THE EFFECT OF DE-ICING CHEMICALS ON THE STIFFNESS OF ASPHALT SURFACE COURSE MATERIALS SUBJECTED TO FREEZE-THAW CYCLES

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ABSTRACT: De-icing chemicals used in the winter to combat the formation of ice and snow on the pavement surface only suppress freezing temperatures, and do not act to alleviate the effects of low temperatures and subsequent freeze-thaw cycles. The low temperatures endured by the pavement can have detrimental effects on the structural integrity of the asphalt, and freezing fluids can cause frost heave that can cause pavement failure. The in-direct tensile test was used to determine the in-direct tensile strength (ITS) of various asphalt mixes ranging from close graded bitumen macadam's to stone mastic asphalt, to determine the susceptibility to low temperature cycling and de-icing chemicals. Analyses of data show how bitumen embrittlement affects the strength of the asphalt, a problem that causes pavement failure. The test temperatures focus on the upper and lower ranges that can be experienced during the winter season. Samples were created to mimic the real world situation, with samples being sawn on a plane perpendicular to its axis to expose aggregate, but keeping the samples within the specification for testing. Initial tests have show how the ITS at –20°C for the samples was five to six times those at 20°C.

KEYWORDS: Freeze, heating, highway, pavement, road, temperature, thaw, salt, and stiffness

1. INTRODUCTION

During the winter season highways are subjected to low temperatures that are often sub-zero. The affect of temperature changes between daylight and night time due to the warming effects of the sun, and hence the pavement can experience a freeze-thaw cycle. It is not the purpose of this study to look at various temperature changes occurring due to climatologically or geographical position, but purely the effect of temperature change based on standard testing procedures. In many areas around the world, the onslaught of freezing temperatures on the pavement surface requires the application of de-icing chemicals due to the freezing of fluids on the pavement surface and the falling of snow leading to dangerous driving conditions for motorists. And so while one may test the effects of temperature, and another may test the effect of de-icing chemical(s), this study will look at the effects of both cases combined. It used to be the idea that salt did not cause damage to bituminous surfacing, unlike concrete that is shown to have increased scaling as a result of the use de-icing salts Lyon, [6], however the combination of de-icing chemical and low temperature may prove different.

Standard testing procedures for the effects of both de-icing chemicals and freezing conditions is not available in the United Kingdom's British Standards Institute (B.S.I.). It was only in 2005 that the B.S.I. made available a draft standard (conceived in 2002) that was applicable to the resistance of hot mix asphalt to de-icing chemicals (prBS 12697 – 41: 2002). This draft outlines the cohesion test as the method to determine the resistance, or affect of de-icing chemicals B. S. I., [3]. However, the standard is only a draft, and therefore cannot be used as such. Although prBS 12697 – 41: 2002 does discuss how the samples should be cycled, it does not regard freezing of samples. Frost heave and soundness testing of aggregates (BS 812 – 124: 1989 & BS 812 – 121: 1989 respectively) does exist, and may help to test asphalt mixes. But as far, as testing asphalt (a load bearing material), tests that observe the change in mechanical properties are the most widely used, in particular axial loaded in-direct tests. Hassan et al., [5] tested the effects of freeze-thaw cycling and various de-icing chemicals with the In-direct Tensile Strength (ITS), but did not specify freeze-thaw cycling temperatures, and cycled samples in a manner that is not totally representative of the highway environment. However, is was shown how ITS tests would allow for the in-direct tensile stiffness modulus, and how these tests are good methods of observing effects of de-icing chemicals and repeated temperature fluctuations on asphalt.

The materials to be tested are only those that make up the surface course of the pavement, since it is this layer of the highway that is directly subject to the low temperatures, the application of the de-icing chemical, and subsequent penetration of de-icing chemical based fluids ingression into the asphalt. The types of surface course materials to be used are bitumen macadam's, and have been selected from a list of different macadam's being used for this and other research. The main aim of this study is to see what affect the simultaneous applications of freezing temperatures and de-icing chemicals in the winter have of asphalt surface course materials. The affect of low temperature will be taken forward to observe free-thaw cycling of the asphalt mixes. This will be achieved by:

- To see how the in-direct tensile stiffness modulus changes as temperature decreases.
- Observing the in-direct tensile strength of these asphalt materials during the frozen phase of the freeze-thaw cycle.
- Monitoring how the in-direct tensile strength of the asphalt materials changes when subjected to freeze-thaw cycles in the presence of de-icing chemicals.

2. EXPERIMENTAL METHODS

Samples for the tests are to be prepared in a gyratory compactor according to BS EN 12697 Part 31: Specimen preparation by gyratory compactor. When producing the samples, the compactor is to be set to reach to a specific density, pre-determined based on aggregate type, void content, and specimen height. Samples will be 100mm in diameter, with a target height of 62.5mm (to fall within the range for a Marshall sample).

