The impact of Hisingerite on asphalt shear resistance

Greg White*

School of Science and Engineering, University of the Sunshine Coast, Sippy Downs, Queensland, Australia

(Received 18 May 2015; accepted 22 February 2016)

Two Marshall-designed asphalt mixtures were developed with common coarse aggregate, added filler (1% hydrated lime) and bituminous binder (5.8% acid-modified bitumen). The target aggregate grading and overall volumetrics were not significantly different. Different fine aggregate (dust sources) were used in each asphalt mixture. Dust A contained predominantly Nontronite clay minerals while 82% of the Dust B clay minerals were identified as Hisingerite. Hisingerite is a rarely encountered and poorly studied clay mineral. Specialist geotechnical interpretation of its unique properties indicated potentially adverse impact on mastic stability and asphalt shear response. This research assessed the impact of Hisingerite clay on otherwise identical asphalt mastic, as an indicator of the impact of Hisingerite-rich dust sources on asphalt performance. Six M1000 (acid-modified) bitumen samples were each used to manufacture two mastic samples, one containing each fine aggregate. The same hydrated lime was added, and all mastic samples were manufactured with constant proportions of bitumen, added filler and fine aggregate, representative of the asphalt mixture designs. Wheel tracking, resilient modulus, tensile strength and moisture resistance testing did not indicate any detrimental effects from Hisingerite-rich Dust B nor did mastic complex modulus master curves. Multiple stress creep recovery testing found the Dust B mastic to deform less than Dust A mastic under cyclic shear. This was concluded to likely reflect the lower density and higher absorption of the Dust B fine aggregate. Despite the concerning physical properties, it was concluded that the incorporation of Hisingerite-rich fine aggregate presented no risk to asphalt performance. It follows that there is no basis for additional routine testing of potential dust sources to identify Hisingerite content. This research also highlighted the importance of controlling mastic composition during testing, with further testing at a constant ‘effective’ binder volume required. Maintaining the filler dosage constant relative to the mass (or volume) of the dust (or the bitumen) must also be considered.

Keywords: Hisingerite; Smectite; asphalt shear; airport asphalt; asphalt mastic

Introduction

Two Marshall-designed asphalt mixtures were developed as candidates for an airport resurfacing project. Both asphalt mixtures contained the same grade of acid-modified bitumen (known as M1000), the same hydrated lime (filler) and the same coarse aggregate. As detailed below, the two asphalts differed only by the source of the fine aggregate (dust). The two fine aggregate sources (Dust A and Dust B) were both crushed from basalt quarries and produced asphalt mixtures with similar particle size distributions. Both fine aggregate sources

*Email: greg.white@fultonhogan.com.au

© 2016 Informa UK Limited, trading as Taylor & Francis Group
complied with the various specification requirements for Marshall-designed airport asphalt in Australia.

During a petrologic analysis of the materials, Dust B was found to contain a rare clay mineral called Hisingerite. Hisingerite is a rarely encountered and potentially detrimental clay (Shayan, 1984). No assessment of the effects of Hisingerite on the engineering properties of crushed aggregate, concrete or asphalt was available. Specialist geotechnical interpretation of the documented properties of Hisingerite indicated the potential to create ductile or unstable asphalt mastic with the potential to adversely impact asphalt shear resistance. Dust A contained other more commonly encountered Smectite clay minerals, such as Nontronite.

Dust B was logistically and economically preferable to Dust A. However, the risk of Hisingerite-rich Dust B adversely impacting the asphalt shear performance was concerning. At the project level, exclusion of the Dust B asphalt was considered, despite its logistical and economic benefits. The potential addition of routine testing for Hisingerite content of all aggregate sources for future airport asphalt projects was also discussed at the project level.

The aim of this research was to determine the impact of Hisingerite-rich fine aggregate on the shear resistance of a typical airport asphalt mixture. The existing knowledge indicated that for a common coarse aggregate matrix, the mastic properties would determine asphalt mixture shear resistance. Therefore, performance-based comparison of mastics containing Dust A (free of Hisingerite) and Dust B (Hisingerite rich) is presented. Conclusions address the effect of Hisingerite on asphalt mastic shear resistance and the justification for avoiding fine aggregate sources containing Hisingerite for asphalt production.

**Background**

Asphalt is a complex heterogeneous material consisting of aggregate, air voids and bituminous binder (Zelelew & Papagiannakis, 2012). Asphalt is used around the world in many applications, including the surfacing of airport pavements (Elnasri, Airey, & Thom, 2013). The time and temperature dependence of bitumen properties has a significant impact on the response of asphalt to loading (Scarpas, Al-Khoury, van Gurp, & Erkens, 1997). At the extremes, asphalt is purely elastic at low (< 0°C) temperatures and fast loading and purely plastic at high (100°C) temperatures and slow loading (Al-Qadi, Yoo, Elseifi, & Nelson, 2009). During vehicle braking, asphalt is subjected to increased shear stresses, up to 68% of the vertical contact stress (Horak, Maina, & Emery, 2009). This has resulted in horizontal surface shearing, particularly at higher (> 60°C) surface temperatures (White, 2014).

**Asphalt shear resistance**

Permanent vertical asphalt deformation (rutting) was previously considered to be caused by viscous flow of the asphalt binder/mastic (D’Angelo, Kluttz, Dongré, Stephens, & Zanzotto, 2007). Asphalt rutting is correctly defined as the cumulative permanent deformation of the asphalt layer(s) through incremental densification under loading (Sousa, Solaimanian, & Weissman, 1994). True vertical deformation rarely occurs in asphalt surfaces and is often confused with what is actually a shear failure, characterised by heaving or slip-circle-type deformation. True rutting is free of heaving at the extremities. Recent research has shown that most asphalt permanent deformation is the result of shear creep rather than viscous flow (D’Angelo et al., 2007).

