MICROSTRUCTURAL PAVEMENT MATERIAL CHARACTERIZATION: SOME EXAMPLES

M. B. MGANGIRA

CSIR, Built Environment, P.O. Box 395, Pretoria, 0001

ABSTRACT

The utilisation of advanced measurement techniques should assist in the characterization of pavement materials at a micro-scale. The motivating factor for such an approach is that material properties at the micro-level have a critical role in the way materials respond to loading at the macro-level. The objective of the paper is to demonstrate how Scanning Electron Microscopy (SEM) as an example of advanced measurement techniques was used for material characterization. A range of samples of pavement engineering materials were examined at a micro-scale using the technique. Selected examples of material characterization are presented. The potential application of microstructural material characterization for the identification of key elements which provide the opportunity to understand fundamental behaviour of pavement engineering materials is demonstrated.

1. INTRODUCTION

A long-term project on the utilisation of advanced measurement techniques for the characterization of pavement materials at a micro-scale has been initiated within the Transport Infrastructure Engineering Group of CSIR Built Environment. The study has been motivated by the fact that material properties at the micro-level have a critical role in the way materials respond to loading at the macro-level. Steyn (2007) presented an overall background to the project. In this paper the focus is on the use of one of the examples of advanced measurement techniques, scanning electron microscopy (SEM).

A literature review was conducted on the use of SEM, with a focus on microstructural assessment of materials in general and on studies related to pavement materials. While SEM has been in use in such fields as metallurgy, in the understanding of material properties, particularly after its commercialization in the mid-1960s (Newbury and Williams, 2000), it is not yet a commonly used technique for material evaluation within the pavement materials engineering field. There are, however, some examples in the literature where SEM analysis has been used in pavement materials engineering related studies. Table 1, gives a list of selected publications that were identified as being closely relevant to the understanding of the use of SEM and its application in pavement engineering materials.

A range of road materials is being analysed under the long-term research project to develop the ability to carry out accurate interpretation of the SEM analysis, Examples of the analyses are presented in Mgangira and Paige-Green (2006). This paper will present selected examples of material characterization illustrating that microstructural material characterization using SEM analysis can add value to the process of pavement material evaluation.

Table 1 Selected publications on SEM application

Publication	Scope/SEM application
Cross, W.M., Duke, E.F., Kellar, J.J and Johnston. 2001	Fracture surface characterization
Hanson, K.F., Van Dam, T.J., Peterson, K.R and Sutter, L.L. 2003.	Chemical composition and morphology
Hussein Malkawa, A.I., Alawneh, A.S and Abu-Safagah, O.T. 1999.	Physicochemical characterization
Katti, D and Shanmugasundaram, V. 2001	Microstructure analysis
Lohnes, R.A and Demirel, T. 1978	Soil fabric characterization
Newbury, D.E and Williams, D.B. 2000	Material characterization.
Shondeep, L. S and Little, D.N. 1999.	Microstructural characterization
Tovey, N.K and Sokolov, V.N 1981.	Qualitative analysis

2. SAMPLE PREPARATION AND EQUIPMENT

2.1 Sample preparation

Samples of pavement material for testing were prepared by technical personnel at the National Metrology Institute of South Africa (NMISA), formerly known as the National Metrology Laboratory (NML). Generally, materials for analysis have to be electrically conductive or rendered so with a carbon coating. In this study, the samples were mounted on double-sided carbon tape on an aluminium 10 mm diameter stub and carbon was evaporated on them prior to analysis.

2.2 Scanning electron microscope

A microscope model A LEO 1525 SEM was used with Oxford INCA software for the Energy Dispersive X-ray Spectrometer (EDS) analysis. The SEM was operated at a voltage of 20 kV, with the RBSD detector with an aperture of 60 µm at a working distance of 12 mm. The EDS spectra were collected for a live time of 100 seconds over the acceleration potential range of 0-10 keV. The process time was 32 µs. The magnification level was varied depending on the level of detail required. Particle size in the images can be determined by the scale bar shown in the lower left corner of each image. Microanalysis was accomplished using the (EDS) coupled to the Scanning Electron Microscope (SEM).

3. EXAMPLES OF MICROSTRUCTURAL MATERIAL CHARACTERIZATION

In this paper, material characterization describes those features of composition and structure of the material that were significant, determined through SEM image acquisition during the analysis. Once selected features of interest were identified on the images, the area was marked and magnified to the required scale. A spot analysis was made to determine the elemental composition, from which it was possible identify the mineral or material within a matrix, for example. Each feature of interest was then marked and referenced by a letter on the image. EDS analysis was performed on each of these spots as shown in the SEM images presented below. For brevity, only representative EDS results are presented for illustration in this paper. More details are given in Mgangira and Paige-Green (2006). The following sections present the results of selected examples of the analysed samples.

