

Low-Temperature Fracture Toughness of Polyethylene-Modified Asphalt Binders

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One of the key factors that determines the susceptibility of a pavement to thermal cracking is the ability of the binder to withstand or relieve thermal stresses before they reach a critical point where cracks form. The mechanisms by which the addition of polyethylene improves the low-temperature properties of asphalt binders are discussed. Also examined is the degree to which the quality of the additive dispersion influences these mechanisms. Notched bending beam tests conducted at -20°C yielded fracture toughness values for three different polyethylene-asphalt systems. In all systems studied, the fracture toughness was found to increase linearly with the additive content. The chlorinated polyethylene-modified binders produced the toughest samples, followed by the stabilized polyethylene and the unstabilized polyethylene binders, respectively. High-magnification photographs, made with an environmental scanning electron microscope, yielded additional information concerning the nature of the failure mechanisms in these systems. The combined results show that toughening occurs with more finely dispersed polymers and with greater compatibility at the interface between the polymer and the asphalt matrix. These observations are consistent with the mechanisms of crack pinning and shear yielding, both of which are regularly found in multiphase thermoplastic materials.

One of the most promising methods of improving asphalt performance at both low and high service temperatures is by using additives. Of the many additives investigated, polymers show the greatest versatility in modifying the structural and adhesive properties of asphalt (1). Polymer additives currently available include natural and synthetic rubber latex (2,3), ground rubber tire (4,5), styrene-butadiene block copolymers (6,7), in situ reacted elastomeric polymers (8,9), and polyethylene (10,11).

Polyethylene has a number of characteristics that distinguish it from the other additives previously mentioned. Being a plastomer rather than an elastomer (rubber or rubberlike polymers), polyethylene remains a semicrystalline solid throughout regular pavement service temperatures and will show greater rigidity and less elasticity (12). Polyethylene is not miscible in asphalt and naturally separates into two phases. The quality of the dispersion is dependent on the process and the stabilizer used to emulsify the polyethylene (13). Finally, the general availability of recycled polyethylene from waste materials is high, providing a further incentive to study its potential as an additive (14).

Polymers alter the bulk viscosity of asphalt, thereby allowing softer asphalt grades to be used without causing adverse performance at higher service temperatures. The lower viscosity of the softer binder is commonly understood to be the key factor that increases the resistance to thermal cracking (15). However, other mechanisms of toughening may also exist. Isolating and exploiting these mechanisms could lead to further improvements in low-temperature performance.

This paper examines the low-temperature toughening mechanisms for different systems of polyethylene dispersed in asphalt. Part of this study included measuring the low-temperature fracture toughness of asphalt binders modified with different quantities of polyethylene. Also examined was the role of the dispersion process and whether the average polyethylene particle size, particle size distribution, or the degree of adhesion to the asphalt matrix significantly affected the resulting fracture toughness. Finally, toughening mechanisms for asphalt at low temperatures based on multiphase toughening of brittle and pseudoductile polymers will be proposed.

BACKGROUND

Toughness and Thermal Stress Cracking

Thermal cracking results from the stresses created by different rates of thermal contraction within an asphalt-aggregate composite. As the temperature decreases, the asphalt will contract at a greater rate and to a greater degree than the base layer. This thermally induced stress condition causes microcracks to form at the pavement surface. As microcracks, they have little or no effect on pavement performance and may eventually "heal" (16). However, with repeated thermal cycles and continual vehicle loading, many of these microcracks grow into macrocracks. Rapid deterioration results when macrocracks grow to span the depth of the asphalt concrete, thereby allowing water to penetrate into the base layer (17). Therefore it is of great benefit to limit the propagation of cracks at the microscopic level, when the damage is still reversible.

Toughening Mechanisms in Multiphase Materials

For the purposes of this study, toughness is defined as a measurement of the energy expended in fracturing a material. The amount of yielding that takes place during the fracture process greatly influences the toughness. Even in the fracture of a brittle material, some localized yielding, permanent (plastic) deformation that occurs when a material is stressed beyond its recoverable (elastic) limit, always occurs. Enhancing this localized yielding by introducing a dispersed secondary phase into the brittle matrix is the basis of many toughened plastics.

The three main toughening mechanisms that occur in multiphase materials are crack pinning, shear yielding, and crazing. All three involve plastic deformation in the microscopic region sur-

rounding the dispersed phase. With crack pinning, this deformation occurs when the moving crack front encounters a row of dispersed particles (18). If these particles have a greater strength than the surrounding matrix, the crack front must travel around the particles to reform as a new fracture surface. Since the plastic flow occurs at the particle-matrix interface, the level of adhesion influences the toughening effect. Interparticle spacing is another important factor, since the formation of each new fracture surface consumes additional energy.

