

Improved Low-Temperature Fracture Performance for Rubber-Modified Asphalt Binders

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The low-temperature fracture toughness was determined for rubber-modified asphalt binders. Crumb rubber tire, both plain and surface modified, and devulcanized rubber tire were investigated for their effectiveness in improving low-temperature asphalt binder performance in a notched, three-point bending beam fracture test. The increases in low-temperature fracture toughness for unmodified crumb rubber-asphalt mixtures were highest for fine ground rubber. At loading levels of 4 to 10 wt percent on the binder, the fracture toughness for 10- and 20-mesh ground rubber tire-modified samples was not significantly different from that of the unmodified binders. Reasonable increases in toughness were found for binder modification with 4 to 10 wt percent 30-, 40-, and 80-mesh rubber samples. An in situ sulfur ygrafting reaction of low-molecular-weight polybutadiene onto the crumb rubber dramatically increases the fracture performance for both coarse and fine crumb rubber-modified binders. A 9- and 15-fold increase in added fracture toughness was observed for binders containing 7 wt percent of 10- and 20-mesh cryogenically ground rubber tire. Whether these improvements will translate into a reduction in transverse thermal stress cracking remains to be investigated in further tests on binder-aggregate mixes and in field trials. The fracture toughness for a devulcanized rubber-modified asphalt was also investigated. The homogeneous sample contained as much as 10 wt percent devulcanized rubber tire but performed only marginally better than an 85-100 penetration control sample. Reaction of the devulcanized rubber-modified binder with sulfur increases its fracture toughness by 18 percent.

For more than 25 years discarded rubber tires have been used in asphalt binders to improve the low- and high-temperature performance of the road surface (1,2). During hot summer months the rubber improves the rheological performance of the asphalt binder, and this may result in a pavement with better rutting resistance and durability (3). The rubber tire can lower the stiffness of the binder, and as a result this reduces the brittle temperature (4). Advocates of this technology have often been "suggesting that this material will perform noticeably better at low temperatures, compared with standard asphalt concrete" (5). However, field trials to look for a possible reduction in transverse thermal stress cracking have so far been disappointing (6,7) and have been inconclusive in their judgment on the performance/cost benefits of these materials. (8).

The development of rubber-modified asphalt pavements has not always been driven solely by economic concerns; environmental agencies and political forces have been strong advocates for the disposal of rubber tire waste in "linear landfills" (i.e., asphalt pavements). Research in this area has often been promoted by government agencies that are concerned with reducing the estimated 2 billion to 3 billion discarded rubber tires that are believed

to be stockpiled in North America alone (9). This number is growing by approximately 200 million tires each year, which suggests that the waste rubber tire issue will not go away easily. Moreover, it is regularly brought back to the attention of the public when a tire disposal site catches fire, resulting in significant air and water pollution and high costs associated with the cleanup.

The escalation of the scrap tire storage problem has led the United States government to pass legislation forcing the state governments to use waste rubber tire in projects supported by federal funds. The Intermodal Surface Transportation Efficiency Act of 1991 (ISTEA) (Section 1038) requires that states use waste rubber tire in asphalt pavements receiving government funding starting in 1994. The act stipulates that by 1997 all states that receive government funding should use approximately 15 percent rubber tire waste on the asphalt binder in 20 percent of their projects to remain eligible for such governmental support.

Whether this legislation will have the desired effect (i.e., increased usage of recycled rubber in asphalt pavements with an accompanying reduction in waste tire stockpiling) remains to be seen. However, the law has been a strong incentive for significant research and development efforts in this area. A recent survey conducted by Villanova University found that 38 states are currently using scrap rubber tire in their pavements (10). However, there are still many concerns and unanswered questions regarding the use of recycled rubber tire in large-scale paving practice.

BACKGROUND

C. H. McDonald, a materials engineer in the roads department of Phoenix, Arizona, pioneered the development of rubber-modified asphalt binders as they are now most often used throughout North America and in many other countries. Since the developments in the late 1960s by McDonald and coworkers, a number of variations on the initial technology have been developed, often with mixed results in terms of performance enhancement.