3.1 Material identification and comparison

Two sand samples were collected from a good and poor performing unsealed road. The poor performing road exhibited surface deterioration such as faster corrugation development than would generally be considered under similar conditions. The textural features of the samples seemed to be similar, with colour being the obvious difference. According to the standard Atterberg limit tests, i.e. performing tests on material finer than 0.425 mm, both samples were classified as non-plastic. However, when the Atterberg limit tests were performed on the fraction finer than 0.075 mm, the first sample (NAM1) had a plasticity index value of 9.8 and a liquid limit value of 24 while the second sample (NAM2) was again classified as non-plastic. This was an indication that the micro-scale characteristics of the materials played a major role in the performance of these materials.

The samples were then subjected to SEM analysis. Through the SEM images (Figures 1 and 2), it is easier to see the contrast between the grain texture and structure of the two samples. In both images, the magnification was 1000x and the two materials represent samples passing the 53µm sieve size. The grains for sample NAM1 are surrounded by matrix while the grains for sample NAM2 are generally clean, but traces of much smaller particles (1 or 2 microns) can be seen on the surfaces of these grains. The presence and extent of the matrix in samples can be related to the plasticity characteristics of the sands as determined on the <0,075 mm material.

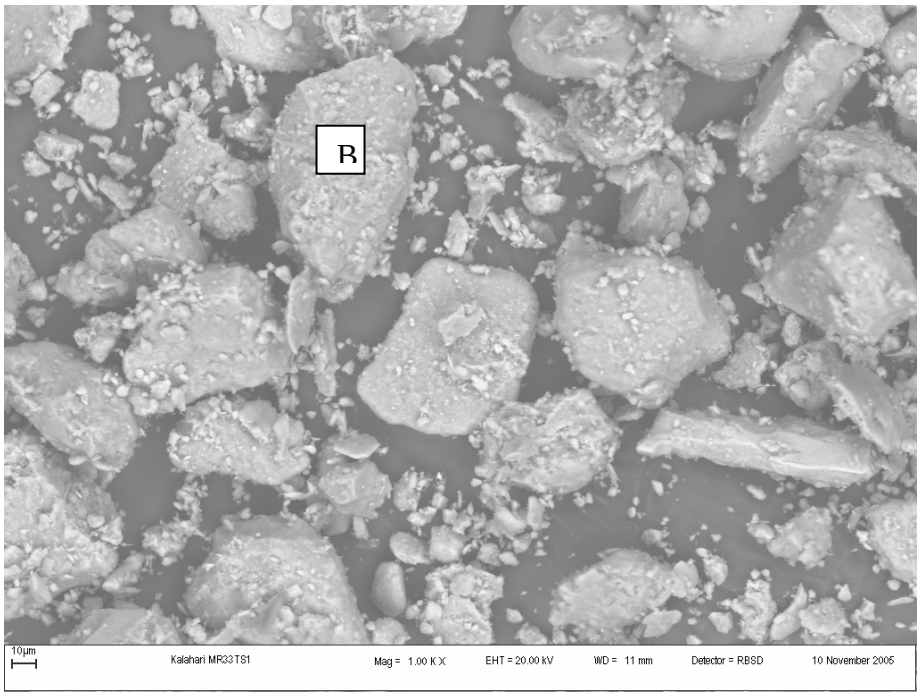

Figure 1 SEM image of sand sample NAM1 (Scale bar 10µm)

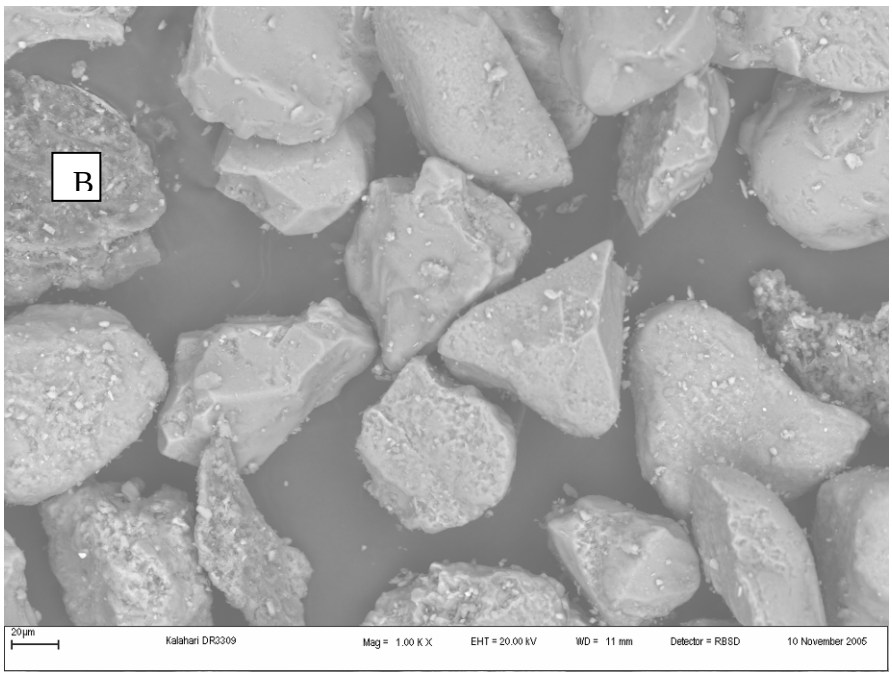

Figure 2 SEM image of sand sample NAM2 (Scale bar 20µm)