In high-shear-stress conditions, asphalt can permanently deform horizontally, without any vertical deformation (White, 2014). Runways experience high horizontal shear forces during aircraft braking (White, 2016). A number of instances of horizontal asphalt surface shearing failures
have occurred at airports (Bognacki, Frisvold, & Bennert, 2007; Monismith, Vallerga, Harvey, Long, & Jew, 2000; Vallerga, Jew, & Nokes, 2000). The temperature-dependent nature of bitumen increases the risk of such failures at elevated pavement temperatures when the asphalt is less creep resistant. It follows that for a typical coarse aggregate skeleton, the bituminous mastic governs the asphalt shear resistance.

The importance of mastic
Mastic is the ‘real’ binder in an asphalt mixture (Delaporte, Di Benedetto, Chaverot, & Gauthier, 2007; Qiu, Tan, Shu, & Zhang, 2013). Tashman, Masad, Little, and Zbib (2005) supported this by stating that the micro-constituents governed behaviour of the overall mixture. Elnasri et al. (2013) agreed, noting that testing mastic provides greater insight into asphalt performance than bitumen testing. Mastic has greater impact on the performance of asphalt mixtures with dense grading and high binder content, than the performance of stone-on-stone mixtures, such as stone mastic and porous asphalt (Muraya, Molenaar, & van de Ven, 2009). Airport asphalts are commonly specified to be dense-graded with high binder content and rely heavily on mastic performance to resist stresses and deformation (Emery, 2005). It follows that airport asphalt mixtures are highly affected by the properties of the mastic.

Despite the recognised importance of mastic for asphalt performance, less is known about mastic properties than those of bitumen (Liao, Airey, & Chen, 2013). Mastics of seemingly similar constituents can behave differently (Faheem & Bahia, 2010). This can only be explained by physio-chemical interaction between the bitumen and mineral elements. Such interactions cannot be assessed by considering the bitumen and mineral components separately.

The ratio of filler to binder in mastic is critical to mastic characterisation. As the filler portion increases, the mastic stiffens (Pérez-Jiménez, Miró Recasens, & Martínez, 2008; Qiu et al., 2013). Liao et al. (2013) found that the filler portion had a greater impact on mastic response than filler type. It follows that accurate replication of the binder:filler:aggregate ratio in the asphalt mixture is crucial to mastic testing.

Clay and Hisingerite
Clay minerals are commonly present in quarried aggregate sources (Little & Epps, 2001). Clay minerals vary widely and their chemical composition and morphology can have significant impact on the engineering performance of fine aggregates (Brigatti, Galan, & Theng, 2013). Although clay minerals also occur in coarse aggregate particles, the comparatively small surface area of coarse aggregate results in most of the clay minerals being trapped inside the aggregate particles. It follows that clay mineral properties are more important for fine aggregate sources, where they are incorporated in the mastic and are more readily exposed to air, moisture and bitumen.

X-Ray diffraction
X-Ray diffraction (XRD) is a semi-quantitative analysis of the chemical composition of powdered (30 μm and below) rock samples. It is routinely used for mineral composition analysis of aggregate sources. Following calibration of equipment and analysis software, XRD is highly accurate and assesses the whole of the powdered sample (Amaral, Cruz Fernandes, & Guerra Rosa, 2006). Other methods (such as X-Ray Fluorescence) only assess the composition of the exposed particle surfaces. Further, XRD allows visually identifiable particles of interest within a crushed aggregate sample to be separated and powdered in order to assess their contribution to
the fine aggregate chemical composition. Separation of the actual fine aggregate particles would be impractical.

**Hisingerite**

Hisingerite is a rarely encountered and poorly studied clay mineral. First described in 1810, Hisingerite has been variously regarded as a non-crystalline silicate, a ferric allophane, a poorly crystallised Nontronite and an iron-rich spherical Halloysite (Brigatti et al., 2013). Hisingerite is characterised by the presence of curved or spherical bodies which are largely in a random and concentric arrangement. These spherical bodies are substantially different from the flat sheets observed in Saponite, Nontronite and other more common clays (Eggleton & Tilley, 1998).

Hisingerite has previously been identified in a basalt quarry within the same region as the basalt quarry from which Dust B was sourced (Shayan, 1984). The previously identified local Hisingerite had a high pH and low cation exchange capacity, indicative of negatively charged particle surfaces. This was confirmed by the absorption of 3.4 g of methylene blue dye by 1 g of Hisingerite in a 4% aqueous suspension. The locally identified Hisingerite was found to be highly hydrous, even more so than many other Hisingerite sources around the world (Shayan, 1984).

No literature could be found detailing the physical effects of Hisingerite on the performance of civil construction materials, such as asphalt, concrete or crushed rock. Specialist geotechnical interpretation of the unique properties of Hisingerite minerals suggested potential interaction with acid-modified bitumen (such as M1000) and lime (used in both mixtures) in the production and performance of asphalt. The highly hydrous nature of Hisingerite could trap excess moisture in the asphalt mastic. These potential interactions may adversely affect mastic stability and result in poor shear resistance of an asphalt surface. This would imply that additional testing of fine aggregate sources for Hisingerite content is warranted during future projects and that known Hisingerite-rich sources of fine aggregate should be avoided.

**Multiple stress creep recovery**

The USA introduced the Performance Grading (PG) system to the specification of bitumens in the 1990s (D’Angelo, 2009). The Multiple Stress Creep Recovery (MSCR) protocol subsequently replaced the parameter known as \(\frac{|G^*|}{\sin(\delta)}\) as the PG high-temperature criterion in 2010. This repeated shear test was developed to be blind to modification and site location and to assess binder response to shear in both the linear and nonlinear stress ranges. MSCR has shown better correlation to full scale and field deformation of asphalt mixtures (FHA, 2011). MSCR represents best practice for performance-based assessment of paving-grade bituminous binders.