In the "wet process" as it was developed by McDonald, 14 to 20 wt percent ambiently ground rubber tire (8 to 20 mesh) is swollen in the asphalt's oily phase at high temperatures to form a gellike material after mixing for approximately 45 min at 175°C to 220°C. To this gel is added some aromatic kerosene fraction to increase its workability. This asphalt-rubber is mixed with aggregate to form the pavement. The process requires at least 20 percent more liquid asphalt than is used in a conventional hot-mix pavement. In some cases 40 to 60 percent more asphalt is used in the mix, which may in part account for the increase in both cost and performance (11). The thicker films of asphalt at the aggregate

interface result in greater durability. These asphalt-rubber binders also have higher softening points, which can result in less bleeding and permanent deformation. Moreover, a 70 to 90 percent reduction in traffic noise has been observed on pavements with asphalt-rubber mix overlays compared with conventional pavements (12). Because of the substantially higher initial cost of the wet process, the technology has been primarily used for the control of reflective stress cracking. Asphalt-rubber has found applications in stress-absorbing membranes, in stress-absorbing membrane interlayers, as a crack and joint sealer, and to a lesser degree as a binder in thin asphalt concrete overlays (13).

Another method for incorporating recycled rubber crumb into asphalt pavements that has received considerable attention is the "dry process." It was originally developed in Sweden under the trade name Rubit (14) and subsequently registered in the United States under the trade name Plusride. It differs from the wet process in that the rubber crumb is slightly larger ($1/16$ to $1/4$ in.) and is directly mixed with the aggregate before the asphalt binder is added. The rubber is added at a loading of 3 to 4 wt percent of the aggregate. This process requires a special aggregate gradation to avoid any interference of the rubber crumb with the aggregate, which can lead to premature stripping. In addition, this process also calls for typically 1.5 to 3 percent more liquid asphalt than a conventional hot mix (15,16). The increased asphalt content is needed to achieve a voids content of less than 3 percent to prevent premature raveling of the pavement (17). In cold climates the Plusride technology has been used successfully to reduce the harmful effects of ice formation on roads. A 25 percent reduction in stopping distances on ice-covered roads has been recorded for pavements made by this process compared with conventional hot-mix pavements (18,19). Takallou (20) has developed a dry process that uses conventional aggregate gradations by specifying finer crumb rubber grades. The process, which is known under the Generic name, has been used in a number of paving test sections throughout the United States (21).

Another variation of the dry process has recently been investigated by the United States Army Corps of Engineers at the Cold Regions Research and Engineering Laboratory (CRREL) (21). CRREL has investigated the use of much larger quantities of a coarse rubber crumb in hot-mix pavements. The crumb rubber was added at 3 to 12 wt percent of the aggregate, compared with typically 3 wt percent for the conventional Plusride or Generic processes. The optimum binder content in the mix increases with the amount of crumb rubber added. By using such large quantities of rubber crumb in an asphalt pavement, one gets rid of a lot of scrap tire. However, the added cost of the rubber and the higher binder content of the pavement, the extra cost associated with the use of equipment to handle such large quantities of crumb rubber, and the added mix design and production complexity may increase the overall pavement cost by as much as 150 percent over that of an unmodified mix.

The fact that the cost of rubber-modified hot-mix pavements, in general, is currently anywhere from 60 to 150 percent more than the cost of a conventional asphalt pavement has deterred many municipal and state governments from using this technology. Today, asphalt-rubber is mainly used in low-volume applications such as crack and joint sealants, stress-absorbing membranes, and stress-absorbing membrane interlayers.

Concern about high initial costs combined with uncertainty about future benefits has hindered the large-scale acceptance of asphalt-rubber technology. It may be more sensible to use less

crumb rubber to lower the initial cost and make the technology more affordable. If modest amounts (4 to 10 wt percent) of fine crumb rubber are applied to the asphalt binder, the pavement may be constructed with normal binder contents, which would result in only a slight overall increase in cost. This by itself may make the technology more widely acceptable, which could eventually mean that much more scrap tire will be used in hot-mix asphalt pavements.