EDS scanning results in Figures 3 and 4 show that both materials are predominantly quartz, from the large Si peaks. From the EDS scanning of spots marked B in Figures 1 and 2, the results in Figure 3 distinctively show the presence of iron (Fe), but not in sample NAM2 as can be seen in Figure 4. Sample NAM1 had a reddish colour which can be attributed to the presence of the iron, forming a coating on the sand and silt grains. The reddish sand sample was collected from the unsealed road categorized as a good performing road. Since the textural features of both samples seemed to be similar, this example illustrates that the visual observation of the two sand samples would have led the observer to be only able to characterise the difference between the samples on the basis of colour. In addition the EDS scans from the points marked B in both figures 3 and 4 illustrate that it can be misleading to rely only on the image in interpreting results of the analysis.

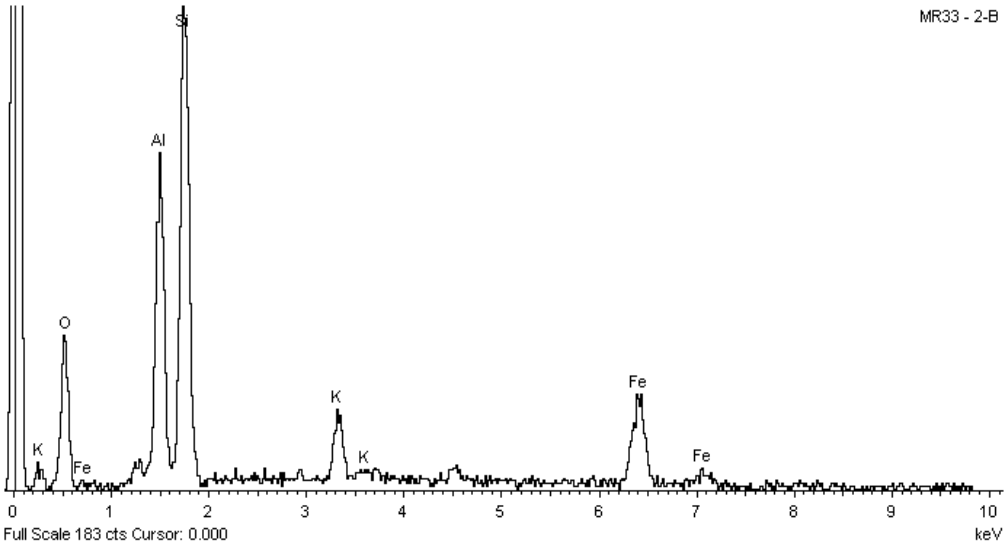

Figure 3 EDS scan from spot B sample NAM1 in Figure 1

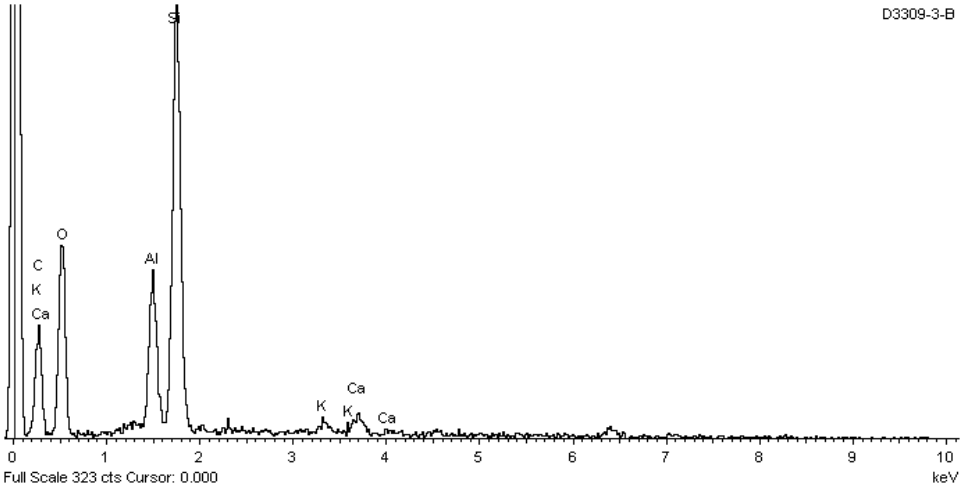

Figure 4 EDS scan from spot B sample NAM2 in Figure 2

On the basis of the microstructural analysis it was possible to determine that particle characteristics such as grain structure morphology are other contributing factors to the difference in performance exhibited by the sand samples. The results show that the combination of the SEM images and EDS analysis add value to material identification and comparison.

