polymer, it comes under this code. The other code IS 17079 focuses only on rubber modified bitumen. Let us now first look into 15462, and then we will move on to 17079.

So this standard for polymer modified bitumen specifies bitumen under five grades. So if we have a PG 64 -10, which means that this binder can withstand up to a high temperature of 64 and a low temperature of -10. So, we all know this, so there are 4 high temperature grades 64, 70, 76 and 82 for a similar low temperature grade, the reason behind this is that, in India we do not have more locations where the low temperatures will follow sub zero.

Hence, because of this, we do not want very low temperature grades in India and that is why the low temperature grade is limited to -10. There is only one grade which is specified for -22. So you can see we have 2 grades of binder with the same high temperature, PG 76, but the low temperature for these 2 grades of binders are different, one is –10 and the other one is -22.

So this means that PG PMB 76 -22 can be used for colder climates compared to PMB 76 -10. So, these are the 5 grades of polymer modified bitumen, again we need to note that it can be prepared with any type of polymer. Now, let us look into what are the tests that are carried out on this binder. So, it is given as the tests carried out on original binder; by original binder they mean unaged bitumen.

So, we see the first one is softening point. So, we know what a softening point is, how it is done, elastic recovery using that is nothing but a ductility test at 15 degrees Celsius here, we have a flashpoint, we measure the viscosity at 150 degrees Celsius and it is also specified to measure complex modulus and this is $|G^*|/\sin\delta$ which is specified in the PG specification.

This $|G^*|/\sin\delta$ should have a minimum of one kilo Pascal at the test temperature and we also have a phase angle which is defined as a maximum of 75. So, we know that for viscoelastic materials which are more fluid like the phase angles will be relatively higher. So we are here we are limited to a maximum of 75 degrees and then we also have a separation test, which is specifically given for modified bitumen.

So, this separation test quantifies the phase separation that happens in the material when it is stored under hot conditions. So we said that the interactions between the modifier and bitumen should be irreversible at all conditions. So what this test does is, it stores bitumen at 163 degrees Celsius for about 4 hours. So, what they do is they have an aluminium tube in which it is filled with bitumen and it is stored at 163 degrees Celsius for 4 hours.

We need to ensure that it is stored vertically, so that if there is any phase separation we allow it to happen and after the end of 4 hours this kept in freezer for about 1/2 hour or 1 hour and then it is removed from the freezer, cut into three portions. So if there is any phase separation, depending upon the specific gravity of the modifier, it can either settle down or it will move up to the top portion.

So what they do here is, they discard the middle portion and measure the softening point of the top and the bottom portion. So if there is any phase separation, we will be able to see a significant difference in the softening point of the top and bottom portion, and we also know what a Fraass breaking point test is. So, these are some of the tests which are specified in this code for modified bitumen in unaged condition.

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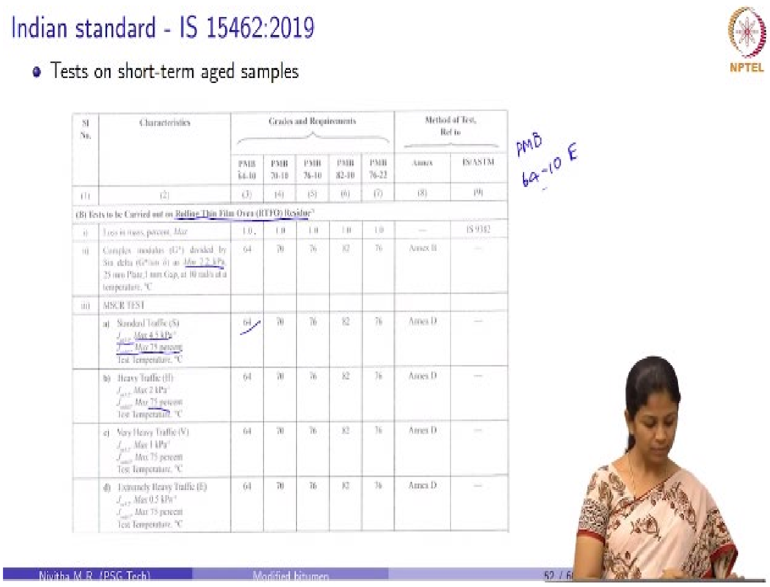

Next we move on to the tests which are performed in short term aged conditions. So, you can see this is test carried on rolling thin film or residue. So, we know what an RTFO procedure is; there are three tests which are specified here, the first one is loss in mass in percentage. So, in aging we have seen what is the loss in mass and what is it, what it is due to. So, this loss in mass is mainly due to the loss of volatiles in bitumen upon aging.