MSCR has been demonstrated to be easy to perform in the laboratory using modern Dynamic Shear Rheometer (DSR) equipment (D’Angelo, 2009) and takes only around 15 minutes to complete (DuBois, Mehta, & Nolan, 2014). Six parameters are calculated from the MSCR protocol (FHA, 2011). Of these, the cumulative unrecovered strain over ten high (3.2 kPa)-stress-level cycles is the primary PG criterion. This is termed the creep compliance or Jnr(3.2). Other parameters are the Average Recovery (AR) at 0.1 and 3.2 kPa stress levels. The stress sensitivity (%Jnr) is determined from the difference between the Jnr(0.1) and Jnr(3.2) and indicates the bitumen sensitivity to increased shear stress magnitude.

It is normal to artificially age samples using the Rolling Thin Film Oven (RTFO) prior to the MSCR testing. The RTFO conditioning is intended to simulate bitumen ageing during asphalt production (Wu, Lepercq, & Airey, 2007). Most researchers have performed MSCR after RTFO conditioning (Clopotel & Bahia, 2012; D’Angelo & Dongré, 2009; Domingos & Faxina, 2015; Motamed & Bahia, 2011; Riaz et al., 2013).
Research methods

Materials

Two asphalt mixtures were designed, one containing Dust A and the other containing Dust B. The two mixtures were similar and are compared against the key specification targets and limits in Table 1, while the overall aggregate grading of the two mixtures is given in Table 2.

Both dusts were sourced from basalt quarries and contained non-plastic fines as required by the specification. A petrographic analysis of the two dusts found both to be olivine basalt of hard, grey, robust particles of slight to moderate weathering. Dust B was slightly more weathered than Dust A as indicated by the higher Smectite (clay) and accessory mineral contents. Both dusts were found to contain secondary minerals in and around cracks in the olivine structure. Secondary minerals in both dusts were initially identified as Nontronite (a common clay within the Smectite group). A summary of the petrographic report findings is given in Table 3.

Non-routine XRD assessment initially agreed with the petrography, indicating the two dust sources contained comparable amounts of clay minerals within an otherwise typical olivine basalt. Further XRD analysis of observed brown chips within Dust B determined that what the initial petrographic assessment reported as Nontronite clay was in fact the rare clay mineral Hisingerite. Negligible Hisingerite existed in Dust A (Table 4). The higher percentage of clay minerals in Dust B explained the higher absorption of methylene blue solution.

Both asphalt mixtures contained the same grade of acid-modified bitumen (M1000). Each of six M1000 bitumen samples was used to produce two mastic samples, one with each of the two dust sources. The retained bitumen samples each represented a different production batch of M1000 binder and key specification properties are given in Table 5. The M1000 samples

| Parameter               | Asphalt mix | Specific target/limit |
|-------------------------|-------------|-----------------------|
| Fine aggregate          |             |                       |
| M1000 binder content (%)| 5.8         | 5.8                   | > 5.6 |
| Hydrated lime content (%)| 1.0         | 1.0                   | 0.5–1.5 |
| Marshall stability (kN)  | 15.3        | 17.5                  | > 12.0 |
| Marshall flow (mm)       | 3.3         | 3.1                   | < 3.5 |
| Air voids (%)            | 4.4         | 4.2                   | 3–5   |

| Australian standard sieve (mm) | Percentage passing by mass (%) | Dust A asphalt | Dust B asphalt | Specification target |
|--------------------------------|--------------------------------|----------------|----------------|----------------------|
| 19.0                           | 100                            | 100            | 100            |
| 13.2                           | 99                             | 98             | 100            |
| 9.5                            | 84                             | 83             | 82             |
| 6.7                            | 70                             | 71             | 70             |
| 4.75                           | 60                             | 62             | 60             |
| 2.36                           | 63                             | 47             | 44             |
| 1.18                           | 29                             | 31             | 33             |
| 0.600                          | 20                             | 22             | 25             |
| 0.300                          | 13                             | 15             | 16             |
| 0.150                          | 8.8                            | 9.8            | 10             |
| 0.075                          | 6.1                            | 6.5            | 5–7            |
Table 3. Summary of fine aggregate petrographic report.

| Item/mineral content                  | Dust A                | Dust B                |
|--------------------------------------|-----------------------|-----------------------|
| Rock type                            | Olivine basalt        | Olivine basalt        |
| Apparent density (t/m³)              | 2.89                  | 2.79                  |
| Absorptivity (%)                     | 2.0%                  | 2.5%                  |
| Methyl blue value for fine aggregate (%) | 4                     | 8                     |
| Plagioclase                          | 71%                   | 59%                   |
| Magnetite                            | 12%                   | 4%                    |
| Olivine                              | 4%                    | 5%                    |
| Augite                               | 4%                    | 13%                   |
| Smectite group                       | 8%                    | 13%                   |
| Glass and accessory minerals         | 1%                    | 6%                    |

Table 4. Fine aggregate Hisingerite content.

| Dust source                            | Dust A | Dust B |
|----------------------------------------|--------|--------|
| Percentage of clay minerals in dust    | 8%     | 13%    |
| Percentage of clay that was Hisingerite| <1%    | 82%    |
| Percentage of Hisingerite in dust     | Negligible | 10.7% |

Table 5. M1000 point of release compliance testing results.