3.2 Particle bonding in treated materials

The performance of treated pavement materials depends on the existence of good particle bonding. Kezdi (1979) has discussed the importance of understanding the particle bonding mechanism in order to explain the performance of treated materials. Two examples of particle bonding are given. The first example deals with two samples collected from a road where problems were experienced in that the foam bitumen treated base material was not bonding properly. The second example is that of a sandy sample treated with two types of emulsion, a bitumen emulsion and a water-based polymer emulsion for the purpose of quantifying particle bonding type as a function of the type of stabilization additive.

Figures 5 and 6 show the SEM images of particles of two samples of foamed bitumen at a magnification level of 200x. Both samples had 4.0 per cent bitumen, but differed in the cement content (Mgangira and Paige-Green, 2006). The image in Figure 5 is that of a sample with no cement while the image in Figure 6 is that of a sample with 1.0 per cent cement added.

In Figure 5, the bitumen has accumulated many of the fines and bonded them to many of the quartz particles, but the image also reveals that some of the quartz particles have no bitumen attached to them. Compare this with the image in Figure 6 of a foamed bitumen sample with an addition of 1.0 percent cement. A moderately connected inter-particle bonding can be observed, forming a unified mass.

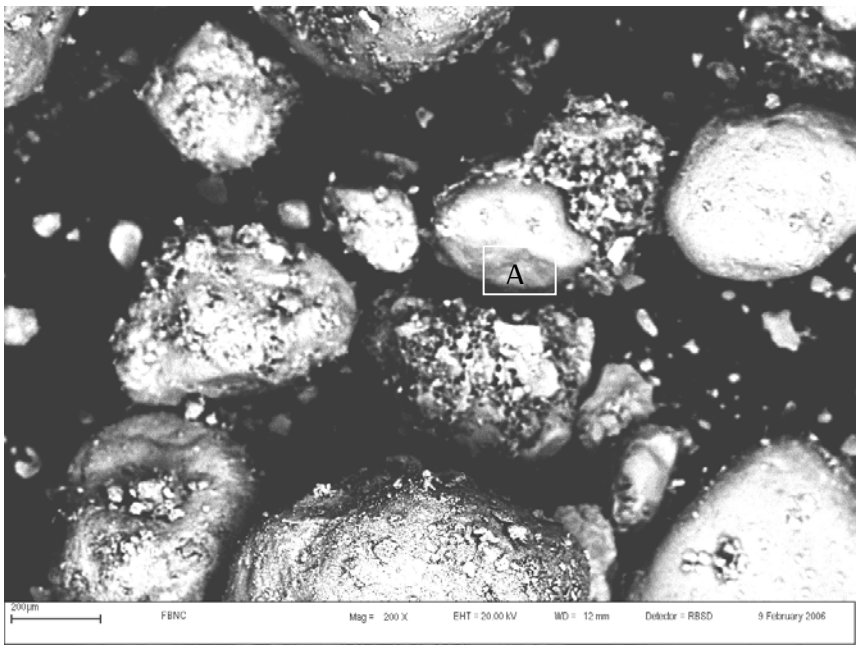

Figure 5 SEM image of foamed bitumen, no cement (Scale bar 200µm)

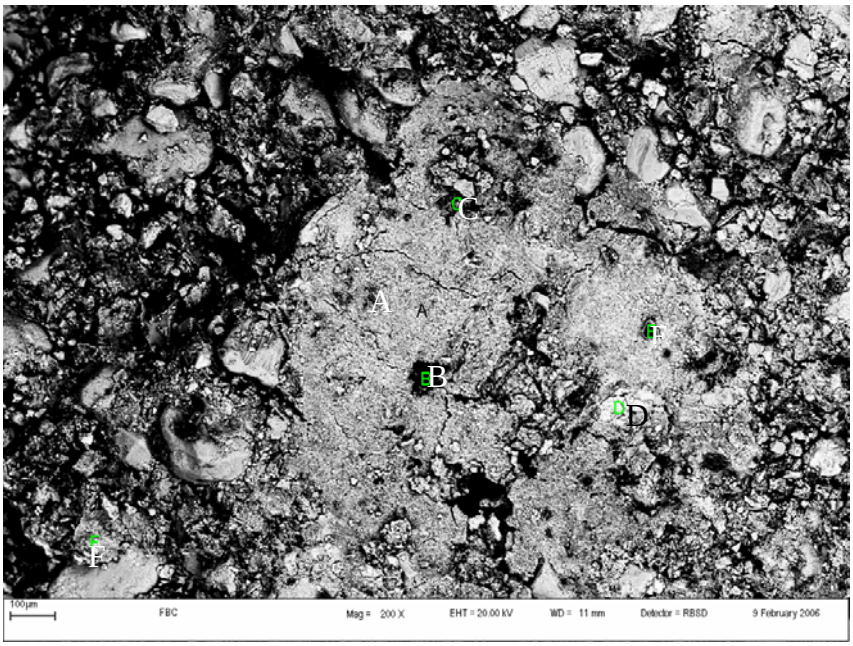

Figure 6 SEM image of foamed bitumen with 1 percent cement (Scale bar 100µm)

EDS scans were performed at several points on the foamed bitumen samples. Figure 7 is the EDS scan for the foamed bitumen without cement, spot A in Figure 5. It shows a large Si peak representing Quartz with moderate Ca and a presence of Mg, Al, Na, Cl and S. Figures 8 and 9 are the EDS scans for the foamed bitumen containing 1.0 per cent cement on the SEM image in Figure 6. Only the EDS results for spots A and E are presented, for the purpose of illustrating the analysis. Si and Ca peaks dominate over C, K, Al, Mg, Fe and Cl in Figure 8, corresponding to spot A. Figure 9 is the EDS scan for spot E showing presence of Cl, Mg, Al and K with minor levels of S, Si and a dominating Ca peak.