And we do not want a significant loss of volatiles because that will disturb the compatibility of the different components in bitumen. So, this is limited to a maximum of 1%. The next test is again complex modulus divided by sine delta which is a minimum of 2.2 kilopascals. This is nothing but $|G^*|/\sin\delta$ a minimum of 2.2 kilopascals at the test temperature in short term aged conditions.

The third one is an MSCR test what we have seen before. So, this MSCR test is defined for four traffic conditions; a standard traffic condition, heavy, very heavy and extremely heavy traffic condition. So, these are the four cases which we have defined earlier. The next slide I also show you what they correspond to. So, for a standard traffic case, this Jnr value at 3.2 kilopascal should be limited to a maximum of 4.5 kilopascal inverse and this Jnr difference should be a maximum of 75 %.

So, these are the specifications for standard case. So, at test temperature we need to check these parameters? The test temperature depends upon the grade of binder. So, for 64 grade binder, we need to test this at 64 degrees Celsius similarly, different temperatures depending upon the grade of binder. For a heavy traffic case, this should be limited to 2 kilopascal inverse again Jnr difference a maximum of 75 %.

So, this Jnr difference maximum value remains the same for all the 4 traffic conditions. Only the Jnr value at 3.2 kilo Pascal varies depending upon the traffic condition. So, how we denote a binder is, so, this is how we denote the binder, PMB 64-10 E extremely high, right? So, this is the requirement of a binder is required to be used for a climate with temperatures 64 and minus 10 and extremely heavy traffic conditions.

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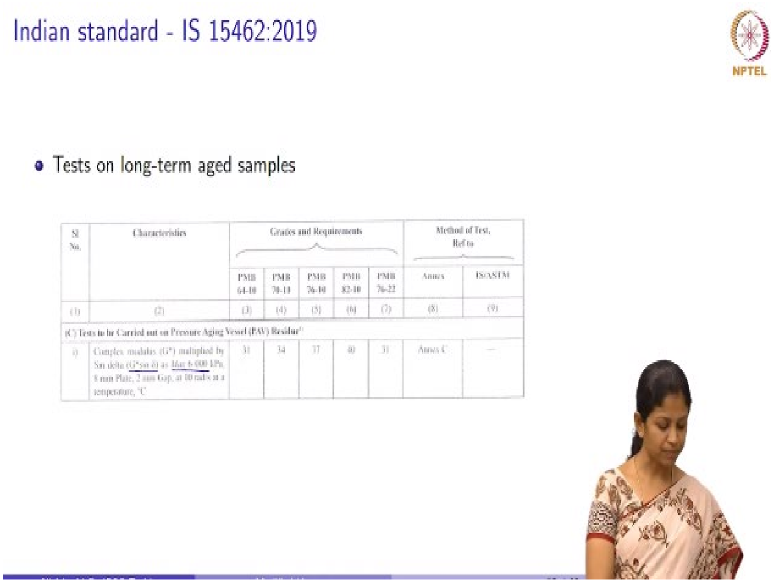

Next, we will move on to the tests which are performed on long term aged samples. So, in the PG grade we had three tests. One is $|G^*|\times\sin\delta$, the second one is the creep stiffness test and third one is a direct tension test, but here they have mentioned only the tests which are performed using a dynamic shear rheometer. So, we have G*|×sinδ and here the maximum limit is 6000 kilo Pascal.

Again the intermediate temperature in which we test depends upon the grade, we have it varying from 31 to 40 degrees Celsius. So, these are some of the tests which are specified for modified bitumen.

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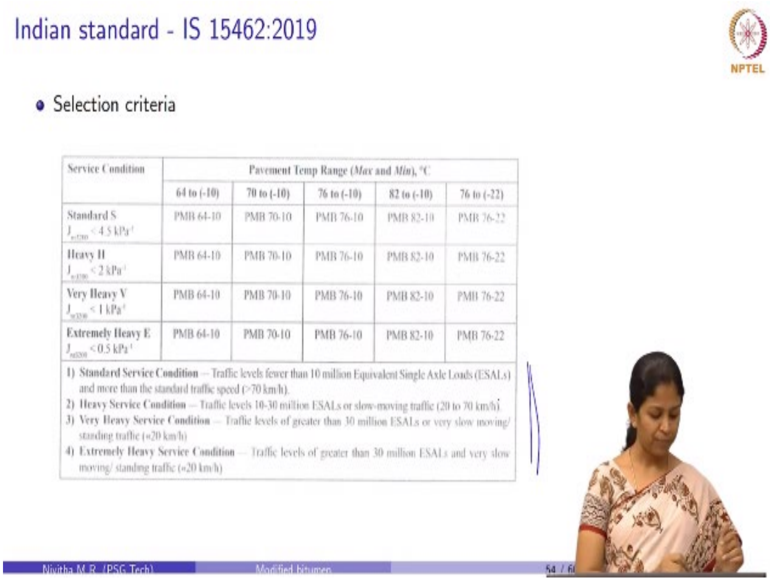