The details referring to sample identification are given in Table 1. It should be noted that when it is stated in Table 1 that mix 5C has 100% filler substitution for carbon black, this means that the filler content and the particles of less than 75 microns in the dust content have been replaced with the carbon black. Sample preparation for the freeze-thaw test (see section 2.3) will be slightly different. Preparation using the gyratory compactor will be the same as outlined previously, however the samples will be made higher than the target of 62.5, as the top surface will be sawn off to expose aggregate, to make the sample more representative of the real world environment. Normal samples are totally covered in bitumen, and would not lend themselves particularly well to immersion in a salt solution (as will discussed in section 2.3). If the top surface of the sample is sawn off, the cross section reveals aggregate, and areas where there are voids, however the surface texture cannot be mimicked.

Table 1 Sample details and identification designation

Mix details	Sample identification		Notes
Mix 1 – 10mm close graded surface course materials	01AR	01AF*	The first letter in the sample ID refers to the number of cycles a sample has had, i.e 0 = no cycles, 1 = 5 cycles, 2 = 10 cycles, and 3 = 15 cyclesThe last letter of the sample ID refers to the test temperature. R stands for the closest standard temperature to room temperature – 20°C. The F represents a test temperature of –20°C. * In the case of In-direct tensile modulus tests, F refers to 0°C for reason that will be explained later.
	11AR		
	21AR		
	31AR		
Mix 3 – 14mm close graded surface course material	03AR	03AF*	
	13AR		
	23AR		
	33AR		
Mix 5 – 6mm stone mastic asphalt	05AR	05AF*	
	15AR		
	25AR		
	35AR		
Mix 5C – 6mm stone mastic asphalt with 100% filler substitution by carbon black	05CR	05CF*	
	15CR		
	25CR		
	35CR		

Figure 1 shows how the samples will look after the top surface was sawn off, and it can be seen that there is quite a large percentage of the sample face being visible aggregate, which should help the salt solution penetrate the aggregate and the sample as a whole. The type of aggregate to be used is an andesite, with bitumen of 125 pen. The reason for opting for 125 pen bitumen was due to the use of this grade in previous

tests that may be integrated with a test such as this in future. The bitumen will be prepared by mixing 50 pen and 200 pen bitumen at a ratio of approximately 30% 50 pen and 70% 200 pen bitumen according to equation (1).

$$\left(\frac{((A\times\log a)+(B\times\log b))}{100}\right)=\log X \tag{1}$$

where:

A = percentage of lower pen bitumen
a = lower pen bitumen in the composition
B = percentage of higher pen bitumen
b = higher pen bitumen in the composition
X = target bitumen penetration value



Figure 1 Sawn sample for free-thaw testing

2.1. Change in in-direct tensile stiffness modulus with decreasing temperature

British standard DD 213: 1993 'Methods for Determination of the in-direct tensile stiffness modulus of bituminous mixtures' will be used to determine the in-direct tensile stiffness modulus of the asphalt samples. The University of Ulster has a Nottingham Asphalt Tester (NAT) available to do this test. Samples will be tested at the standard test temperature of 20°C, however there will also be tests carried out at 0°C, but this does not changes set-up and test procedures. Before testing commences the samples are placed in a climate-controlled cabinet for at least 24 hours at the test temperature. This test will not entail cycling or immersion in a salt solution.

2.2. In-direct tensile strength test during the frozen phase

After preparation, the samples are stored for 24 hours prior to testing. After the initial 24 hours the samples to be tested at what will be termed room temperature, are put into a temperature controlled cabinet for a further 24 hours to reach a 20°C throughout, while the samples to be tested at –20°C will be placed in a freezer for the same period to reach –20°C. BS 12697 – 23: 2003, a test for the ITS forms a basis for this test, but due to the difference in test temperature and set temperature water immersion B. S. I., [4], the test is slightly different, but the principle remains the same. The test output yields a load vs. extension graph, that can be inserted to Microsoft Excel, and gives all data required to determine the ITS. The equation used to determine the ITS is given in equation (2).

$$ITS=\frac{2P}{\pi DH} \tag{2}$$

where:

ITS[1] = in-direct tensile strength, expressed in GPa, rounded to three significant figures.
P = peak load expressed in kN, rounded to three significant figures.
D = diameter of the specimen expressed in millimetres (mm), to one decimal place.
H = height of the specimen expressed in millimetres, to one decimal place