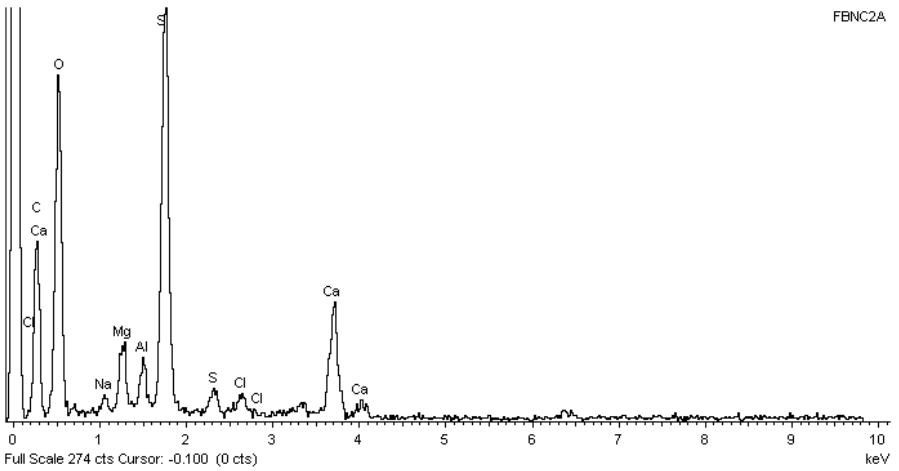

Figure 7 EDS scan of spot A in Figure 5

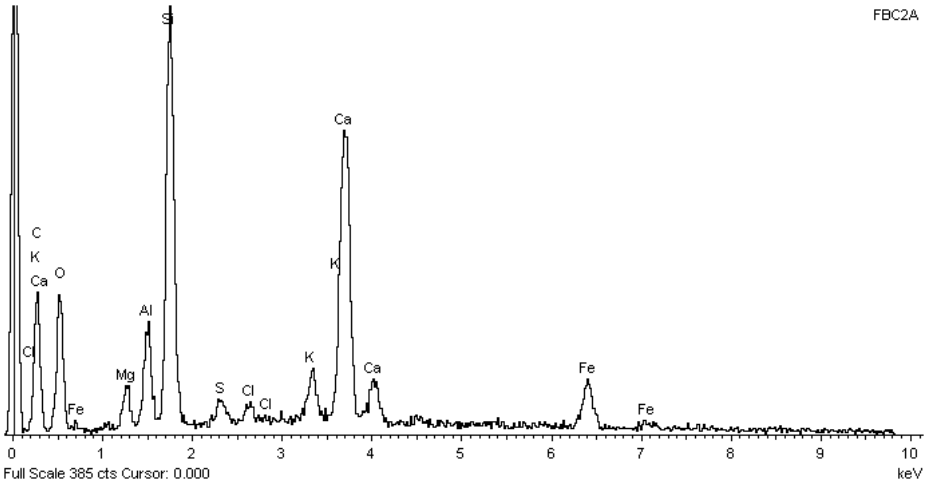

Figure 8 EDS scan from spot A of Figure 6, foamed bitumen with 1 percent cement

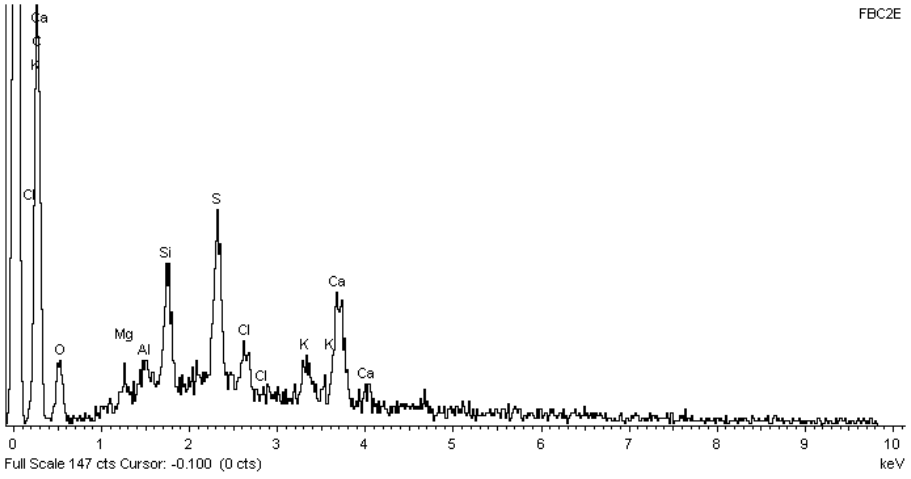

Figure 9 EDS scan from spot E of Figure 6, foamed bitumen with 1 percent cement

The EDS analyses indicated the presence of magnesium, potassium, sulphur, sodium and chlorine, indicating the likely presence of soluble salts for example. While such elemental compositions are dependent on environmental conditions, they show the potential formation of sodium sulphate, sodium chloride, magnesium sulphate and sodium carbonate (Mgangira and Paige-Green 2006). With such potential of salt product formation it was more likely that no significant bonding could be expected with the material in the absence of the cement and therefore this may have accounted for the problems

experienced, as soluble salts could cause damage to the bituminous material (Mgangira and Paige-Green 2006). The SEM image of the untreated material is shown in Figure 10.

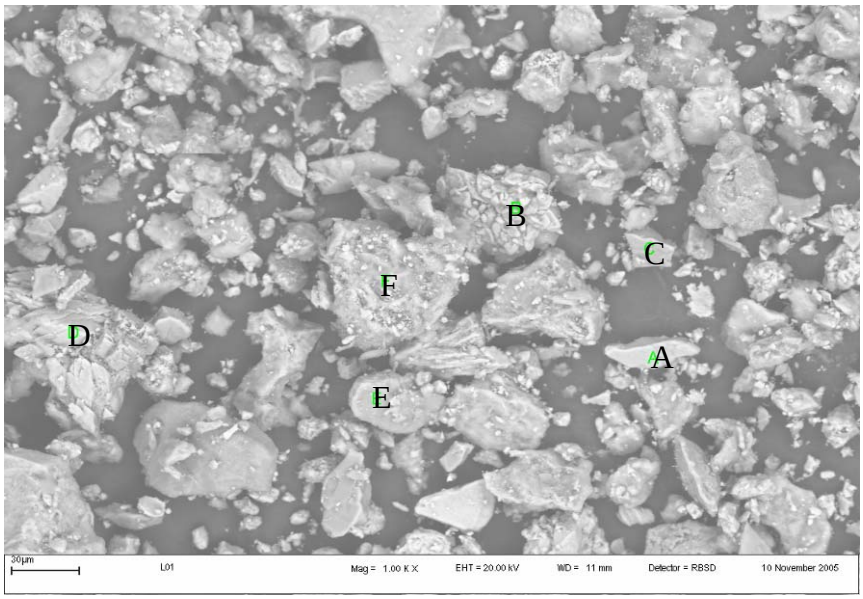

Figure 9 Untreated material (Scale bar 20µm)

The combination of the SEM images and EDS scans reveal that while the majority of the particles were quartz, particles of gypsum, calcite and rock fragments were also present. It can be clearly seen in Figure 10 that some of the quartz particles have masses of smaller particles adhering to their surfaces, indicating that there are possibly surface charges on these particles. The small particles adhering to larger silt particles and coalescing in the matrix appear to be bonded electrostatically. The observed characteristics have the potential to affect bonding. Thus the SEM/EDS analysis revealed the elemental composition, not determined during construction, which could have been the potential cause of the problem experienced on site.

The second example on particle bonding is that of a sand sample treated with bitumen emulsion and a synthetic water-based polymer emulsion. Figures 9 and 10 show SEM images of the sand sample treated with bitumen emulsion and a synthetic water-based polymer emulsion respectively. In order to evaluate the effectiveness of the treatment, the treated samples were subjected to the Abrasion test as suggested by Jones (2003). For the purpose of visually quantifying particle bonding, SEM analysis was also performed on samples taken from the interior of the treated samples after the abrasion test. The SEM images clearly reveal the difference in the particle bonding characteristics. The images show that the particles in the samples treated with 1% bitumen emulsion (Figure 11) are comparatively more closely connected than those in the sample treated with 1% synthetic water-based polymer emulsion shown in Figure 12).