So, now, they have given a table which says which modified bitumen should be used where. So, on the x axis here we have the pavement temperature range again that is already defined in the binder grade here and on the other side we have the traffic conditions. So when the temperature varies from 64 to minus 10 and when the traffic is extremely heavy, we need a PMB 64 - 10 with an E grade as I mentioned before.

And they also have mentioned what are these 4 traffic conditions that correspond to, which is similar to what I have explained before using the AASHTO standard, Right? So these are some of the guidelines for polymer modified bitumen.

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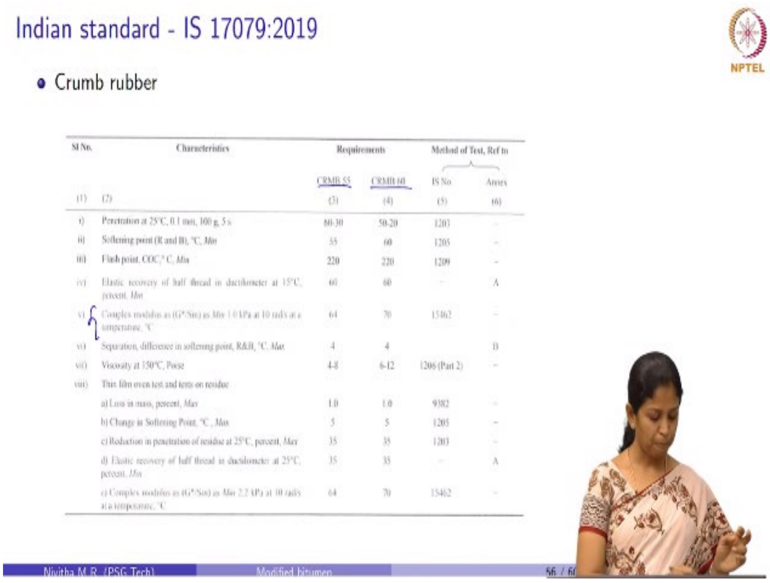

Sl No.	Characteristics	Requirements		Method of Test, Ref to	
		CRMB 55	CRMB 60	IS No	Annex
(1)	(2)	(3)	(4)	(5)	(6)
i)	Penetration at 25°C, 0.1 mm, 100 g, 5 s	60-30	50-20	1203	–
ii)	Softening point (R and B), °C, *Min*	55	60	1205	–
iii)	Flash point, COC, ° C, *Min*	220	220	1209	–
iv)	Elastic recovery of half thread in ductilometer at 15°C, percent, *Min*	60	60	–	A
v)	Complex modulus as (G*/Sinδ) as *Min* 1.0 kPa at 10 rad/s at a temperature, °C	64	70	15462	–
vi)	Separation, difference in softening point, R&B, °C, *Max*	4	4		B
vii)	Viscosity at 150°C, Poise	4-8	6-12	1206 (Part 2)	–
viii)	Thin film oven test and tests on residue				
	a) Loss in mass, percent, *Max*	1.0	1.0	9382	–
	b) Change in Softening Point, °C, *Max*	5	5	1205	–
	c) Reduction in penetration of residue at 25°C, percent, *Max*	35	35	1203	–
	d) Elastic recovery of half thread in ductilometer at 25°C, percent, *Min*	35	35	–	A
	e) Complex modulus as (G*/Sinδ) as *Min* 2.2 kPa at 10 rad/s at a temperature, °C	64	70	15462	–

Next we will move on to crumb rubber modified bitumen and natural rubber modified bitumen which is given an IS 17079. So, they have two tables one for natural rubber and the other one is for crumb rubber. So, we have the standard test penetration, softening point, Flashpoint, elastic recovery, separation, viscosity at 150 and also tests on residue. So we have a loss in mass, change in softening point, reduction in penetration of residue, and then elastic recovery.