| Bitumen sample | Vis. post-RTFO | Vis. pre-RTFO | Pen. post-RTFO | Pen. pre-RTFO | Vis. 135°C |
|----------------|----------------|---------------|----------------|---------------|------------|
| 1              | 4740           | 1085          | 31             | 46            | 1.138      |
| 2              | 5860           | 1057          | 32             | 48            | 1.106      |
| 3              | 4077           | 987           | 31             | 45            | 1.036      |
| 4              | 6221           | 1260          | 35             | 48            | 1.199      |
| 5              | 6274           | 1229          | 36             | 47            | 1.170      |
| 6              | 6388           | 1249          | 38             | 46            | 1.211      |

Specification limit | 4000–6500 | Report only | > 26 | Report only | < 1.500 |

were reliably PG 64 E or PG 70 V when tested against AASHTO M332-14 (Appendix 1). At 76°C stress sensitivity often exceeded the 75% limit for a PG to be assigned (White, 2015). The significant differences in M1000 properties for samples 1, 2 and 3, compared to samples 4, 5 and 6, reflected a known change in crude oil source (White, 2015). Despite the change in crude oil source and the significant differences in some properties, all M1000 samples were compliant with the Australian paving-grade bitumen specification (AS 2008-13).

As detailed below, all mastic samples included the hydrated lime used in the design of the two asphalt mixtures. The lime used was typical of hydrated lime in Australia and contained acceptable levels of trace chemicals, including aluminium, boron, iron, fluoride and strontium.

Mastic sample preparation

Mastic samples were manufactured in the laboratory from retained bitumen and representative dust and hydrated lime samples. First, the hydrated lime (referred to as ‘filler’) was mixed into heated bitumen. Separately the dust samples were sieved to remove all particles exceeding 75 μm. The required mass of sieved dust (referred to as ‘aggregate’) was then incorporated into the bitumen-filler mixture by further mixing. Mastic samples were manufactured to 6:1:7
(binder:filler:aggregate) mass ratio. This ratio was selected to replicate the average mastic composition within the two asphalt mixture designs, as detailed in Tables 1 and 2.

Mastic samples are referred to by a two-character alpha-numeric code. The first indicates the bitumen sample and the second indicates the dust source. For example, mastic sample 4B was manufactured from retained bitumen sample 4 (Table 5) and Dust B (Table 1).

Test methods
Initially, mastic samples 1A and 1B were subjected to temperature/frequency sweeps in the DSR before and after RTFO conditioning. Master curves for mastic complex modulus were generated. To demonstrate the impact of filler addition on mastic stiffness, the equivalent master curve was also generated for neat M1000 bitumen (sample 1).

Subsequently, all twelve mastic samples were subjected to the MSCR protocol after RTFO conditioning. The MSCR testing was performed at 64°C, 70°C and 76°C to allow the impact of temperature to be assessed. The same MSCR evaluation was also performed on the six neat M1000 bitumen samples, at the same three temperatures, after RTFO conditioning.

Further, as part of the mix design process, a number of additional tests results were available for the two asphalt mixtures. The additional testing included:

- **Resilient modulus.** Hundred millimetre samples were tested by the indirect tension method in accordance with AS 2891.13.1. A test temperature of 25°C was adopted as is common practice in Australia for modulus testing of airport asphalt.
- **Indirect tensile strength.** Samples were prepared in the gyratory compactor to a target density representing 8% air voids. Samples were tested for indirect diametrical tensile strength at 25°C according to Austroads AG:PT/T231 without moisture conditioning.
- **Tensile strength ratio.** Indirect tensile strength was tested after moisture conditioning (known as the Standard Lottman Test) as detailed in AG:PT/T232. The average difference between conditioned and unconditioned tensile strength was calculated as an indicator of asphalt moisture damage resistance.
- **Wheel tracking.** Laboratory prepared samples were traversed by the wheel tracker, as detailed in Austroads AG:PT/T220. The Australian test is performed at a load of 700 N over 10,000 cycles. Samples were conditioned to 60°C, as detailed in Austroads AG:PT/T231.

Results and analysis

**Results**
The MSCR results for RTFO conditioned bitumen are contained in Appendix 1. The corresponding results for the RTFO conditioned mastic MSCR are in Appendix 2. Results from supplementary mix design testing are contained in Table 6 and the binder and mastic master curves for complex modulus from DSR sweeps are in Figure 1.

**Asphalt mixture design and testing**
The two asphalt mixtures were similar. Both used the same course aggregate source, the same binder and the same hydrated lime added filler. Both targeted the same volumetric composition. It follows that the two mixtures had similar Marshall properties measured during mixture design (Table 2). The Dust B design returned 14% higher Marshall stability and a 6% lower Marshall flow. The difference in air voids after Marshall hammer compaction was negligible.
Table 6. Additional asphalt mix design results.

| Parameter                        | Dust A | Dust B |
|----------------------------------|--------|--------|
| Resilient modulus (MPa)          | 3550   | 2790   |
| Indirect diametrical tensile strength (kN) | 903    | 960    |
| Tensile strength ratio (%)       | 99     | 98     |
| Wheel tracking (mm)              | 3.7    | 3.4    |

The additional mix design properties were also comparable for the asphalt mixtures containing the different fine aggregates (Table 6). The Dust B asphalt had a 21% lower resilient modulus but a 6% higher tensile strength. Dust B asphalt also showed 8% lower rut depth after wheel tracking and an almost identical tensile strength ratio to the Dust A asphalt.

Overall the mix design Marshall properties, volumetrics and the additional mix design testing did not indicate any adverse impact of Hisingerite-rich fine aggregate on the Dust B asphalt mixture.

