TEMPERATURE INDUCED HEALING IN STRAINED BITUMINOUS MATERIALS OBSERVED BY ATOMIC FORCE MICROSCOPY

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ABSTRACT

Bitumen is the binder in the composite material named asphalt concrete. Under cyclic mechanical loading of traffic passing over the pavement, eventually damage will initiate in the pavement, leading to eventual structural failure. This damaging process is accelerated by time dependent change of the mechanical properties of asphalt concrete due to ageing mechanisms like oxidation. Bitumen displays spatial heterogeneity at the micrometer scale, which has been observed by atomic force microscopy (AFM). The mechanical properties of the elliptical, microstructural domains of bitumen are distinct from those of the continuous phase. This introduces stiffness discontinuities in the material, which under mechanical loading will concentrate stresses at the interfaces, and thus the locations where early stages of damage will develop.

This work aims at in situ probing of the crack healing of bituminous materials as a function of moderate temperature changes. The bitumen was prepared on a flexible substrate which was mechanically strained to induce damage. AFM measurements of the strained bitumen specimen provides evidence of the crack initiation at the interface and the predominant propagation of cracks through the elliptical domain phases. Healing of these cracks was observed after applying modest amounts of heat to the material. Meanwhile the process was monitored in situ with AFM. With increase of temperature one of the phases starts softening, while the material as a whole remains solid. This allows the phases to rearrange and meanwhile eliminating micro cracks at the interface.

1. INTRODUCTION

When asphalt concrete is mechanical loaded, cracks will easiest form within the bituminous binder that holds together the other components of asphalt, i.e. the fillers and aggregate particles. Bitumen is mechanically speaking the weak link in the asphalt concrete composite. Damage may occur within the bitumen (cohesive damage) or at the interface between the bitumen and the aggregates (adhesive damage). Yet, not much information is available on the damage characteristics of bitumen at the micrometer scale. Though several researchers emphasized on the importance of probing the pre- macro crack regime for improved understanding of the damage process in bituminous materials at the macroscopic level [1, 2].

AFM is a scanning probe technique which measures both topological morphology and the spatial variations of mechanical properties. AFM has been used to probe

bituminous for more than a decade. Various authors have reported that on the micrometer scale bitumen exhibits a two phase morphology at the micrometer scale. AFM measurements of the bitumen microstructure reveals elliptical domains with a corrugated topology in the middle (termed as wrinkling pattern), ordered along the long axis of these domains which are surrounded by a continuous phase [3-5].
The present study focuses on probing with AFM the morphology of early stage cracks at the micrometer scale in bitumen as well as the disappearance of these micro cracks as function of moderate changes of temperature.

2. MATERIALS

Two bitumen samples of penetration grade 70/100 and 160/220, obtained from Q8 have been selected for this study. AFM images were taken in tapping mode using ‘Multimode V’ from Bruker. All scans were performed in air with RTESP (Bruker) cantilevers with a nominal force constant of 40 N/m and a resonance frequency of 330 kHz. An aluminium tape was used as the primary substrate and an AFM sample puck (12 mm diameter steel disk) was used as a secondary substrate.

3. METHODS

The sample was prepared on a piece of aluminium tape which was adhered to the steel substrate from the adhesive side. An amount of 20mg bitumen was placed on the tape and was heated using a heater plate for 30 seconds at 100°C to obtain a smooth thin and shiny film.

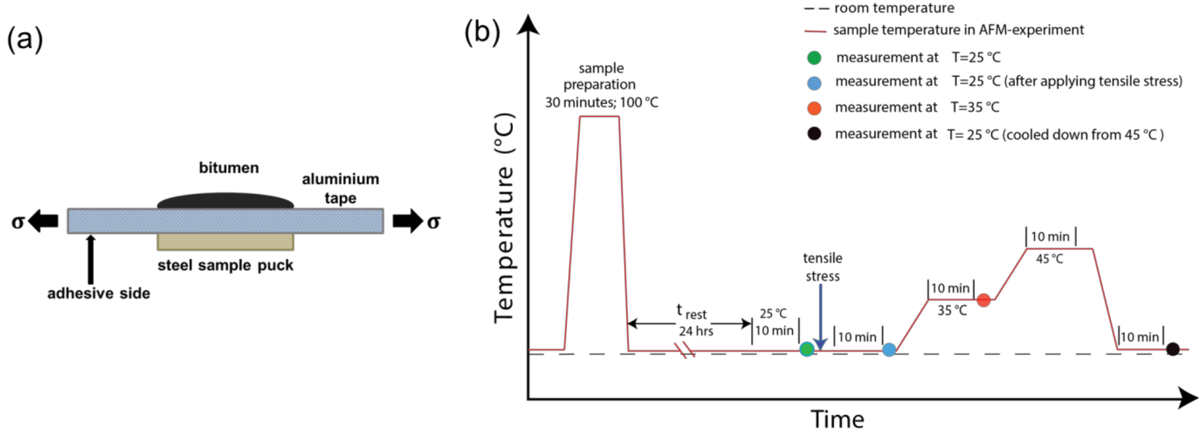

Figure 1: Schematic of (a) sample straining and (b) sample thermal conditioning and imaging protocol.