Another concern relates to the difficulties involved with the recycling of asphalt-rubber pavements as they have been constructed until now. The high rubber content of asphalt-rubber binders may complicate the recycling of old pavements and could possibly create a serious solid waste problem in the years ahead. This would ultimately defeat the purpose of the ISTEA legislation. To facilitate hot-mix recycling of the pavement, it may be necessary to use less crumb rubber in the asphalt binder.

Recently, paving trials in Florida (22) and Ontario (23) have used binders containing only 7 to 9 percent fine crumb rubber (80 mesh) directly blended into the asphalt cement. Initial laboratory test results are promising, but it is too early to draw any conclusions from the performance of these trial pavement sections (24).

The work presented in this paper investigates fundamental fracture properties of a number of such recently developed asphalt binders containing only modest amounts of recycled rubber. Moreover, new products are developed by reacting the crumb rubber in situ with the asphalt phase to improve interfacial adhesion and stability.

A commercially available homogeneous asphalt binder containing devulcanized rubber tire is also investigated for its fracture performance. This binder contains 10 wt percent rubber tire and is marketed under the trade name Ecoflex. In the Ecoflex process, the crumb rubber is mixed with the asphalt and the binder is subsequently heated for up to 5 hr at 220°C to 260°C while compressed air is blown through to break down the rubber and facilitate dissolution (25). The company that produces this product states that its price is only 3 to 5 percent higher than conventional hot-mix asphalt binders with an increase in performance over unmodified products (26). If true, this may provide an incentive for the disposal of large quantities of scrap tire by devulcanizing them according to the Ecoflex process. However, it is doubtful that a process using such high temperatures and such harsh oxidative treatment will ever become acceptable.

EXPERIMENTAL

Materials

The asphalts used in this study were an 85-100 penetration grade and a 150-200 penetration grade, both from the Bow River area in Alberta, Canada. The 85-100 penetration grade control sample has been used in the U.S. Strategic Highway Research Program as a core asphalt under code AAN. Both asphalt samples were obtained from the Lake Ontario refinery of Petro-Canada in Oakville, Ontario.

The wet-ambiently ground scrap passenger car tire was obtained from Rouse Rubber, Inc., of Vicksburg, Mississippi. The 10-, 20-, 30-, and 40-mesh cryogenically ground passenger car tire samples were obtained from Recovery Technologies, Inc., of Mississauga, Ontario. The sample of 20-mesh ambiently ground tire for the preparation of the wet process asphalt-rubber binder was obtained

from Baker Rubber, Inc., of South Bend, Indiana. The Ecoflex sample containing 10 percent devulcanized scrap tire was obtained from Bitumar, Inc., of Montreal, Quebec.

The liquid polybutadiene (LPBD) was obtained from Ricon Resins, Inc., of Grand Junction, Colorado. Its conformation is 80 percent 1,4-trans and 1,4-cis and 20 percent 1,2 vinyl, and it has a number average molecular weight of 12,000 g mole⁻¹.

Procedures

Sample Preparation

The rubber-modified asphalt binders were prepared by slowly adding a known amount of rubber to the asphalt at 170°C ± 10°C. Additional LPBD and sulfur were added to a number of the samples. The liquid butadiene was reacted with sulfur in the hot asphalt binder to facilitate a reaction between the asphalt and the crumb rubber particles. The asphalt molecules graft onto the polybutadiene to produce an asphalt-compatible polymer (27). The polymer can at some point anchor itself onto the crumb rubber surface through a sulfur cross-linking reaction. This then strengthens the rubber-asphalt interface, and a much tougher asphalt binder can be obtained.

The mixtures were processed under moderate shear using a laboratory mixer (Polytron Mixer, Brinkman Instruments) for at least 2 hr, after which the samples were poured into silicone molds for fracture testing.