2.3. In-direct tensile strength of samples subjected to freeze-thaw cycling

As mentioned previously, this test requires the samples to be sawn at an angle that is perpendicular to the axis that runs through the centres of both faces of the samples. The test is made up of a cycling phase before ITS testing starts. The ITS test will be executed using the Zwick 100 materials testing machine. The cycling phase consists of a period of freezing and a period of thawing, both elements being carried out at constant temperatures. Due to the fact no standard methods exists for freeze-thaw testing of asphalt, BS 812 – 124: 1989 Testing aggregates Part 124: Methods for determination of frost heave will be used to plan what one cycle should entail. A cycling schedule will be put together in order to plan when cycling and testing can timetabled to coincide. The most important point when drawing up this plan is that the samples must be stored for 24 hours in a temperature controlled cabinet, although the period between the end of a cycling run and carrying out the ITS test must be kept to a minimum.

Figure 2 shows what occurs during two cycles. The diagram clearly illustrates that samples are stored at the lower temperature (-20°C) for a longer period of time (approximately 3 hours), while the duration of the cycle at 20°C only takes place over one hour. The most important part of the cycle is the cooling and warming trends, but Figure 2 shows that the cooling and warming times are equal. Prior experiments have shown that samples can reach 20°C from –20°C in this period. To suit laboratory access, and cycling, this study will use a cycle that lasts for 24 hours. The schedule will begin with a soaking stage at ambient temperatures (varied between 20°C and 23°C) lasting 12 hours (four hours of which will include the warming time thereafter). Samples will be stored in a large plastic container, and immersed in a salt solution. After twelve hours, the samples are taken directly from the solution without any drying and are placed a rigid plastic board that are then placed into a large chest freezer. The temperature inside the freezer was monitored and found to vary from –17.5°C to –20.5°C.

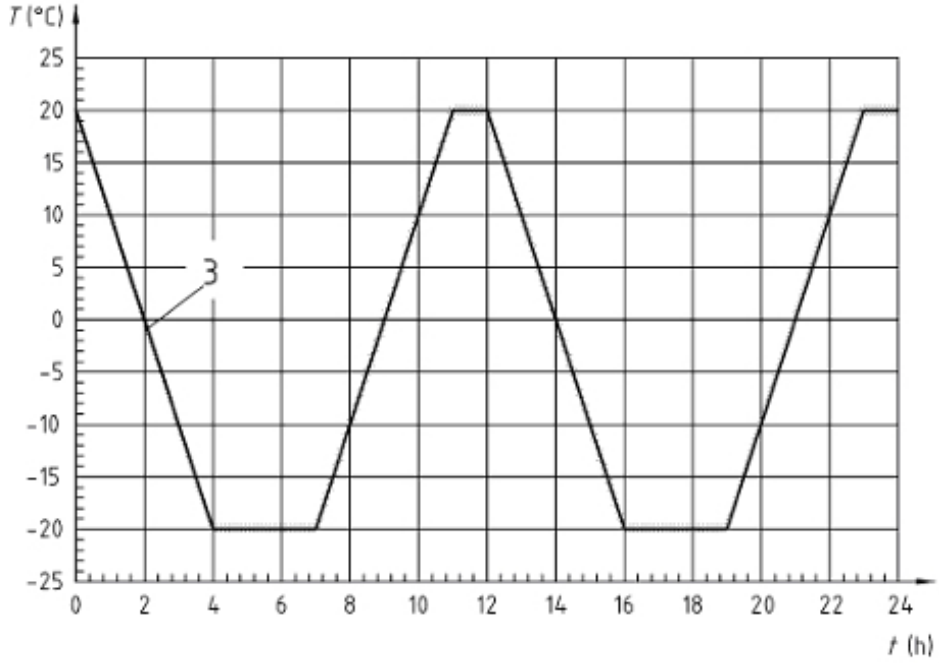

Figure 2 Cooling and heating chart. Source: B. S. I., [1]

[1] Note: ofr the purpose of this study, the ITS will be expressed in kPa as the same quantity expressed in Gpa is such a small quantity.

The schedule is shown in Figure 3, and shows each batch of samples will five, ten, and fifteen cycles respectively after prBS 12697 – 41: 2002 that uses 14 cycles. One possible concern is the inconsistency is the time elapsing after fifteen cycles with respect to five and ten. This could not be helped to fit in with the access to the Zwick 100. After each batch of samples has been cycled, the samples will be taken and dried in a climate-controlled cabinet for 24 hours at 20°C. Once normalization of the samples at 20°C has occurred, the samples are tested according to BS 12697 – 23: 2003.