Two specimens were prepared for each bitumen grade. The samples were thermally conditioned inside an oven at 100°C for 30 minutes as shown in Figure 1b, followed by cooling in ambient air and storage at room temperature for 24 hours. One of these specimen was subjected to damage and a subsequent temperature dependent healing study, while the other sample was used as control. The neat specimen was first imaged by AFM at 25°C. The tape was then strained to initiate damage in the bitumen sample, Figure 1a. Then the strained sample was held for 10 minutes at the measuring temperature of 25°C and then imaged, Figure 1b. Next, the heat conductive aluminum tape was heated to 35°C, and then probed by AFM after 10 minutes of equilibration. The same procedure was followed, but now the temperature was raised to 45°C, imaged after 10 minutes rest. Finally the sample was cooled down to 25 °C, and imaged again, Figure 1b.

4. RESULTS AND DISCUSSION

For both the bitumen samples (70/100 and 160/220) AFM topography and phase images (30×30 µm) were recorded, for both the neat and mechanically loaded samples, as a function of temperature, Figure 2. The characteristic features observed for the microstructure of the neat material are the presence of elliptical domains and the continuous phase surrounding these domains, Figure 2 a(i) and b(i). The phase images show a significant phase contrast between the domains to the continuous phase and an existence of lamellar phase (tertiary phase) around these domains. The morphology of the microstructure is found to depend on bitumen grade.