Shear yielding is a constant-volume process and occurs without loss of intermolecular cohesion. Whereas all materials show shear yielding to some degree, its contribution to toughening may be small, as in the case of a brittle, single-phase material. When a dispersed phase has a different elastic modulus from the matrix, it creates conditions of localized stress concentration (19). This in turn increases the potential size and effect of the shear-yielded regions and may lead to significant increases in fracture toughness. In materials where shear yielding is the dominant toughening mechanism, the number of dispersed particles governs the overall toughening effect.

Crazing is a toughening mechanism found in high-molecular-weight materials. With crazing, there is a significant change in volume as small voids, called crazes, form within the matrix. Instead of coalescing and propagating as a macrocrack, an interpenetrating network of fibrils stabilizes the voids. The fibrils form when the matrix material becomes highly oriented. The orientation process consumes energy and also makes the fibrils strong enough to stabilize the craze. As with shear yielding, fibril formation is dependent on localized matrix softening resulting from the inclusion of a finely dispersed second phase. Since overcoming the intermolecular cohesion in the region of crazing consumes a significant amount of the fracture energy, the degree of interfacial adhesion is important (20). It is also important for there to be a quantity of larger particles that serve to limit the crazes from growing into true cracks. Thus the toughening effect due to crazing will be sensitive to both the degree of adhesion between the particles and the matrix and the particle size distribution.

Measurement of Toughness Using Performance-Based Tests

Performance-based tests do not isolate localized toughening mechanisms from changes in bulk properties. Instead, they measure the effect of many microscopic and macroscopic factors acting together. For example, Joseph et al. (21) found thermal shrinkage strains in mixes containing (unstabilized) polyethylene-modified binders that were markedly lower than those made with either the unmodified or ground rubber tire-modified binders. As shown in Figure 1, the temperature versus induced strain curve for the mix containing the polyethylene-modified binder has the smallest slope. In an unstable system, though, coalescence of the polyethylene will occur, and most of the polymer will exist in large (greater than 100- μm) domains. Without an even distribution of finely dispersed particles, the increase in fracture toughness will be limited. Large polyethylene domains, though, may serve to bridge the aggregates and thus reduce thermal shrinkage and failure. In this way unstable polyethylene may be a beneficial additive even though it may not greatly increase the binder toughness.

Binder tests based on failure strain and failure stress were also not intended to measure fracture toughness. Neither the force ductility test nor the direct tension test developed under the Strategic Highway Research Program (SHRP) can distinguish changes in bulk viscosity from effects that occur at the microscopic level. What the newly developed binder tests clearly demonstrate, though, is that the addition of polymer to the binder can significantly alter the thermal cracking properties of an asphalt mix. Using the thermal stress restrained specimen test, Jung and Vinson (17) showed that the low-temperature performance of the mix is influenced by the thermal and mechanical properties of the binder. This test was also used to validate the low-temperature binder specification tests developed by the SHRP program. When performed with polymer-modified binders, these tests confirmed that the addition of polymers often improves the low-temperature performance of a given asphalt grade. Such performance-based testing has led to a general conclusion that "different polymers achieve their effects by significantly different mechanisms" (22). However, performance-based testing alone cannot provide any additional information concerning the actual mechanisms involved.

Where To Look for Evidence of a Toughening Mechanism in Asphalt

When testing an unstable, polyethylene-asphalt system at low temperatures, Jew and Woodhams (23) found that fracture properties were sensitive to polymer concentration. They suggested both shear yielding and crazing as possible explanations for the increase in toughness. Also suggested as having significance were the particle characteristics of the secondary phase. The latter suggestion agrees with observations made in other studies that recognized the importance of achieving fine dispersions in multi-phase polymer-asphalt systems (24,25). However, to date no one has quantitatively analyzed the results of altering the quality of the dispersion to investigate theories of low-temperature toughening.