So previously, we saw the loss in mass is common across all specifications, but the change in softening point and the change in penetration are the two parameters which are specified for natural rubber. Next, we move on to crumb rubber, we have similar test set of tests except that we have a complex modulus value, which is specified as $|G^*|/\sin\delta$, similar to the PG grade.

So, for the unaged material, this value has to be a minimum of 1 kilo Pascal at the same specifications, 10 radians per second and for the desired test temperature, this value is also given on the short term aged residue. So, this value should be a minimum of 2.2 kilo Pascals at 10 radians per second for the short term aged residue depending upon the test specification.

So, this is the standard which is corresponding to rubber modified bitumen, but like the polymer modified bitumen, they have not given any selection criteria, under which case we have to use which grade of modifier, we only have two grades of binder for natural rubber NRMB 70 and NRMB 40 and again, two grades of binder CRMB 55 and CRMB 60.

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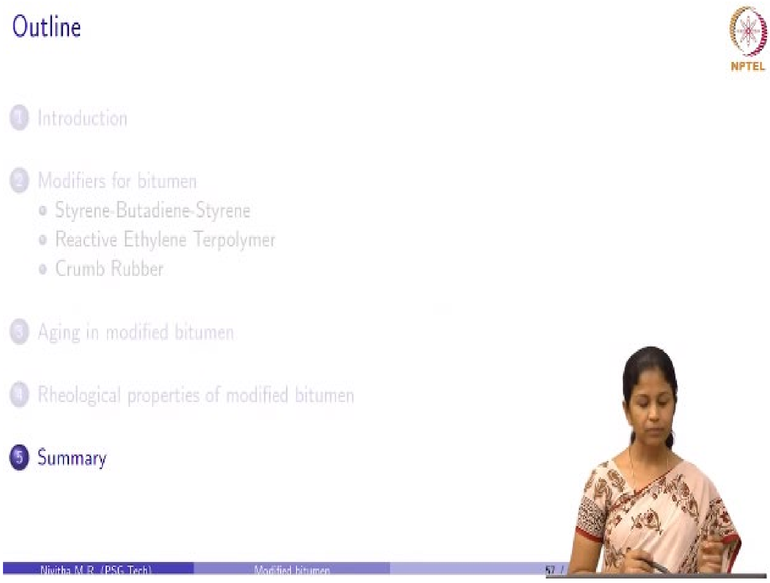

So, finally, we will now summarize the learnings what we have understood from the topic on modified bitumen.

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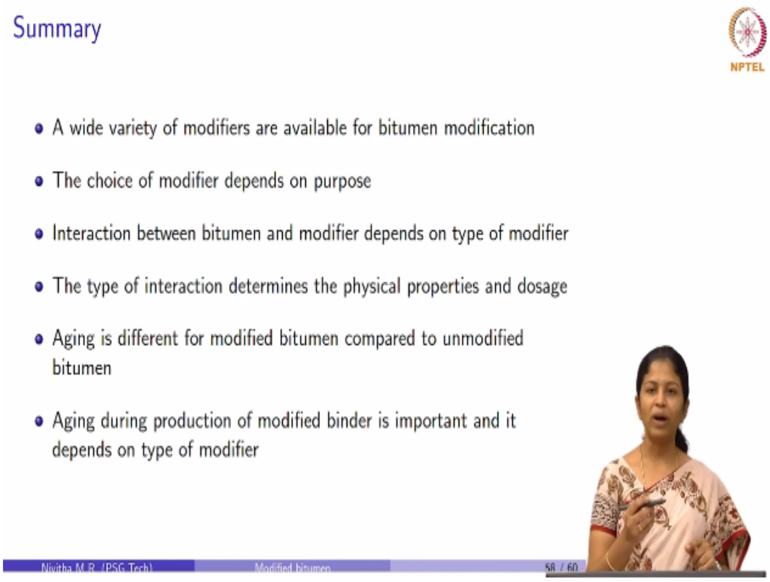

So, the first thing is there are a wide variety of modifiers that are available for bitumen. So, depending upon the purpose we need to choose the type of modifier. So, we have seen that there are fillers, there are antistrips, antioxidants and different types of polymers, elastomers, plastomers, crumb rubber. So we have seen a wide variety of them. So, depending upon the purpose, we need to choose an appropriate type of modifier.

The next one is the interaction between modifier and bitumen depends upon the type of modifier. So, we have seen that in the case of elastomers, there is only a physical interaction whereas in the case of plastomers there is physical as well as chemical interaction. In the case of crumb rubber, we see a physical interaction, an inert case and in some specific conditions, chemical interaction also.