**Binder and mastic complex modulus**

The master curves for bitumen and mastic complex modulus (Figure 1) demonstrated the significant stiffening resulting from the addition of fine aggregate and active filler to bitumen. The addition of fine aggregate lifted the master curve over its full length. The gradient of the master curve did not change significantly. The significant impact of conditioning the mastic by RTFO can also be seen. However, the RTFO conditioning affected the mastic complex modulus more at lower (\(< 10^{-2}\) Hz) reduced frequency. At higher (> 1 Hz) reduced frequency the pre- and post-RTFO complex moduli converged. This indicated that ageing during asphalt production
has greater impact on mastic complex modulus in high-temperature/low-speed traffic conditions, which are represented by lower reduced frequency.

Interestingly, the RTFO conditioning affected mastic 1A more than it did 1B. The master curve for pre-RTFO mastic 1A was slightly below the 1B curve, however, after the RTFO conditioning the mastic 1A master curve sat slightly above the 1B curve. This difference in the impact of RTFO could not be explained. The master curves generated by temperature/frequency sweeps of a single bitumen sample and mastics manufactured from both fine aggregates did not indicate any detrimental mastic performance associated with Hisingerite-rich Dust B.

**Binder and mastic MSCR**

The shear strains measured during the MSCR testing of neat binder and each mastic sample are illustrated in Figure 2(a) for 70°C test temperature. Figure 2(b) shows the same data with the neat binder samples removed and the axis scales modified to focus on the 3.2 kPa stress-level cycles for the mastic samples only. Trends were consistent at 64°C and 76°C, with the magnitude of the strains increasing with higher temperature.

The mastic accumulated strain was consistently a full order of magnitude lower than for the bitumen samples (Figure 2(a)). The significant impact of stress level was also evident.

The impact of MSCR stress level on bitumen and mastic samples is given in Table 7, where the maximum and minimum cumulative unrecovered strains are summarised after the 0.1 and 3.2 kPa MSCR stress cycles. Ratios between mastic and binder, as well as between stress levels, are also shown. The stress sensitivity of mastic samples was less than for bitumen samples, as indicated by the lower values of Ratio (3.2/0.1) for mastic and the higher values of Ratio (B/M) at 3.2 kPa stress level.

The consistence of mastic and bitumen response to shear stress across the range of test temperatures is illustrated in Figure 3. All binder and mastic samples showed similar increases in Jnr(3.2) with increasing temperature. The Jnr(3.2) difference between samples increased at higher test temperature. Mastic Jnr(3.2) values were two orders of magnitude smaller than typical bitumen Jnr(3.2) values. At 70°C and 76°C the order of Jnr(3.2) for mastic samples reflected the order of the binder sample Jnr(3.2). However, at 64°C, some mastic sample Jnr(3.2) values were ordinally different from the bitumen samples. This is illustrated by the crossing of lines between 64°C and 70°C for some mastic samples. This results from the small strains experienced by mastic at lower temperatures in comparison to the accuracy of the measuring equipment. Expected errors in measurement became significant when the total strains were relatively small.

The effect of the two different dust sources on each of the bitumen samples is shown in Figure 2(b). The Dust A samples consistently deformed more under cyclic shear stress than Dust B samples did. This was also reflected in Jnr(3.2). Summary statistics for binder and mastic Jnr(3.2) values are presented in Table 8. P-values for t-tests of paired (Dust A and Dust B) measured Jnr(3.2) are also given.

Although marginal at 64°C, at both 70°C and 76°C the Dust A mastic samples had significantly higher Jnr(3.2). This indicated that Dust B mastic, and therefore asphalt containing Dust B fine aggregate, would deform less under shear stress. The results implied that the Hisingerite-rich fine aggregate was advantageous to asphalt shear resistance. It is more likely, in fact, that the lower mastic deformation in Dust B samples reflected the lower apparent density and higher absorption of Dust B (Table 3).

The lower apparent density required a larger volume of Dust B in order to maintain the 6:1:7 mass ratio. Although the binder:filler:aggregate ratio was the same for all mastic samples by mass, when expressed by volume, the Dust B mastic samples had a lower portion of bitumen than for Dust A mastic. The higher volume of aggregate in Dust B mastic samples stiffened the
Figure 2. (a) Binder and mastic MSCR strains at 70°C during 0.1 and 3.2 kPa shear stress cycles and (b) Mastic MSCR strains at 70°C during 3.2 kPa shear stress cycles.
Table 7. Cumulative unrecovered strain during the MSCR protocol.

| Stress level | Mastic samples | Bitumen samples | Ratio (B/M) |
|--------------|----------------|-----------------|-------------|
| 0.1 kPa      | 1.14–2.99      | 16.5–49.3       | 15–17       |
| 3.2 kPa      | 44.5–107.8     | 836–2340        | 19–22       |
| Ratio (3.2/0.1) | 36–39    | 47–51           | –           |

Notes: Ratio (3.2/0.1) is the ratio between the cumulative strain after the ten 3.2 kPa stress cycles and the cumulative strain after the 0.1 kPa stress cycles. Ratio (B/M) is the ratio of the cumulative strain for binder samples divided by the equivalent cumulative strain for the mastic samples.

Figure 3. Binder and mastic MSCR Jnr(3.2) at all test temperatures.

Table 8. Mastic Jnr(3.2) summary statistics at all test temperatures.

| Statistics | 64°C | 70°C | 76°C |
|------------|------|------|------|
|            | Binder | Dust A | Dust B | Binder | Dust A | Dust B | Binder | Dust A | Dust B |
| Average    | 0.195 | 0.014 | 0.012 | 0.504 | 0.027 | 0.021 | 1.247 | 0.055 | 0.039 |
| Standard deviation | 0.057 | 0.004 | 0.003 | 0.147 | 0.005 | 0.004 | 0.341 | 0.009 | 0.008 |
| CV         | 29%   | 25%   | 22%   | 29%   | 19%   | 21%   | 27%   | 17%   | 22%   |
| P-value (paired t-test) | –   | 0.10 | –   | –   | 0.01 | –   | –   | < 0.01 |

mastic to a greater extent than Dust A did. The higher absorption of Dust B exacerbated this by reducing the ‘effective’ binder available. Reduced effective bitumen content is known to increase mastic resistance to shear deformation.