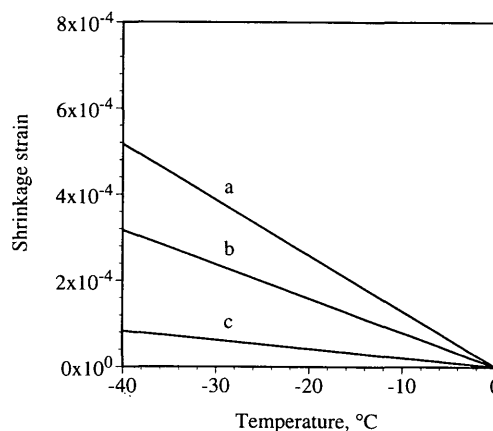


FIGURE 1 Unrestrained shrinkage strain in various asphalt-aggregate mixes (21): a = unmodified binder, b = rubber-modified binder, c = unstabilized polyethylene-modified binder.

EXPERIMENTAL

Materials

The base asphalt used for the polyethylene-modified binders was a Bow River 85–100 penetration grade asphalt (SHRP code AAN). An 85–100 and a 200–300 penetration grade asphalt from Venezuela were used as the base asphalt for the samples modified with chlorinated polyethylene.

Stable polyethylene-modified binders were prepared by previously established procedures (11), only a brief description of which is given here. In addition to the polyethylene, a low-molecular-weight elastomer was added to heated, molten asphalt. Under high shear mixing at 165°C, the additives react in situ to form micron-sized polyethylene particles surrounded by a layer of elastic, asphalt-grafted polymer. As the elastic interface becomes swollen by the asphalt, it acts to sterically stabilize the polyethylene and prevents coalescence from occurring.

The unstable polyethylene-modified binder was also created using the described procedure except that a coupling agent necessary for the polyethylene-elastomer reaction was not added. As a result, there was no compatibility at the interface between the polyethylene and the asphalt matrix. Coalescence occurred as the test samples cooled down, resulting in a much coarser dispersion.

The chlorinated polyethylene additive (Tyrin 2552 from Dow Chemical of Midland, Michigan) contains 25 wt percent chlorine and is partially soluble in most asphalts. The mixture requires no additional additives to remain thermally stable. High shear mixing at 195°C with an 85–100 pen and 200–300 pen Venezuelan asphalt created modified binders with a very fine dispersion of partially dissolved polymer.

Control samples for the polyethylene-modified systems were created by adding only the elastomeric stabilizer to the base asphalt and reacting it under high shear for 2 hr. Unmodified asphalts served as the control for the chlorinated polyethylene-modified samples.

Fracture Mechanics

Preparing samples for fracture testing involved pouring hot liquid asphalt into a notched silicone rubber mold and cooling the filled mold in a freezer set at the testing temperature of -20°C . After 2 hr, the asphalt bars, measuring 25 mm wide by 12.5 mm deep by 175 mm long, were cold enough to be easily removed from the molds. The cast bars were then returned to the freezer to cool before testing. This cooling period ranged from 18 to 36 hr.

Fracture testing was performed using a three point bend configuration based on ASTM E399–90. Tests were performed on a computer-controlled Sintech 2/G testing frame equipped with an environmental chamber. As shown in Figure 2, each sample bar was cast with a 5-mm-deep, 90-degree starter notch and centered on two supports that were set at a 100-mm span. Immediately before each test, the notch was sharpened with a razor blade. The point of loading was opposite the sharpened notch, and the cross-head speed was set at a constant rate of 0.01 mm/sec. Samples were monotonically loaded until failure. Load and deflection data were measured using a 200-lb load cell, digitally recorded, and stored for later analysis.

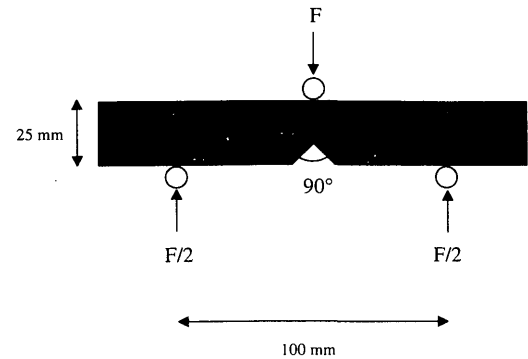


FIGURE 2 Bending beam configuration for low-temperature fracture tests on asphalt binders (test temperature = -20°C and 0.01 mm/sec loading rate).

Brittle fracture studies were completed for 15 samples. Measured data included the failure load and the modulus. Fracture toughness values were calculated according to the following equation (26):

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

where

- K_{Ic} = critical stress intensity factor or fracture toughness ($\text{N m}^{-3/2}$),
- P_f = applied failure load (N),
- S = loading span (m),
- B = specimen depth (m),
- W = specimen width (m), and
- a = crack length (m).