Low-Temperature Fracture Testing

The reacted rubber-modified binders were poured into silicone molds and subsequently left in a freezer at -20°C for a minimum of 18 hr before testing. The samples were tested using a three-point bending test based on ASTM E399-90. Testing was done in a temperature-controlled environmental chamber maintained at -20°C using a computer-interfaced Sintech 2/G testing frame. The sample bars measured 25 mm wide by 12.5 mm deep by 175 mm long and had a 90-degree starter notch 5 mm deep in their center, which was sharpened with a razor blade just before testing. The length of the loading span was 100 mm.

Brittle fracture studies were completed for all rubber-modified samples and the 85-100 and 150-200 penetration control asphalts. Measured data included the failure load and the modulus. The fracture toughness, K_{Ic} , was calculated according to Equation 1 (28,29).

$$K_{Ic} = \frac{P_f S}{BW^{3/2}} \times \left\{ \frac{3 \left(\frac{a}{W} \right)^{1/2} \left[1.99 - \frac{a}{W} \left(1 - \frac{a}{W} \right) \left(2.15 - 3.93 \frac{a}{W} + 2.7 \frac{a^2}{W^2} \right) \right]}{2 \left(1 + 2 \frac{a}{W} \right) \left(1 - \frac{a}{W} \right)^{3/2}} \right\} \quad (1)$$

where

K_{Ic} = fracture toughness (N m^{-3/2}),

P_f = applied failure load, (N),

S = loading span (m),

B = specimen depth (m),

W = specimen width (m), and

a = crack length (m).

For each composition between 15 and 25 beams were fractured. At -20°C, all samples broke in a brittle mode. The standard deviation of the mean and the number of samples were used to calculate a 90 percent confidence limit according to *t*-test statistical methods (30).

Lee and Hesp (31) give a more detailed discussion of the use of linear elastic fracture toughness (K_{Ic}) measurements in modified binders to study the fundamental fracture toughening mechanisms in polyethylene-modified binders.

RESULTS AND DISCUSSION

The low-temperature fracture data for ground rubber tire-modified asphalt binders (with 90 percent confidence limit) are given in Tables 1 to 3. Various quantities of wet-ambiently and cryogenically ground rubber tire in the 80- and 40-mesh size range were added to Bow River 150-200 penetration grade asphalt. The samples were also reacted with minor amounts of LPBD and 1 wt percent sulfur. Table 4 gives the results for a series of differently sized ground rubber tire samples tested at 7 wt percent crumb rubber, with and without reactive processing with 2 wt percent LPBD and 1 wt percent sulfur.

To distinguish between the effect of the dissolved from the interfacially reacted LPBD on the fracture toughness, control samples of 150-200 Bow River asphalt containing 2, 4, and 6 wt percent LPBD were also tested. The results for the effect of dis-

TABLE 1 Fracture Toughness of 40-Mesh Cryogenically Ground Rubber Tire-Modified 150-200 Bow River Asphalt Binders

Ground Rubber (wt %)	LPBD (wt %)	Modulus (GPa)	K_{Ic} (kN m ^{-3/2})
0	0	0.76 ± 0.09	64.9 ± 7.1
4	0	0.79 ± 0.05	75.9 ± 3.6
7	0	0.85 ± 0.09	80.3 ± 2.1
10	0	0.79 ± 0.04	100.6 ± 2.6
4	2	0.90 ± 0.03	89.7 ± 4.3
7	2	0.88 ± 0.08	104.4 ± 3.4
10	2	1.02 ± 0.09	118.9 ± 3.3

TABLE 2 Fracture Toughness of 40-Mesh Wet-Ambiently Ground Rubber Tire-Modified 150-200 Bow River Asphalt Binders

Ground Rubber (wt %)	LPBD (wt %)	Modulus (GPa)	K_{Ic} (kN m ^{-3/2})
0	0	0.76 ± 0.09	64.9 ± 7.1
4	0	0.74 ± 0.05	76.6 ± 3.3
7	0	0.82 ± 0.09	81.4 ± 4.4
10	0	0.92 ± 0.10	101.5 ± 5.7
4	2	0.99 ± 0.08	85.6 ± 4.2
7	2	1.02 ± 0.17	114.1 ± 3.8
10	2	1.11 ± 0.09	140.7 ± 5.4