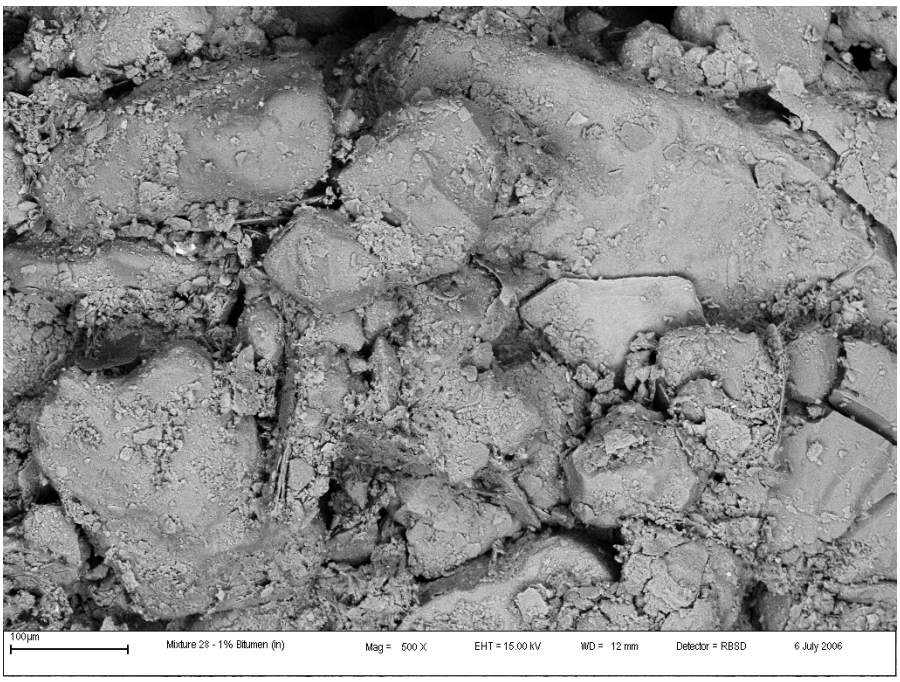

Figure 10 SEM image of a sand sample treated with bitumen emulsion (Scale bar 100µm)

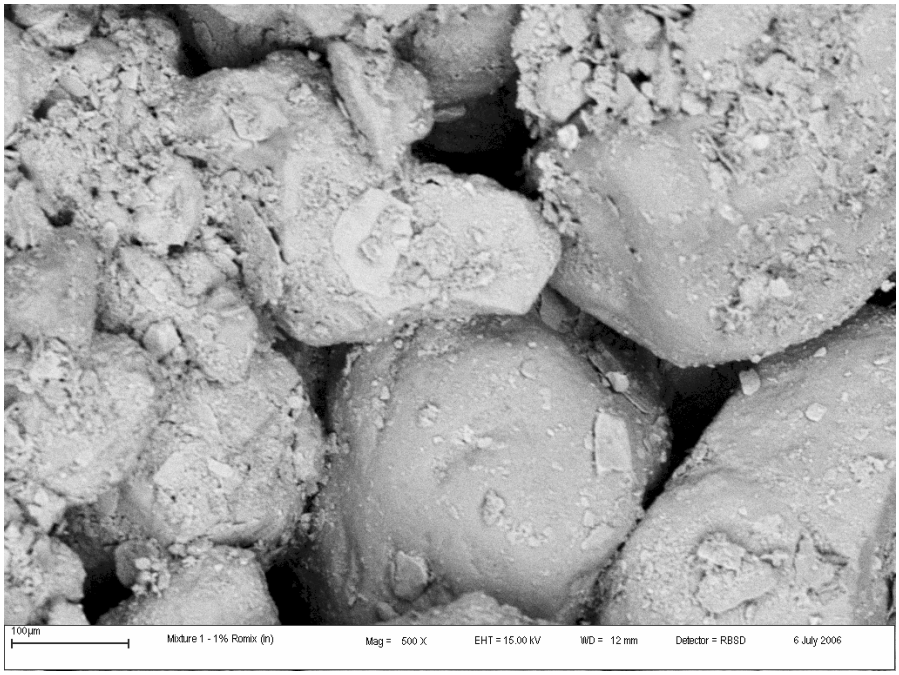

Figure 11 SEM image of a sand sample treated with synthetic water-based polymer emulsion (Scale bar 100µm)

The material treated with the synthetic water-based polymer emulsion had a 28 % loss of material in the Abrasion test after 500 revolutions of brushing compared with 14 % for the same material treated with a bitumen emulsion. Considering that it is the same material and compacted using the same effort, the particle bonding characteristics are obviously product dependent. The example demonstrates that the examination of the particle bonding characteristics at a micro-scale can shed light on the effectiveness of material treatment. Mgangira (2007) suggests that a greater understanding of the physical characterization of the particle bonding of treated soils can also form a basis for the development of effective constitutive models for the mechanical behaviour of soils treated

with non-traditional additives.

3.3 Quantification of carbonation in stabilized material

The final example on microstructural characterization illustrates how SEM/EDS analysis was used to examine carbonation of a sample of stabilized pavement material. The problem of carbonation of stabilized materials has been under investigation at the CSIR since the 1980s. Notable works are cited in Paige-Green (2007). The problem in using materials that are prone to carbonation and that have not been adequately assessed, in road construction, is that the stabilized layer may be weakened without disintegrating and it may be judged strong enough to surface, only to fail later under traffic, especially if the base is of marginal quality and the traffic is unexpectedly heavy (Netterberg and Paige-Green, 1984). It is therefore essential that carbonation susceptibility of materials is determined.