The two main parameters (the fracture toughness, K_{Ic} , and the stiffness modulus, E) were calculated assuming linear elastic conditions. Creating conditions where the equations are based on linear elastic fracture mechanics (LEFM) avoids the difficulties associated with viscoelastic behavior. LEFM equations are valid when the modulus of the matrix and the added polymer phase vary only slightly with time (19). Choosing a low test temperature of -20°C ensured that the asphalt matrix would not exhibit any viscous behavior during testing, even at low strain rates. Selecting a sample thickness that was large compared with the plastic zone size ensured that plane strain conditions existed at the crack tip.

Fracture toughness is a fundamental material parameter that measures the stress intensity at critical conditions. When the stress distribution is uniform throughout the material, the fracture toughness is a measurement of strength. However, with a well-defined stress field, such as in a notched sample under conditions of plane strain, the fracture toughness measures the magnitude and distri-

bution of the stress at the crack tip (27). Unlike tests that measure material strength, strain-to-failure, or ductility, the fracture toughness test is directly related to the energy released during crack propagation. For this reason, the fracture toughness of a binder may be a better parameter for measuring its ability to withstand internal stresses before they build up and lead to catastrophic failure.

Morphology

Scanning electron microscope (SEM) photographs yielded particle size distributions for the four asphalt samples containing 6 wt percent polyethylene. Approximately 0.5 g of asphalt was dissolved in 10 mL of tetrahydrofuran (THF), filtered through a Nuclepore polyester membrane filter (0.2- μm pore size and 25-mm diameter) and rinsed twice with additional 10-mL portions of fresh THF. The residue was then gold coated for SEM examination. Using the SEM, the samples were scanned and photographed at 1000X magnification. Particle diameters were measured manually using enlargements of the SEM photographs.

Each particle size distribution is the result of measuring more than 1,000 particle diameters. Distributions are expressed in cumulative wt percent (equivalent to vol percent) to better illustrate the effect of large particles. Particle volumes were calculated using the measured diameters and assuming a spherical geometry. Volumes for each particle size category were divided by the total volume to produce a distribution that approximated a log-normal distribution (28). The distributions are defined by two parameters: the mean particle size and the standard deviation of the mean.

RESULTS AND DISCUSSION

Low-temperature fracture results, presented in Table 1, clearly distinguish between regular and polymer-modified binders. With this test, nonbrittle fracture may occur with samples that warm up even slightly, and thus maintaining the test specimen to within 1°C of

-20°C was essential to getting consistent test results. Slight inconsistencies in the sharpness of the notch, the geometry of the samples tested, and the point of loading also add to the scatter in results. In the results presented, samples showing bulk yielding, indicated by an inflection point on the stress-strain curve, were discarded.

Test results show different low-temperature modulus values for binders of different penetration grades. For linear-elastic conditions, the slope of the stress-strain curve provides the modulus. It is analogous to the Young's modulus for elastic materials. The base asphalt determines the elastic modulus. At -20°C, the stiffness of the binder is not significantly changed by the addition of up to 8 wt percent polyethylene.

Whereas the addition of polymer did not greatly increase the binder stiffness, it increased the fracture toughness (K_{Ic}) of the material. Compared with their respective control samples, the addition of 6 percent stabilized polyethylene led to a 50 percent increase in the fracture toughness. Adding 5 percent chlorinated polyethylene more than doubled the fracture toughness. Of the three systems studied, the unstabilized polyethylene showed the least increase, approximately 25 percent for 6 percent added polymer.

Shown in Figures 3 and 4 are the fracture toughness values plotted against polymer content for the polyethylene system and the chlorinated polyethylene systems, respectively. In both systems, the relationship was linear with correlation coefficients of 0.94 or greater. Compared with the stable polyethylene system, the slope of the chlorinated polyethylene-modified binder was 30 percent greater.

Figure 5 shows the fracture toughness contribution of 6 percent added polyethylene for the three polymer-modified systems studied. With higher additive contents, the toughening effect becomes markedly different. This observation resembles that of multiphase toughening of a pseudoductile polymer, such as polypropylene, that relieves internal stresses primarily through crack pinning and shear yielding. The number of dispersed particles and the polymer-asphalt interfacial adhesion govern the enhancement of these toughening mechanisms. Since each particle creates a yielded region in the

TABLE 1 Results from Low-Temperature Fracture Testing of Polyethylene-Modified Asphalt Binders