Rather than conclude that Dust B (and the associated Hisingerite clay) was beneficial to asphalt mastic shear resistance, it is more appropriate to conclude that Hisingerite-rich Dust B did not
adversely affect the mastic or asphalt response to shear stress at any test temperature. To verify this conclusion, mastic samples must be prepared at the same effective bitumen volume, taking account of any difference in apparent density of the fine aggregate particles as well as their potential to absorb bitumen. Whether the active filler (hydrated lime) dosage is maintained by volume (or mass) of dust (or of bitumen) must also be considered.

Conclusions
Hisingerite is a rarely encountered clay mineral with morphology and physio-chemical properties that appear potentially detrimental to asphalt and other civil engineering materials. Despite these concerning properties, it was concluded that the incorporation of fine aggregate, with predominantly Hisingerite clay minerals, presented no risk to high-temperature shear stress resistance of a typical airport-quality asphalt mixture. It follows that there is no justification for routine testing of fine aggregate sources in order to identify the presence of Hisingerite. Similarly, even high portions of Hisingerite in the crushed fine aggregate should not be grounds for exclusion of quarries as fine aggregate supplies for asphalt production.

The importance of mastic composition to mastic test results is reiterated. The binder:filler:aggregate mass ratio was held constant for all mastic samples. However, slight differences in fine aggregate apparent density and absorption significantly impacted the ‘effective’ bitumen volume in the mastic samples. This likely affected the MSCR results for mastic samples. Further investigation and testing at a constant ‘effective’ binder volume is required. Maintaining the filler dosage constant relative to the volume (or mass) of the dust (or the bitumen) must also be considered.

Acknowledgements
The bitumen and mastic testing reported in this paper was performed and managed by John Lysenko, Khoa Vo, Glynn Holleran and Irina Holleran of Fulton Hogan’s Sydney and Auckland binder laboratories. Chemical composition testing of dust samples was provided by Emeritus Professor of Geology, Philippa Black of Auckland University.

Disclosure statement
No potential conflict of interest was reported by the author.

References
Al-Qadi, I. L., Yoo, P. J., Elseifi, M. A., & Nelson, S. (2009). Creep behaviour of hot-mix asphalt due to heavy vehicular tire loading. Journal of Engineering Mechanics, 135(11), 1265–1273.
Amaral, P. M., Cruz Fernandes, J., & Guerra Rosa, L. (2006). A comparison between x-ray diffraction and petrography techniques used to determine the mineralogical composition of granite and comparable hard rocks. Materials Science Forum, 514–516, 1628–1632.
Bognacki, C. J., Frisvold, A., & Bennert, T. (2007, April 16–18). Investigation into asphalt pavement slip-page failures on runway 04R-22L Newark international airport. Proceedings 2007 FAA worldwide airport technology transfer conference, Atlantic City, USA, Federal Aviation Administration.
Brigatti, M. F., Galan, E., & Theng, B. K. G. (2013). Structural mineralogy of clay minerals. In F. Bergaya & G. Lagaly (Eds.), Developments in clay science (Vol. 5, pp. 21–81). Amsterdam: Elsevier.
Clopotel, C. S., & Bahia, H. U. (2012). Importance of elastic recovery in the DSR for binders and mastics. Engineering Journal, 16(4), 99–106.
D’Angelo, J. A. (2009). Current status of superpave binder specification. Roads Materials and Pavement Design, 10(1), 13–24.
D’Angelo, J., & Dongré, R. (2009). Practical use of multiple stress creep and recovery test. Transport Research Record: Journal of the Transportation Research Board, 2126, 73–82.
D’Angelo, J., Kluttz, R., Dongré, R., Stephens, K., & Zanzotto, L. (2007, March 11–14). Revision of the superpave high temperature binder specification: The multiple stress creep recovery test. In Proceedings of the asphalt pavement technology (Vol. 76, pp. 123–162). San Antonio, TX: Association of Asphalt Paving Technologists.

Delaporte, B., Di Benedetto, H., Chaverot, P., & Gauthier, G. (2007, June 20–22). Filler and binder influence on the linear viscoelastic behavior of mastics. In Proceedings advanced characterisation of pavement and soil engineering materials (pp. 3–13). Athens: Taylor and Francis.

Domíngos, M. D. I., & Faxina, A. L. (2015). Rheological behaviour of bitumens modified with PE and PPA at different MSCR creep-recovery times. International Journal of Pavement Engineering, 16(9), 771–783.

DuBois, E., Mehta, Y., & Nolan, A. (2014). Correlation between MSCR results and polymer modification of binder. Construction and Building Materials, 65, 184–190.

Eggleton, R. A., & Tilley, D. B. (1998). Hisingerite: A ferric Kaolin mineral with curved morphology. Clays and Clay Minerals, 46(4), 400–413.

Elnasri, M., Airey, G., & Thom, N. (2013, June 9–12). Experimental investigation of bitumen mastics under shear creep and creep-recovery testing. In Proceedings T&Di airfield and highway pavement speciality conference (pp. 921–932). Los Angeles, CA: American Society of Civil Engineers.

Emery, S. (2005, September 18–21). Asphalt on Australian airports. In Proceedings AAPA pavements industry conference, Australian Asphalt Pavements Association, Surfers Paradise, Australia.