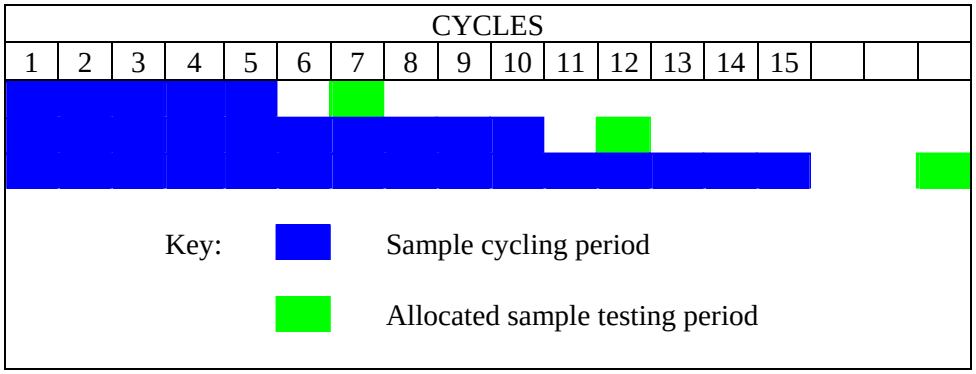

Figure 3 Sample cycling plan with testing periods

The concentration of salt in the solution will be 3.5% by weight of water. Hassan et al., [5] used concentrations from 1 – 50 %. However breakdown of the samples is not desirable as no testing would be possible, therefore a fairly conservative solution will be used. The solution with a 3.5% by weight concentration is approximately equal to using 8g/m^2 to de-ice thin ice films, and falls within the guidelines for salt spreading equipment according to BS 1642: 1989 Specification for spreaders for winter maintenance B. S. I., [2].

3. RESULTS AND DISCUSSION

3.1. In-direct tensile stiffness modulus tests

The test for in-direct tensile stiffness modulus was carried out first to get an idea of the mechanical properties of asphalt mixes at low temperatures compared to standard testing conditions. As lower temperatures were tested, it seemed that the lowest temperature that could be tested was 0°C. As the test temperature was reduced, there were difficulties in setting the strain gauges prior to testing, and even at 0°C it was hard to obtain reliable and sensible values for the in-direct tensile stiffness modulus. The climate-controlled cabinet housing of the NAT also did not react well to opening and closing of the access door, and so maintaining a stable temperature was quite difficult. It was these difficulties that eventually led to the use of the in-direct tensile strength test.

When testing with the NAT, we get a representative term of the stiffness of the asphalt. This is very important due to the fact that the stiffness is determined within the elastic range of the materials. The 'in-direct' nature of the test, coupled with the different test temperatures required the two different values for Poisson's ratio to be used. The standard value for testing at the standard temperature of 20°C is 0.35, however this reduces to 0.25 when the test temperature is reduced to 0°C.

It should be not that that when testing with the NAT and ITS machines, only strain in the diameter of the sample is used, but strain in the height of the sample is assumed to be zero.

It was always anticipated that samples would become more rigid as cooling happened, and this is shown to be evident in Figure 4, were samples tested at 0°C gave stiffness modulus values much than those tested at 20°C. In all samples tested, the in-direct tensile stiffness modulus was found to be in the order of five to six times higher when tested at 0°C than those tested at 20°C. The moduli of those samples tested at 0°C were so much higher that the NAT being used almost reached its maximum loading ability. It was not just the elevated loads that yielded the higher stiffness values, but in all cases there was much less deformation in the sample. However, although we now know that asphalt can become stiffer as temperature decreases, what is it exactly that causes the increase; is it the binder, the aggregate, or a combination of the two? It is for this reason that testing was taken further to include permanent deformation testing. The issues with machine

loading capacity and test temperature that ruled the Marshall Test, however the access to a materials testing machine (Zwick 100) with a 100kN load capacity, allowed for in-direct tensile strength tests to be carried.