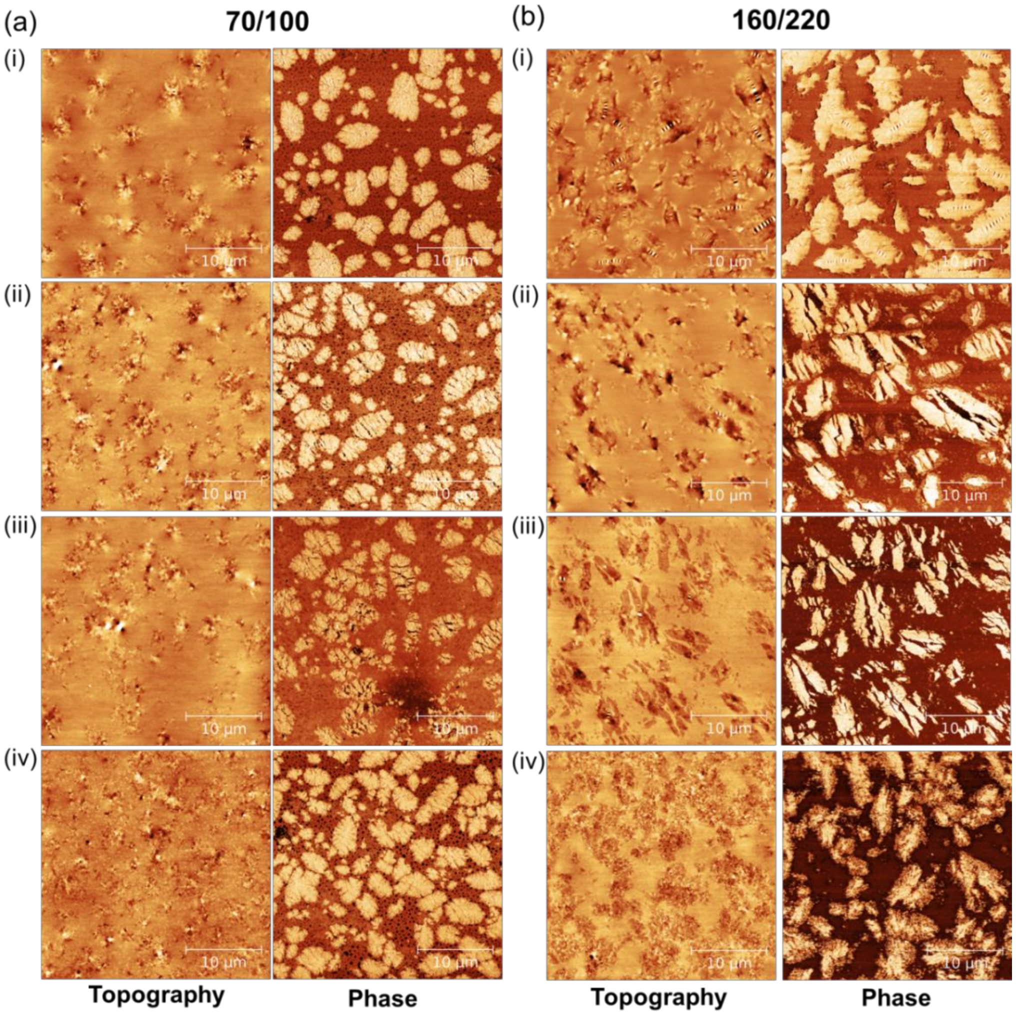

Figure 2: AFM images of a) 70/100 and b) 160/220 bitumen. i) neat sample at 25°C, ii) strained sample at 25°C, iii) same at 35°C, iv) same after cooling to 25°C.

Next, the microstructural response of mechanical loading of bitumen at room temperature is studied. Figure 2 a(ii), b(ii) reveal the impact of loading at the microstructural level. The existence of cracks in elliptical domains is observed. Crack lengths and widths are found to be higher for Q8 160/220 bitumen compared to Q8 70/100. Further, at increasing the temperature from 25 °C to 35 °C, the early cracks remain visible, Figure 2 a(iii) and b(iii). Though around the edges of the elliptical domains softening is observed, possibly due to increased interfacial energy. Moreover the softened continuous phase tends to push the fragmented domains further apart. This may explain the increase of ‘crack widths’ within the domains at elevated temperature.

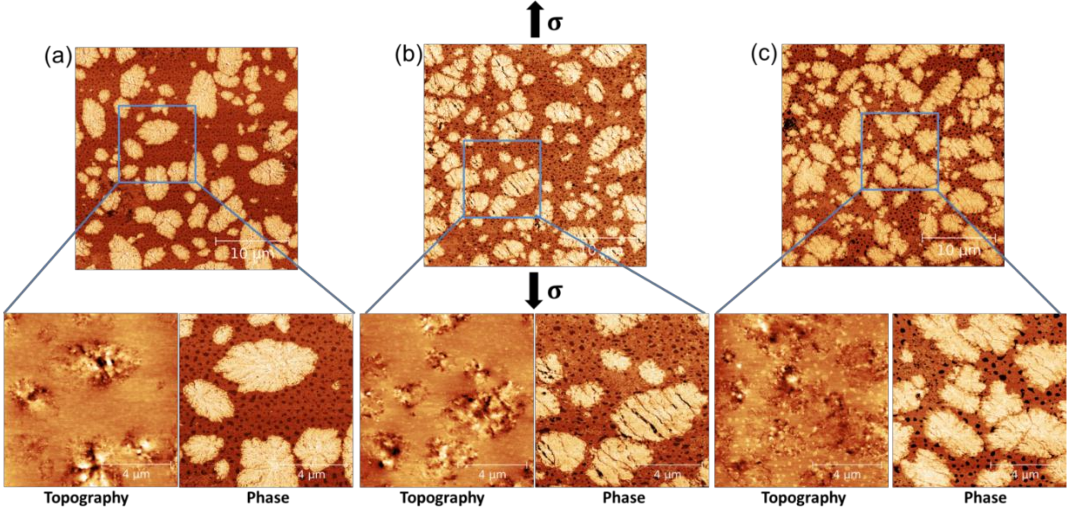

Figure 3: Crack closing observed by AFM of 70/100 bitumen. a) Neat sample at 25°C, b) strained sample at 25°C c) crack closure after cooling from 45°C to 25°C.

At 45°C both material phases have softened and cooling to 25°C, leads to rearrangement of the phases. For 70/100 the earlier observed fragments are pushed together again, allowing for healing by diffusion of molecules across the earlier crack surfaces, Figure 3. For 160/220 the fragments still display gaps at 25°C, so longer times at elevated temperatures would be required to observe the same.

5. CONCLUSIONS

By means of AFM the effect at the microstructural level of mechanical loading of bitumen was shown. Early cracking and fragmenting of domains was observed. Modest thermal changes of only 20°C lead to closing of the gaps between the fragments, allowing for strength recovery by diffusion. Surprisingly, this effect was found to be most pronounced for the harder 70/100 bitumen.

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