Asphalt Grade (Penetration)	Additive (wt. %)	Modulus (GPa)	Fracture Toughness, K_{Ic} ($\text{kN m}^{-1.5}$)
85-100	0% PE ^a	1.4 \pm 0.1	79 \pm 5
Bow River	3% PE	1.4 \pm 0.1	88 \pm 5
	4% PE	1.4 \pm 0.1	98 \pm 6
	6% PE	1.4 \pm 0.1	117 \pm 9
	7% PE	1.4 \pm 0.1	123 \pm 6
	8% PE	1.6 \pm 0.1	140 \pm 9
85-100	0% CPE ^b	1.4 \pm 0.1	44 \pm 4
Bow River	3% CPE ^c	1.4 \pm 0.1	83 \pm 6
	5% CPE	1.4 \pm 0.1	91 \pm 3
200-300	0% CPE	0.45 \pm 0.03	50 \pm 3
Bow River	3% CPE	0.50 \pm 0.08	100 \pm 20
	5% CPE	0.42 \pm 0.03	155 \pm 8

^a Control sample containing only elastomeric stabilizer.

^b Control sample containing 100% Bow River Asphalt.

^c Chlorinated polyethylene (no stabilizer necessary).

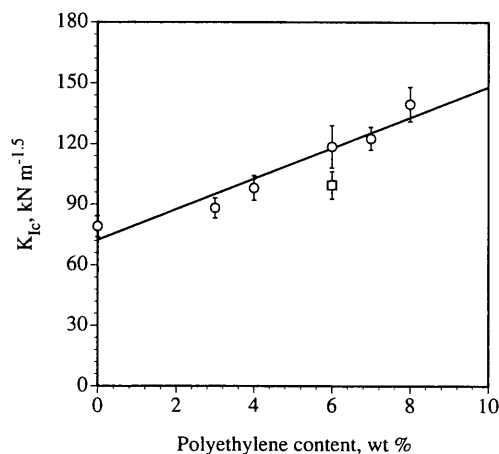


FIGURE 3 Fracture toughness for polyethylene-modified 85-100 Bow River asphalts (\circ = sterically stabilized, \square = unstabilized; error bars give 90 percent confidence limits).

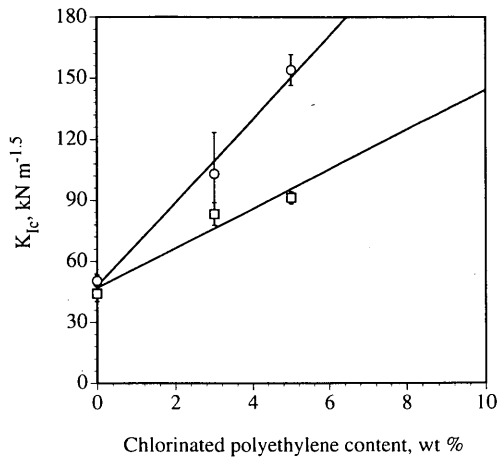


FIGURE 4 Fracture toughness for chlorinated polyethylene-modified 85-100 and 200-300 Bow River asphalts (\circ = 200-300 asphalt, \square = 85-100 asphalt; error bars give 90 percent confidence limits).

surrounding matrix material, a greater number of inclusions will necessarily create a greater toughening effect. With rubber-toughened polypropylene (29), a transition occurs when the rubber content exceeds roughly 12 wt percent. At that concentration, the shear yielding mechanism contributes to a massive increase in toughening. Yield zones overlap and strong particle interactions lead to a much tougher material. Test results in our asphalt systems show no such transition from brittle to ductile behavior. To achieve such a transition, the additive content would most likely have to exceed 10 wt percent, which is neither cost-effective nor feasible with current processing methods.

With the softer-grade Venezuelan asphalt, the toughening effect due to the addition of chlorinated polyethylene was the most pronounced. Without modification, the 200-300 pen binder shows only a marginal increase in fracture toughness over the 85-100 pen asphalt. When this lower viscosity binder was modified with

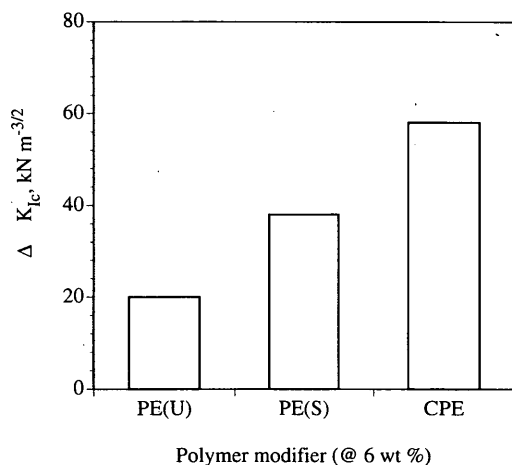


FIGURE 5 Added benefit to low-temperature fracture toughness for different polyethylene modifiers in an 85-100 Bow River binder.