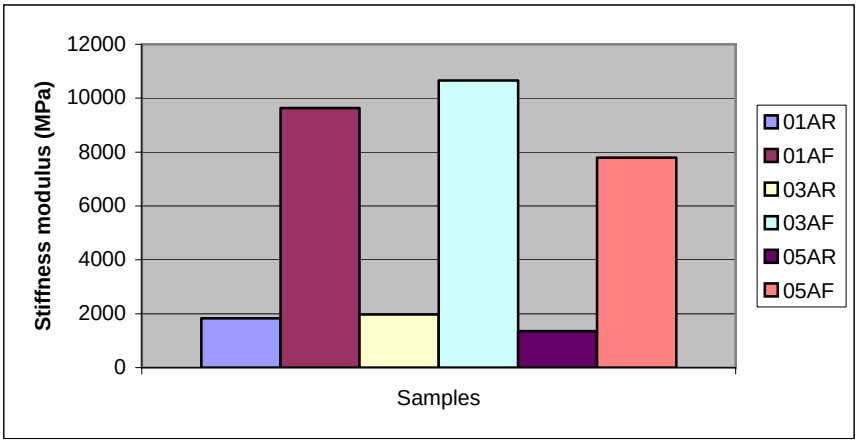

Figure 4 In-direct tensile stiffness modulus of samples tested at 20°C & 0°C

3.2. In-direct tensile strength tests at 20°C & -20°C

The ITS test is a very quick test to carry out. So although there was no temperature controlled cabinet around the machine, for the period of no more than a minute that it takes to carry out the test, there would be no appreciable change in the core temperature of the sample, let alone the surface temperature. This was particularly important when testing at –20°C, were there was a big temperature difference with respect to the ambient surrounding temperature that could result in a faster temperature change in the sample. When executing the tests, it was evident from just observing the tests that there were two clearly definable modes of failure. The first mode of failure was found in the samples tested at 20°C. Failure seemed to be quite plastic and uniform with only surface cracking occurring. When the samples that were being tested at –20°C the failure happened in a totally different way. As the load was increased, there was no visible deformation apparent in the sample, until a particular moment when the sample exploded, and parts of samples around the area of crack propagation splintered in shards.

The modes of failure can be seen quite clearly in Figure 5 and 6. The uniform plastic failure in the sample tested at 20°C shows that it was pure binder failure that occurred. Testing at 20C is not that much less than the softening point of the binder being used. This is evident as no aggregate failure was apparent. There were occasions when aggregate failure occurred around the periphery of the samples, but this may be due to stress concentrations around these areas. However, when failure occurred in the sample tested at –20°C, it seemed that the only reason failure happened was down to the strength of the aggregate. It seems that the as the binder has frozen, loads were transmitted into the aggregate, and in this respect the mix is behaving more as a composite. But the fact that the failure is so abrupt, where loading goes from elastic to instantaneous failure, with no appreciable plastic deformation, would suggest that the material becomes very brittle.



Figure 5 Mix 1 (01AR) tested at 20°C



Figure 6 Mix 1 (01AF) tested at -20°C

It is clearly visible from Figure 7 that the increase in strength follows a similar trend to Figure 4 with samples now being tested at –20°C exhibiting much higher ITS values. This would seem to suggest that the effect of decreasing temperature only will cease to affect the materials, i.e. increasing modulus will stop increasing at a certain temperature drop.

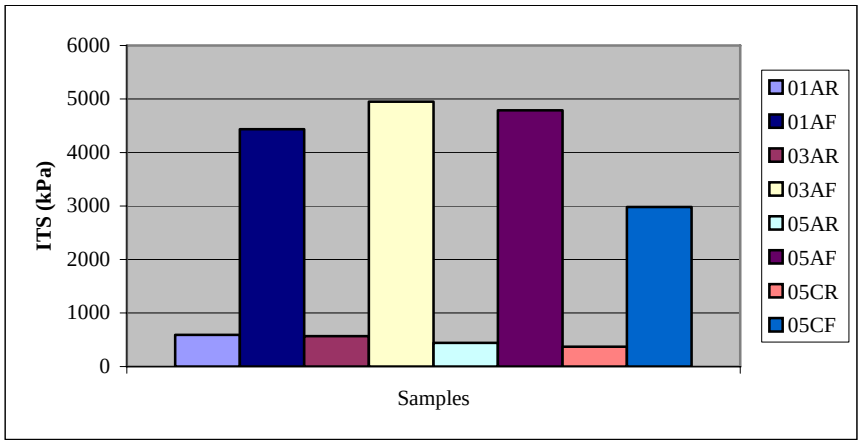

Figure 7 In-direct tensile strength of samples tested at 20°C & -20°C

Figure 8 shows how the decrease in the extension of the samples at which peak loads occurred, showing the brittleness of the material at sub-zero temperatures. It also shows how sudden the failure in the frozen samples was. It is the higher peak loads, and the slightly decreased extension that makes suggests a more brittle material, and the possibility of poorer fatigue strength. Fatigue can be observed in a number of ways, for example repeated loading or cyclic loading, but a repeated freeze-thaw cycles also sets up a fatigue condition.