Faheem, A. F., & Bahia, H. U. (2010). Modelling of asphalt mastic in terms of filler-bitumen interaction. Road Materials and Pavement Design, 11, 281–303.

FHA. (2011, April). The multiple stress creep recovery (MSCR) procedure, Technical Brief FHWA-HIF-11-038, Federal Highways Administration, USA. Retrieved February 8, 2015, from http://www.fhwa.dot.gov/pavement/materials/pubs/hif11038/hif11038.pdf

Horak, E., Maina, J., & Emery, S. (2009, June 29–July 2). A case study: Quantification and modeling of asphalt overlay delamination on an airport pavement. Proceedings eight international conference on the bearing capacity of roads, railways and airfields, Urbana-Champaign, IL, USA, pp. 1475–1483.

Liao, M.-C., Airey, G., & Chen, J.-S. (2013). Mechanical properties of filler-asphalt mastics. International Journal of Pavement Research and Technology, 6(5), 576–581.

Little, D. N., & Epps, J. A. (2001). The benefits of hydrated lime in hot mix asphalt. Arlington, VA: National Lime Association.

Monismith, C. L., Vallerga, B. A., Harvey, J. T., Long, F., & Jew, A. (2000, June 19–21). Asphalt mix studies – San Francisco International Airport. In Proceedings of the 26th air transportation conference (pp. 113–124). San Francisco, CA: American Society of Civil Engineers.

Motamed, A., & Bahia, H. U. (2011). Influence of test geometry, temperature, stress level and loading duration on binder properties measured using DSR. Journal of Materials in Civil Engineering, 23, 1422–1432.

Muraya, P. M., Molenaar, A. A. A., & van de Ven, M. F. C. (2009, June 29–July 2). Contribution of asphalt mix components to permanent deformation resistance. Proceedings Eight International Conference on the Bearing Capacity of Roads, Railways and Airfields, Urbana-Champaign, IL, USA, pp. 259–268.

Pérez-Jiménez, F. P., Miró Recasens, R., & Martinez, A. (2008). Effect of the nature and filler content on the behavior of the bituminous mastics. Road Materials and Pavement Design, 9, 417–431.

Qiu, H., Tan, X., Shu, S., & Zhang, H. (2013). Influence of filler-bitumen ratio on performance of modified asphalt mortar by additive. Journal of Modern Transportation, 21(1), 40–46.

Riaz, K., Hafeez, I., Khitab, A., Hussain, M., Ali, F., Ashiq, S. Z., … Ahmed, I. (2013). Comparison of neat and modified asphalt binders using rheological parameters under virgin, RTFO and PAV aged conditions. Life Science Journal, 10, 2041–2047.

Scarpas, A., Al-Khoury, R., van Gurp, C. A. P. M., & Erkens, S. M. J. G. (1997, August 10–14). Finite element simulation of damage development in asphalt concrete pavements. In Proceedings 8th international conference on asphalt pavements (pp. 673–692). Seattle, WA: International Society of Asphalt Pavements.

Shayan, A. (1984). Hisingerite material from a Basalt quarry near Geelong, Victoria, Australia. Clays and Clay Minerals, 32(4), 272–278.

Sousa, J. B., Solaimanian, M., & Weissman, S. L. (1994, August). Development and use of the repeated shear test (constant height): An optional superpave mix design tool. Report SHRP-A-698, Strategic Highway Research Program, National Research Council.

Tashman, L., Masad, E., Little, D., & Zbib, H. (2005). A microstructure-based viscoplastic model for asphalt concrete. International Journal of Plasticity, 21, 1659–1685.
Appendix 1. Binder MSCR test results

Table A1. Binder MSCR testing results at 64°C.

| Sample | AR(0.1) | AR(3.2) | %AR | Jnr(0.1) | Jnr(3.2) | %Jnr | PG rating |
|--------|---------|---------|-----|----------|----------|------|-----------|
| 1      | 45.8    | 28.0    | 39  | 0.17     | 0.22     | 30   | PG 64 E   |
| 2      | 46.5    | 29.0    | 38  | 0.16     | 0.21     | 30   | PG 64 E   |
| 3      | 42.2    | 24.2    | 43  | 0.21     | 0.28     | 31   | PG 64 E   |
| 4      | 52.3    | 33.3    | 36  | 0.14     | 0.20     | 37   | PG 64 E   |
| 5      | 60.9    | 46.8    | 23  | 0.08     | 0.10     | 30   | PG 64 E   |
| 6      | 49.0    | 33.4    | 32  | 0.14     | 0.18     | 28   | PG 64 E   |

Table A2. Binder MSCR testing results at 70°C.

| Sample | AR(0.1) | AR(3.2) | %AR | Jnr(0.1) | Jnr(3.2) | %Jnr | PG rating |
|--------|---------|---------|-----|----------|----------|------|-----------|
| 1      | 39.6    | 14.4    | 64  | 0.38     | 0.56     | 47   | PG 70 V   |
| 2      | 40.4    | 15.3    | 62  | 0.36     | 0.53     | 47   | PG 70 V   |
| 3      | 35.3    | 11.4    | 68  | 0.49     | 0.72     | 45   | PG 70 V   |
| 4      | 46.6    | 18.6    | 60  | 0.32     | 0.50     | 58   | PG 70 V   |
| 5      | 56.1    | 31.5    | 44  | 0.17     | 0.26     | 55   | PG 70 E   |
| 6      | 42.7    | 18.9    | 56  | 0.31     | 0.45     | 46   | PG 70 E   |

Table A3. Binder MSCR testing results at 76°C.