5 percent chlorinated polyethylene, though, the fracture toughness increased by more than 300 percent. SEM photographs of the fracture surface show yielded regions in the softer matrix. Apparently there is a synergistic effect in which the added polymer contributes to larger regions of plastic deformation within the lower-viscosity matrix. A low-viscosity binder modified with enough chlorinated polyethylene may even begin to demonstrate pseudoductile behavior. To study the brittle-to-ductile transition, techniques based on elastic plastic fracture mechanics, such as J-integral tests (18), are necessary.

For the systems studied in this work, the crazing mechanism is not thought to be of as much importance as crack pinning or shear yielding. First, crazing is highly dependent on the molecular weight of the matrix. Only fibrils formed of high-molecular-weight matrix material have the necessary strength to stabilize crazes. In the case of toughened polystyrene (19), significant crazing occurs only when the molecular weight of the matrix exceeds $70\,000\text{ g mole}^{-1}$. As a comparison, an asphalt matrix has a typical number average molecular weight of approximately 1000 g mole^{-1} . Furthermore, usually toughening due to crazing is sensitive to changes in the particle size distribution. As indicated in Table 2 and Figure 6, for the stabilized polyethylene-asphalt systems, fine changes in the particle size distribution did not lead to any difference in the fracture toughness.

TABLE 2 Particle Size Data for 6 percent Polyethylene-Modified Asphalt Binders (Bow River 85/100 Pen)

Sample	Wt. Average Particle Diameter (μm)	Standard Deviation (μm)	Fracture Toughness, K_{Ic} ($\text{kN m}^{-1.5}$)
Control ^a	-	-	79 ± 5
A	2.2	1.12	119 ± 9
B	3.5	1.18	115 ± 7
U ^b	10.8	5.12	99 ± 7

^a Control sample containing only elastomeric stabilizer.

^b Unstabilized polyethylene

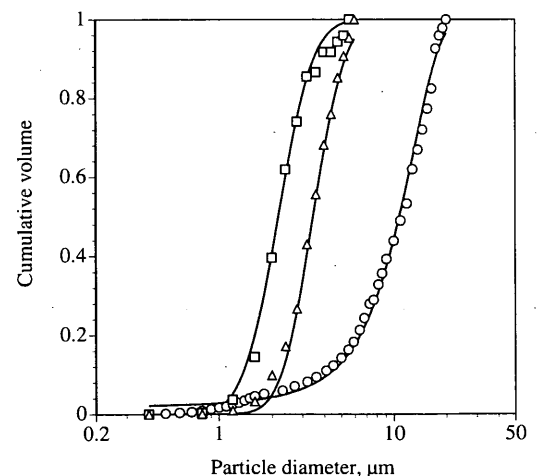


FIGURE 6 Particle size distribution for 6 percent polyethylene-modified samples (\square = Sample A, \triangle = Sample B, and \circ = Sample U).

Results from this single study do not completely discount the possibility of craze formation in all polymer-modified asphalts. In the chlorinated polyethylene system, where the polymer partially dissolves in the asphalt matrix, enough high-molecular-weight material may be present for crazing to occur. Further investigations using techniques such as volumetric strain measurements (19) are necessary before evidence supporting or disproving crazing becomes conclusive.

SUMMARY

1. Low-temperature fracture testing distinguishes between different grades of asphalt binders and between different polyethylene-asphalt binder systems.

2. The addition of polyethylene improves the low-temperature fracture toughness of an asphalt binder without significantly increasing its stiffness at low temperatures.

3. For polyethylene contents below 8 wt percent, the increase in fracture toughness is a linear function of the polymer content. No sudden increase in toughening was seen that would indicate a brittle-to-ductile transition at the chosen test temperature.

4. Finer dispersions of polyethylene contributed to a more pronounced toughening effect.

5. The toughening effect is more pronounced when chlorinated polyethylene was added to a softer matrix.

6. With stabilized and unstabilized polyethylene, the toughening is attributed to a crack pinning mechanism.

7. With the chlorinated polyethylene modifier, there is also evidence of shear yielding at the crack tip.

8. It is unlikely that crazing plays a significant role in any of the systems studied.

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