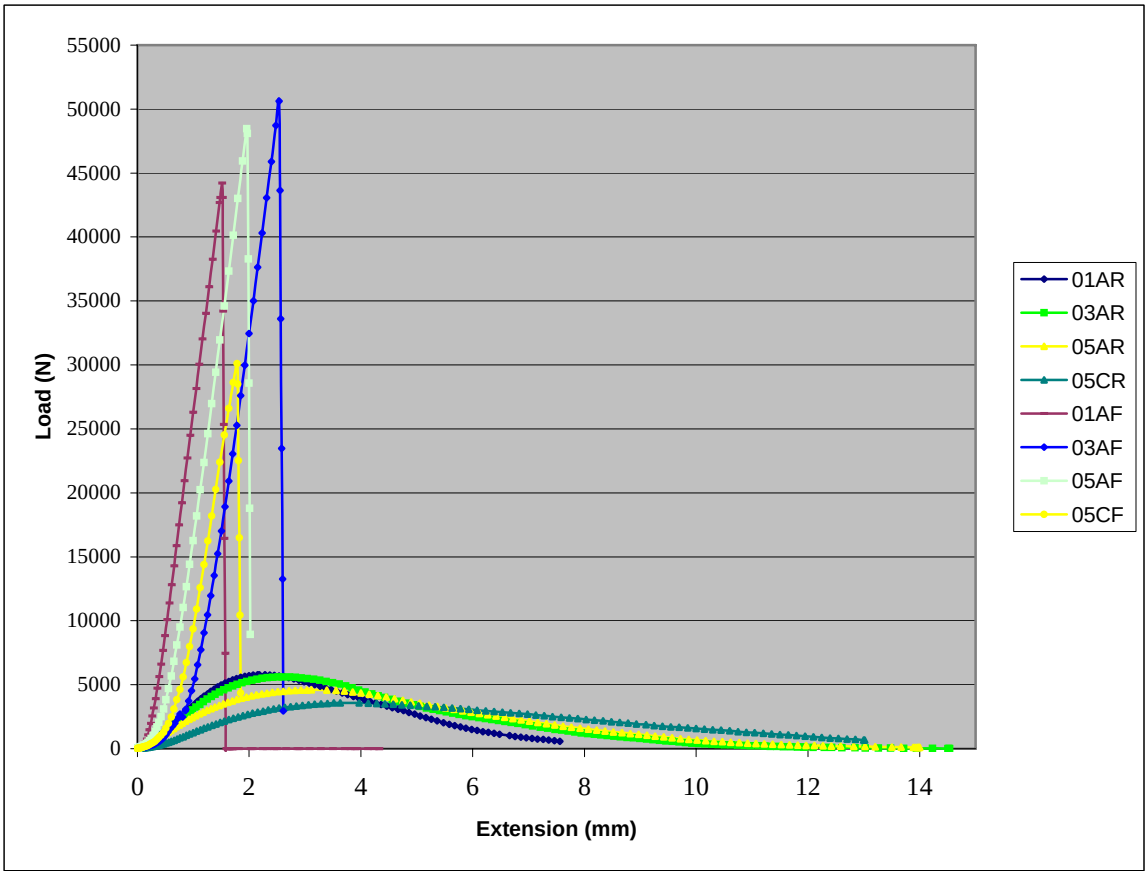

Figure 8 Load vs. deflection curves of samples tested at 20°C & -20°C

3.3. In-direct tensile strength tests with freeze-thaw cycling

When carrying out the freeze-thaw cycling and testing began, possibilities of what the ITS test would yield was anticipated. The reason for exposing one face of the sample was to make contact between the salt solution and all elements of the asphalt mix. Certainly material loss (both binder and aggregate particles), and aggregate dissolution were possibilities as to what may cause any change in the strength of the sample. Indeed, after each set of five cycles, particularly from ten cycles onward, there was a noticeable quantity of debris deposited in the cycling containers. However, it was not possible to analyse this debris to see what the main constituents were, but visual inspection suggested that binder and filler particles where two elements of the mix present, due to size and darkness in colour. The debris left behind from the samples containing the carbon black seemed to be higher that any other mix, and this may be due to the fineness of the carbon black particles.

Figure 9 shows what is expected as a result of the void content, as mentioned previously the surface of the sawn samples was smooth to the touch, except for the voids. However after fifteen cycles the surface of the samples changed. Figure 10 shows the surface deterioration that caused the debris to be deposited in the cycling containers as mentioned previously. Another defect found on the surface of the samples was slight heaving of aggregate on the surface, which led in turn to the deliberation as to what may be going on at the core of the sample. The ‘heaving’ effect’ was apparent due to a roughing of the sample surface in the form of protruding aggregate, though this was only evident in the ‘dust’ content distribution of aggregate. Therefore, one may ask what exactly was happening to the ITS.