TABLE 3 Fracture Toughness of 80-Mesh Wet-Ambiently Ground Rubber Tire-Modified 150-200 Bow River Asphalt Binders

Ground Rubber (wt %)	LPBD (wt %)	Modulus (GPa)	K_{Ic} ($\text{kN m}^{-3/2}$)
0	0	0.76 ± 0.09	64.9 ± 7.1
4	0	0.79 ± 0.05	77.3 ± 5.2
7	0	0.68 ± 0.07	96.0 ± 4.1
10	0	0.53 ± 0.04	120.1 ± 3.2
4	2	0.85 ± 0.08	100.6 ± 5.7
7	2	0.65 ± 0.05	119.3 ± 9.6
10	2	0.62 ± 0.04	137.3 ± 6.4

solved polybutadiene on the fracture toughness of 150-200 penetration grade asphalt are given in Table 5.

From these results, it can be concluded that for each 1 percent LPBD added to the 150-200 Bow River asphalt, the fracture toughness increases by approximately $4.2 \text{ kN m}^{-3/2}$. With this information, it is now possible to show the impact that the interfacial reaction between the polybutadiene and the rubber particles has on the fracture toughness of the binders. Figure 1 shows the added benefit to the fracture toughness imparted by the crumb rubber, the LPBD, and the interfacial reaction between the polybutadiene and the rubber particles, for 10-, 20-, 30-, and 40-mesh cryogenically and 80-mesh wet-ambiently ground samples. These results show that the interfacial modification is beneficial in all samples. However, the effect is largest for the coarse crumb rubber-modified binders. The unreacted 10- and 20-mesh crumb rubber-modified binders do not show any significant improvement in fracture toughness over a 150-200 Bow River control binder. However, when the interface is strengthened, the toughness is vastly improved. The gain in fracture toughness for the interfacially modified versus the unmodified 10-mesh sample was ninefold. For the reactively processed 20-mesh sample the toughness increase due to the crumb rubber was as much as 15 times higher than for the unreacted sample. For the 30-mesh sample the added benefit in fracture toughness due to the interfacial modification was 200 percent, for the 40-mesh sample it was 100 percent, and for the 80-mesh sample it was 50 percent.

As a comparison, a 20-mesh ambiently ground rubber tire sample was mixed according to the wet process (20 wt percent rubber mixed for 45 min at 190°C). The fracture toughness for this material was determined to be $164.0 \pm 4.4 \text{ kN m}^{-3/2}$, which is higher

TABLE 4 Fracture Toughness of 10-, 20, 30-, and 40-Mesh Cryogenically Ground Rubber Tire-Modified 150-200 Bow River Asphalt Binders

Ground Rubber (wt %)	Mesh size	LPBD (wt %)	Modulus (GPa)	K_{Ic} ($\text{kN m}^{-3/2}$)
0	-	0	0.76 ± 0.09	64.9 ± 7.1
7	10	0	0.95 ± 0.12	66.8 ± 4.8
7	10	2	0.88 ± 0.11	93.6 ± 4.9
7	20	0	0.88 ± 0.08	67.2 ± 2.5
7	20	2	1.01 ± 0.06	105.3 ± 5.7
7	30	0	0.84 ± 0.07	81.0 ± 4.3
7	30	2	1.05 ± 0.08	107.9 ± 4.1
7	40	0	0.85 ± 0.09	80.3 ± 2.1
7	40	2	0.88 ± 0.08	104.4 ± 3.4

TABLE 5 Fracture Toughness of LPBD-Modified 150-200 Bow River Asphalt Binders

Polybutadiene (wt %)	Modulus (GPa)	K_{Ic} ($\text{kN m}^{-3/2}$)
0	0.76 ± 0.09	64.9 ± 7.1
2	0.88 ± 0.05	71.3 ± 3.8
4	1.01 ± 0.21	84.9 ± 5.1
6	0.95 ± 0.11	90.4 ± 6.2

than all the samples in Tables 1 through 4. However, some of the reacted 10 percent crumb rubber samples come close to this level of toughness.