| Sample | AR(0.1) | AR(3.2) | %AR | Jnr(0.1) | Jnr(3.2) | %Jnr | PG rating |
|--------|---------|---------|-----|----------|----------|------|-----------|
| 1      | 33.0    | 5.6     | 83  | 0.86     | 1.38     | 61   | PG 76 H   |
| 2      | 33.8    | 6.1     | 82  | 0.81     | 1.30     | 61   | PG 76 H   |
| 3      | 27.9    | 4.0     | 86  | 1.13     | 1.74     | 54   | PG 76 H   |
| 4      | 39.5    | 7.6     | 81  | 0.71     | 1.24     | 75   | PG 76 H   |
| 5      | 49.5    | 15.5    | 69  | 0.37     | 0.68     | 82   | PG 76 V*  |
| 6      | 35.3    | 7.7     | 78  | 0.71     | 1.14     | 60   | PG 76 H   |

*Denotes samples that would not receive a PG grading at this temperature due to high-stress sensitivity.
### Appendix 2. Mastic MSCR test results

#### Table A4. Mastic MSCR testing results at 64°C.

| Mastic sample | Bitumen sample | Dust source | AR(0.1) | AR(3.2) | %AR | Jnr(0.1) | Jnr(3.2) | %Jnr |
|---------------|----------------|-------------|---------|---------|-----|---------|---------|-----|
| 1A            | 1              | A           | 69      | 34      | 44  | 0.012   | 0.015   | 28   |
| 1B            | 1              | B           | 65      | 35      | 46  | 0.012   | 0.018   | 45   |
| 2A            | 2              | A           | 66      | 37      | 44  | 0.012   | 0.017   | 44   |
| 2B            | 2              | B           | 65      | 36      | 44  | 0.009   | 0.013   | 49   |
| 3A            | 3              | A           | 61      | 49      | 30  | 0.006   | 0.007   | 9    |
| 3B            | 3              | B           | 65      | 49      | 31  | 0.009   | 0.012   | 29   |
| 4A            | 4              | A           | 71      | 40      | 41  | 0.007   | 0.011   | 67   |
| 4B            | 4              | B           | 70      | 41      | 37  | 0.009   | 0.013   | 49   |
| 5A            | 5              | A           | 70      | 34      | 44  | 0.012   | 0.015   | 28   |
| 5B            | 5              | B           | 72      | 35      | 46  | 0.012   | 0.018   | 45   |
| 6A            | 6              | A           | 68      | 41      | 42  | 0.007   | 0.011   | 74   |
| 6B            | 6              | B           | 66      | 47      | 33  | 0.007   | 0.008   | 14   |

#### Table A5. Mastic MSCR testing results at 70°C.

| Mastic sample | Bitumen sample | Dust source | AR(0.1) | AR(3.2) | %AR | Jnr(0.1) | Jnr(3.2) | %Jnr |
|---------------|----------------|-------------|---------|---------|-----|---------|---------|-----|
| 1A            | 1              | A           | 63      | 29      | 54  | 0.019   | 0.022   | 19   |
| 1B            | 1              | B           | 61      | 27      | 55  | 0.027   | 0.031   | 18   |
| 2A            | 2              | A           | 60      | 29      | 53  | 0.020   | 0.022   | 12   |
| 2B            | 2              | B           | 60      | 27      | 54  | 0.027   | 0.031   | 16   |
| 3A            | 3              | A           | 56      | 26      | 54  | 0.026   | 0.027   | 4    |
| 3B            | 3              | B           | 57      | 27      | 52  | 0.030   | 0.033   | 10   |
| 4A            | 4              | A           | 58      | 32      | 44  | 0.020   | 0.019   | 2    |
| 4B            | 4              | B           | 63      | 36      | 44  | 0.018   | 0.019   | 11   |
| 5A            | 5              | A           | 68      | 38      | 44  | 0.011   | 0.014   | 19   |
| 5B            | 5              | B           | 66      | 35      | 46  | 0.020   | 0.026   | 28   |
| 6A            | 6              | A           | 59      | 31      | 49  | 0.017   | 0.019   | 15   |
| 6B            | 6              | B           | 60      | 32      | 46  | 0.020   | 0.023   | 19   |

#### Table A6. Mastic MSCR testing results at 76°C.

| Mastic Sample | Bitumen Sample | Dust Source | AR(0.1) | AR(3.2) | %AR | Jnr(0.1) | Jnr(3.2) | %Jnr |
|---------------|----------------|-------------|---------|---------|-----|---------|---------|-----|
| 1A            | 1              | A           | 58      | 21      | 63  | 0.035   | 0.041   | 17   |
| 1B            | 1              | B           | 57      | 19      | 66  | 0.047   | 0.062   | 32   |
| 2A            | 2              | A           | 59      | 21      | 64  | 0.030   | 0.039   | 29   |
| 2B            | 2              | B           | 56      | 19      | 66  | 0.048   | 0.063   | 31   |
| 3A            | 3              | A           | 51      | 19      | 63  | 0.047   | 0.052   | 10   |
| 3B            | 3              | B           | 53      | 19      | 64  | 0.050   | 0.067   | 32   |
| 4A            | 4              | A           | 55      | 23      | 58  | 0.032   | 0.037   | 13   |
| 4B            | 4              | B           | 59      | 25      | 58  | 0.033   | 0.043   | 29   |
| 5A            | 5              | A           | 62      | 28      | 55  | 0.022   | 0.026   | 18   |
| 5B            | 5              | B           | 61      | 27      | 56  | 0.039   | 0.050   | 28   |
| 6A            | 6              | A           | 54      | 22      | 59  | 0.031   | 0.036   | 17   |
| 6B            | 6              | B           | 56      | 23      | 58  | 0.037   | 0.047   | 26   |