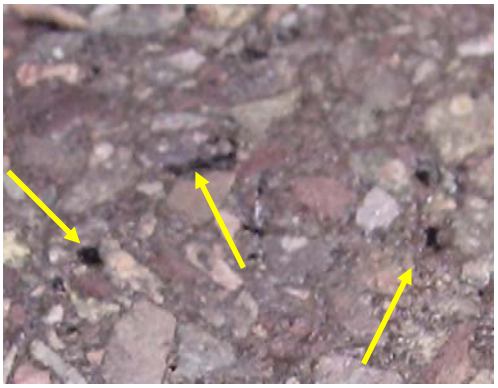

Figure 9 Visible voids on samples surface

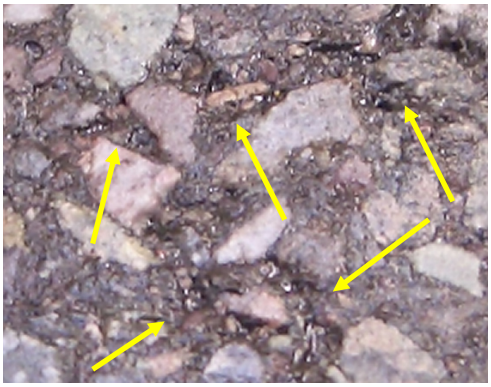

Figure 10 Surface erosion on sample

Figure 11 to 13 show how there is a noticeable decrease in the peak load of approximately 35% and a 23% decrease in the extension at peak load, and this trend was seen in all asphalt mixes tested. Although all samples showed a decrease in peak load, how the samples deformed was different, particularly with the SMA materials that became slightly more elastic to increased deflection at peak load. It is thought that as the salt solution penetrated into the sample, as the solution began to freeze, the solution would have expanded setting up stresses within the sample that may have caused cracking to occur in the binder and binder-aggregate interface. Cracking at the binder-aggregate interface may have been increased as the aggregate ‘thermally’ contracted as the temperature decreased. However this assumption would need further investigation. When the last batch of samples was tested after fifteen cycles however (Figure 14), there was an increase in the peak load at failure, and subsequent ITS values. But what could have caused this? Well, when the testing schedule was drawn up, there was a longer gap in between the end of cycling and ITS testing, and in this time there may have been recovery of the samples. In no instance did any sample regain 100% of its original strength, but the unmodified SMA (mix 5A) came very close. This trend would then raise the quest ‘To what extent can bituminous materials regain strength after freeze-thaw cycling in the presence of de-icing chemicals’?