A simple field test for carbonation was described by Netterberg (1984). The stabilized layer is sprayed with phenolphthalein indicator solution. If it does not turn bright red within a few seconds, the material has become carbonated or the stabilizer was never present. In other words, uncarbonated stabilizers should cause the material to turn red. Material that does not turn red but effervesces with dilute hydrochloric acid shows that stabilizer was added, but has since become carbonated (assuming there was no carbonate in the untreated material and checked against untreated material).

Figure 11 is an image of a sample of stabilized sandy subbase. The image in Figure 11 reveals that the material consisted of very fine particles cemented together. The sample did not show any positive reaction with phenolphthalein and effervesced with hydrochloric acid. However the image shows a formation of calcite crystals in the middle...

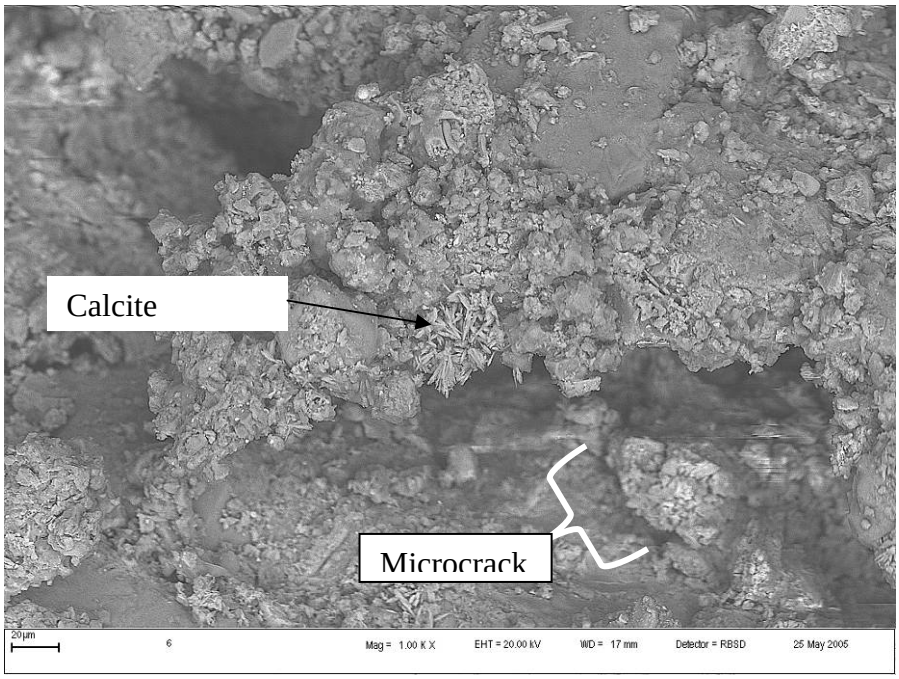

Figure 12 Calcite crystal development on well cemented aggregation particles (Scale bar 20µm)

The formation of the calcite crystals is an indication that carbonation of dissolved lime or cementitious products has occurred (Paige-Green, 2008). Micro-cracks can also be observed in the image. It should be pointed out that, this material was relatively strong, UCS 4 – 5 MPa. Using microstructural characterization it has been possible to illustrate

clearly what Netterberg and Paige-Green (1984) stated, that a layer weakened by carbonation may be judged strong enough to surface only to fail prematurely later.

Other examples on microstructural material characterization are given in Paige-Green (2008). Extensive use of SEM analysis was made to quantify recementation processes in recycled pavement materials, further illustrating the application and benefits of microstructural characterization of pavement materials.

4. CONCLUSIONS

The examples presented above are only but selected examples of the many analyses performed under the project. Over 50 samples have been analysed. The examples discussed have demonstrated that the SEM technique is extremely useful in the characterization of materials at a micro-scale. Micro structural characterization of materials can help to identify key elements contributing to the performance of materials as well as assist in the determination of potential causes of pavement failure.

There are however certain aspects of the technique that one has to pay attention to, when using the technique. For example, sample preparation may affect the quality of results and their interpretation. Higher quality results and reliability will depend on quality and appropriate sample preparation techniques. It should be left to experienced personnel. The examination of replicate or even multiple samples is necessary (based on the small sample examined in each test), but this is time-consuming and costly. For the purpose of forensic investigation and research, this is considered essential.

The examples presented in this paper demonstrate that characterization of materials at the micro-level offers the potential to develop new approaches or complement existing methods in the evaluation of road construction materials, particularly materials that have been treated using non-traditional products. The capability to quantify the microstructure characteristics of materials also offers the opportunity to develop relationships between the microstructure characteristics and performance behaviour of materials.

The study is continuing with the objective of quantifying and defining the parameters that are required to fully describe certain microstructure elements as observed under SEM. In order to achieve this objective, an approach that combines SEM analysis with other advanced observational techniques at the micro-scale and smaller is envisaged. It is anticipated that this project will lead to the development of a systematic methodology in the use of advanced measurement techniques for the characterization of pavement engineering materials at a micro-scale.

To the practitioner, this is a tool to supplement the existing methods for the assessment of structural integrity and durability of pavement materials and to determine differences between design and construction deficiencies in a dispute.

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