So, how the modifier interacts with bitumen depends upon the type of modifier. This interaction determines the physical properties and dosage. So, we said in the case of SBS, it has both styrene and butadiene component, the styrene provides stiffness to the material and butadiene provides elasticity to the material and it swells in volume, forms a three dimensional network.

So, this contributes to the increase in elastic properties and stiffness at high temperature and reduction in stiffness at low temperatures. So, the influence of physical properties again depends upon the type of modifier and how it interacts with bitumen. Similarly, the dosage is also limited by this case. In the case of SBS modified bitumen, we saw that we cannot add more than 7% because there is a phase inversion that will happen and that is undesirable.

Similarly, in the case of reactive ethylene polymer, we saw that it should be limited to 1.5 to 2.5 % otherwise it will form lot of cross linking and it will lead to a chemical gel. So, the dosage depends upon the modifier. The next one is the aging that happens in modified bitumen is different compared to base bitumen. We need to take into account the aging that happens in the modifier as such and the aging that happens in the base bitumen under the influence of the presence of modifier.

So, these are two factors that we need to take into account and we also saw what is the aging that happens during the production of modified bitumen. So, this is something which we need to take into account while preparing modified bitumen.

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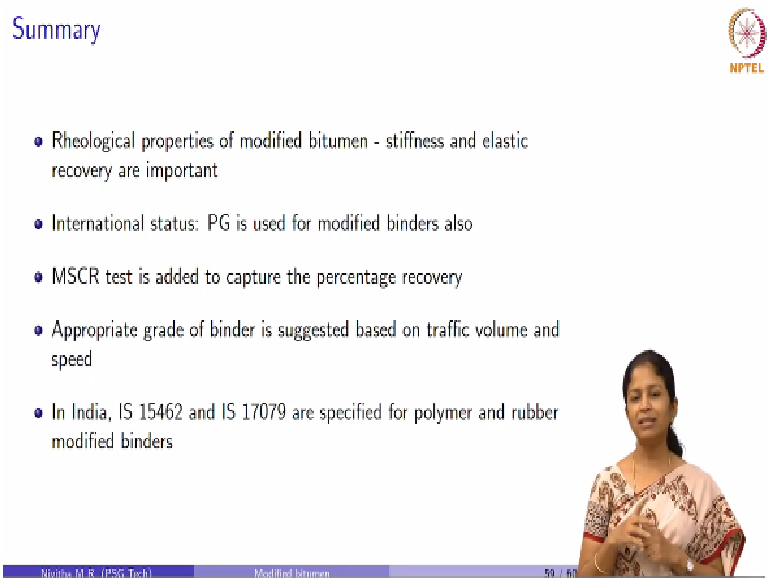

Then, the rheological properties of modified bitumen are quantified using two parameters. One is the improvement in stiffness and the other one is elastic recovery. So, to quantify the improvement in stiffness, generally a PG grade is specified. That is what is used across many countries and in India also the recent revision is in lines with this PG grade. The second one is an elastic recovery, which is quantified using a multiple stress creep and recovery test which is called as an MSCR test.

So, again we need to do PG test and then followed by an MSCR test. So, in the recent Indian code IS 15462 also, we have both PG test and parameters corresponding to the MSCR test and then they have also specified appropriate grade of binder for different climatic conditions and different traffic conditions. So, we saw PMB 64 -10 or PMB 70 -10 indicating the climatic conditions and the traffic conditions defined from standard to extremely very heavy.

So, these are some of the guidelines which is given in this code IS 15462 for polymer modified bitumen whereas, for crumb rubber, natural modified bitumen in the recent version, there is a separate code which is IS 17079 which gives 2 tables; one for natural rubber and for the other one is for crumb rubber modified bitumen. So, this is a summary of what we have learnt for the different types of modifiers from bitumen, their interaction and their influence on the physical properties and what are the coded provisions related to this.

So, there is something important which we need to take into account when we evaluate modified bitumen that is, the effect of modifier on the rheological properties will be predominant only after few years of service in its field. Say for example, we lay a road after one or two years, an unmodified bitumen and the modified bitumen can behave in the same way, can exhibit similar type of distresses.

But only after it has been subjected to high traffic conditions and extremely different weather conditions, we will be able to quantify the beneficial effects of modifier. So we need to take this into account when we do the tests on modified bitumen in the laboratory and when we evaluate a pavement with unmodified bitumen and modified bitumen to access its performance.