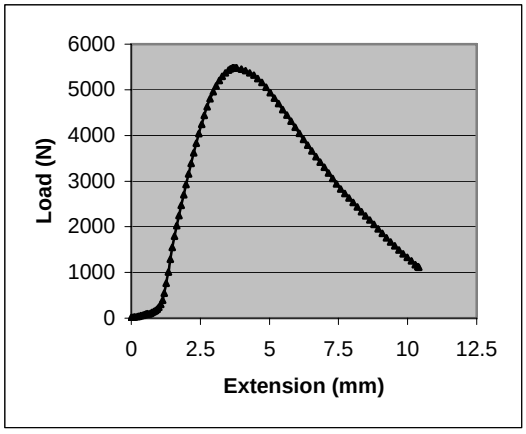

Figure 11 Load vs. deflection curve of mix 1 (01AR) prior to cycling

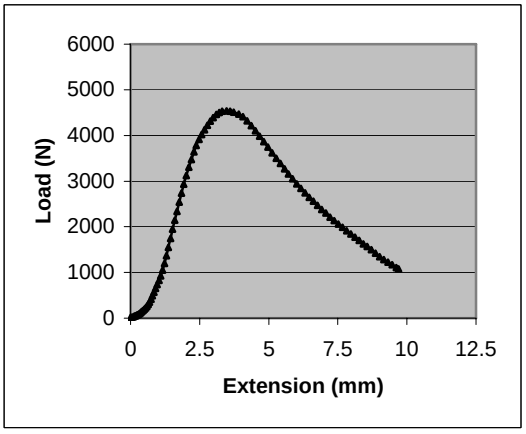

Figure 12 Load vs. deflection curve of mix 1 (11AR) at five cycles

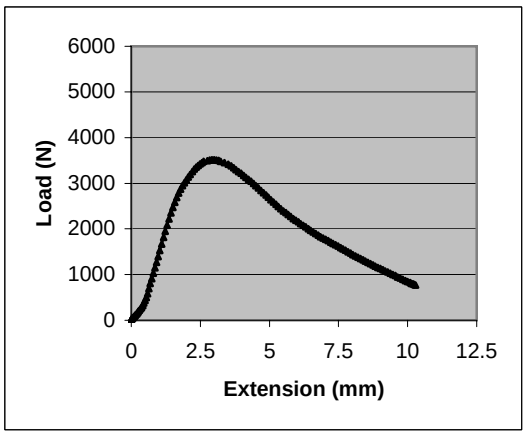

Figure 13 Load vs. deflection curve of mix 1 (21AR) at ten cycles

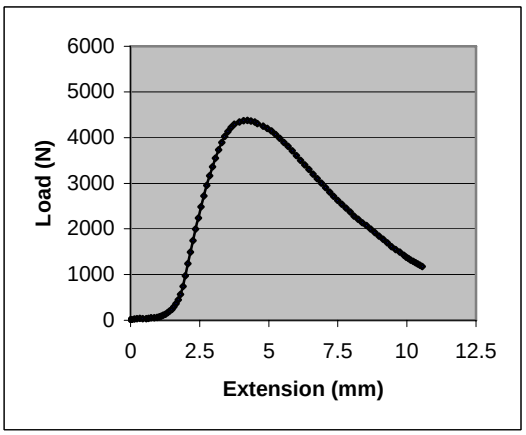

Figure 14 Load vs. deflection curve of mix 1 (31AR) at fifteen cycles

Figure 15 shows graphically how the ITS began to decrease and increase toward the end of cycling. What cannot be seen however is the how is the affect of straining in the mix as would be seen with in-direct tensile modulus test, since a decrease in the extension at peak load was experienced as seen in figures 11 to 14.. Figure 16 goes further to show how this trend occurred in asphalt mixes being tested. Further testing would need to be carried out, cutting down the time between the cycling and ITS testing to see if it was the extended gap that caused the recovery in the materials after fifteen cycles.

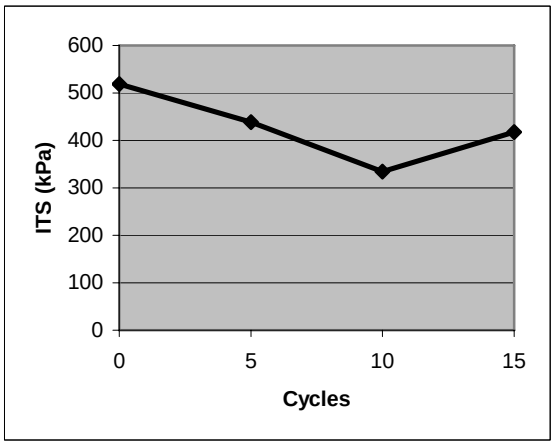

Figure 15 ITS of mix 1 throughout test cycling

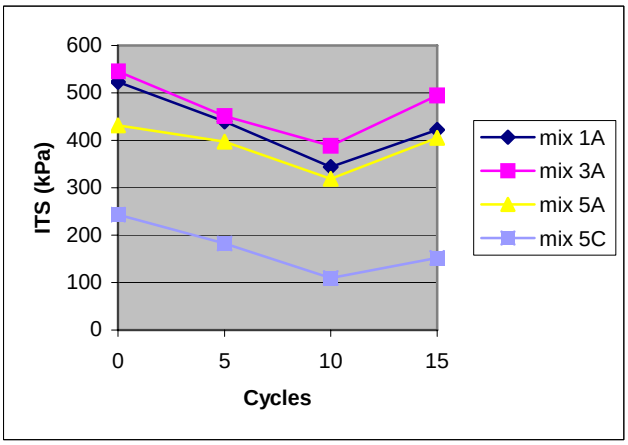

Figure 16 Mean ITS trends for all mixes

4. CONLUSIONS

The study looked at three similar tests used to observe properties of asphalt mixes at low temperature and exposure to de-icing chemicals to get a view of what happens to the pavements (primarily made from bitumen macadam's in the wintertime. The study did show that the strength of asphalt materials does decrease as applications of de-icing chemicals and freezing conditions increase. The ITS decreased by between 25% and 55% at the end of the cycling period.

The unexpected outcome was the level of recovery in the samples when the materials were allowed to normalise to normal conditions. However, the use of the de-icing chemical in the liquid did not seem to have any specific affect. The need to determine why the ITS decreased as the number of freeze-thaw cycles increased would need to be further investigation, perhaps by monitoring strain in the material over the cycling period. If the strain were to be measured, one would be able to see any permanent deformation occurring a result of freeze-thawing in the presence of freezing fluids on the pavement surface without having to carry out permanent deformation testing like the ITS test. It would also be useful to see any relationship between aggregate soundness and free-thaw based ITS tests.

The tests carried out here would definitely suggest a benefit in maintaining a higher pavement temperature in freezing climates where there is the presence of fluids. However, to observe the sole effect of the chemicals being used, particle loss (as experienced here) and aggregate stripping should be investigated to see what the sole effect de-icing chemicals are. There are so many variables that can be included in a study such as this (for example; chemical type, de-icing chemical solution saturation, number of cycle, and temperature change), one must decide what set of parameters best suits the situation that is to be simulated. The ITS test is a quick and highly effective test that allow much about the asphalt material to be known (from an in-direct tensile point of view).

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