Finally, a devulcanized rubber tire-asphalt sample was tested for its low-temperature fracture toughness. This asphalt has an 85-100 penetration grade, and since the base asphalt for this modified material was unknown, it was compared with a Bow River 85-100 penetration grade control sample. The devulcanized sample was also reacted with 1 wt percent sulfur at 170°C for 2 hr to increase the molecular weight of the devulcanized rubber. As indicated in Table 6, the fracture toughness of the devulcanized sample is only marginally better than the 85-100 Bow River control sample (or any other unmodified binder for that matter). Reacting the devulcanized rubber with sulfur increases the fracture toughness by about 18 percent, but the performance is still far below that of the (vulcanized) crumb rubber-modified binders. However, devulcanized rubber may be an ideal material to use in the interfacial reaction between crumb rubber and asphalt. The low price of this material makes it a much more attractive reactive agent than the expensive LPBD used in this work.

However, one advantage of the devulcanized rubber-asphalt systems is that they possess long-term storage stability at high temperatures. The dissolved rubber does not phase separate from the binder during storage or transport. Whether the storage stability of crumb rubber-modified binders can be improved through interfacial modification has to be investigated further.

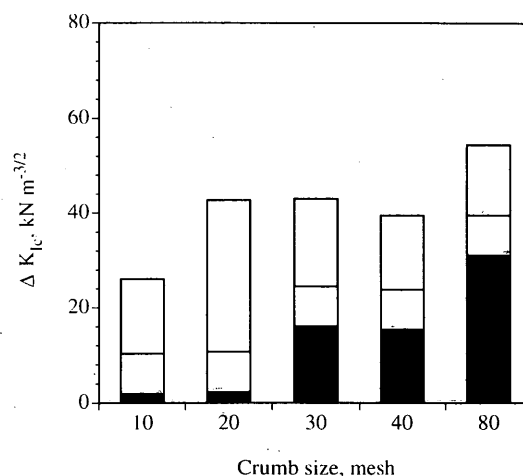
**FIGURE 1 Added benefit to the low-temperature fracture toughness of crumb rubber-modified 150-200 Bow River asphalt binders (black, effect of unmodified crumb rubber; white, contribution of 2 percent LPBD; gray, effect of improved interfacial adhesion).**

TABLE 6 Fracture Toughness of Devulcanized Rubber Tire-Modified Asphalt Binders

Asphalt	Additive	Modulus (GPa)	K _{Ic} (kN m ^{3/2})
85-100	-	1.35 ± 0.11	44.1 ± 3.9
Ecoflex ^a	10 wt % rubber	1.14 ± 0.17	55.8 ± 6.1
Ecoflex	10 wt % rubber + 1 wt % sulfur	1.15 ± 0.12	66.4 ± 4.6

^a Product of Bitumar Inc. of Montreal, Quebec.

CONCLUSIONS

Plain 10- or 20-mesh crumb rubber does not improve the low-temperature fracture toughness of a 150-200 penetration grade Bow River binder at additive contents as high as 10 wt percent. The gain in fracture toughness due to interfacial modification in such systems can be very significant.

Plain 30-, 40-, and 80-mesh crumb rubber tire samples increase the fracture toughness of the binder. However, in these samples, an interfacial strengthening can still give substantial improvements.

Whether the results obtained will translate into improved low-temperature thermal stress cracking resistance has to be investigated further with mix tests developed under the Strategic Highway Research Program and in field trials.

A commercially available devulcanized rubber tire-asphalt binder did not show any significant improvement in fracture toughness over that of an unmodified 85-100 Bow River control sample. However, increasing the molecular weight of the rubber through a sulfur vulcanization reaction increased the fracture toughness by 18 percent.

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