BITUMEN AGEING AND ITS EFFECT ON THE SURFACE MICROSTRUCTURES

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INTRODUCTION

Ageing of roadwork materials causes microstructural changes of the asphalt binder. Therefore, fast ageing simulations of roadwork materials, such as Viennese Ageing Procedure (VAPro) or Pressure Ageing Vessel (PAV)[1], can be conducted in laboratory to determine the asphalt long-time performance on the streets in a quick process. Topological deformations caused by ageing can be detected with cryo-ESEM and Atomic Force Microscopy (AFM). Fluorescence Spectroscopy is used to track the ageing state of the asphalt binder.

EXPERIMENTS / FUNDAMENTAL OF THE PROBLEM / EXAMINATIONS

Sunlight (photo-oxidation), atmosphere (reactions of bitumen with reactive oxygen species (ROS)), rain water (dissolving acids and oxygen containing species) and thermal oxidation (during treatment process in refinery, during transportation from refinery to pavement area and the embedding process itself) are the main factors for the change over its lifetime. The main bitumen components are Asphaltenes, Saturates, Aromatics and Resins. Negative electrospray ionization Fourier transform ion cyclotron resonance mass spectrometry [ESI(-)] FT-ICR-MS experiments show that ageing reduces condensed aromatic compounds to alicyclics and open chain aliphatics [2]. The overall fluorescence intensity decreases with oxidation state (see figure 1) and structural changes can be observed on the surface of the roadwork material with cryo-ESEM.

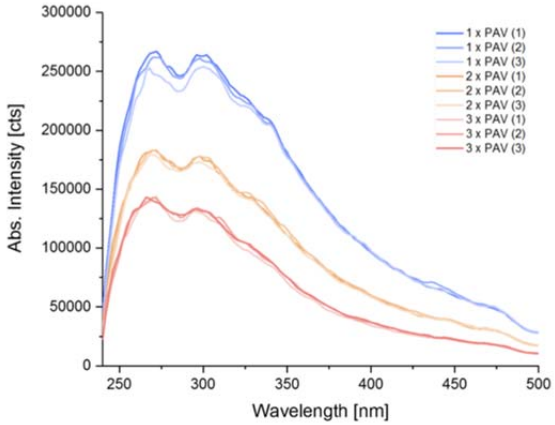

Figure 1: Fluorescence spectra of PAV aged samples. Decrease in overall absolute Intensity.

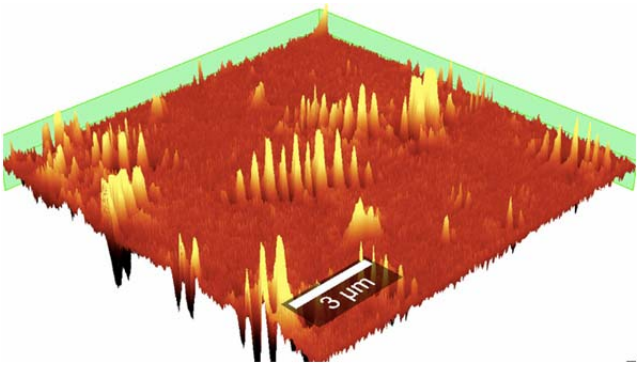

Figure 2: Non - aged AFM of bitumen after 24 h relaxation time.

Cryo-ESEM shows an increase of the surface microstructures with increasing the oxidation state with PAV. A change in the size distribution is observed, where the size of the surface structures alters from 2 – 10 µm in length and up to 1 µm in width.

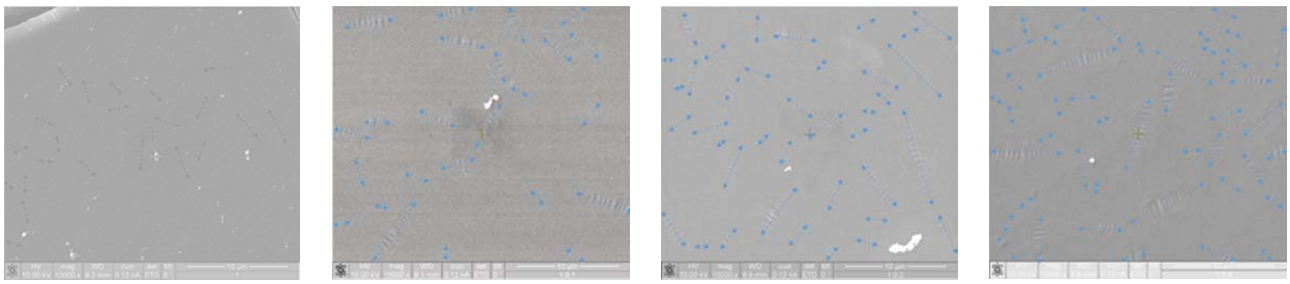

Figure 3a - d: cryo – ESEM of (a) non - aged, (b) 1 x PAV (c) 2 x PAV (d) 3 x PAV aged bitumen samples

A higher resolution of the surface microstructures can be achieved with AFM. A dependence of the ageing state with the surface morphology can be seen during the microstructure formation process. The surface roughness increases with the ageing state (see figures 4a -c).

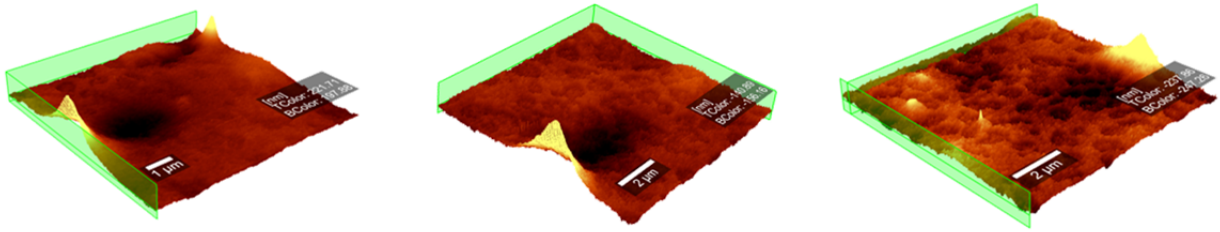

Figure 4a - c: AFM of (a) 1 x PAV rms = 22,41 nm (b) 2 x PAV rms = 34,05 (c) 3 x PAV rms = 46,64 nm aged bitumen samples.

CONCLUSION

A strong correlation between the PAV ageing state and surface properties could be observed. Since microchanges can pave the way to macrochanges (embrittlement and cracks visible to the eye), it is worth to look at the correlations and to elucidate methods that can predict a materials performance to save resources and energy.

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REFERENCES

[1] Daniel Steiner, Bernhard Hofko, Markus Hospodka, Florian Handle, Hinrich Grothe, Josef Füssl, Lukas Eberhardsteiner & Ronald Blab (2016) Towards an optimised lab procedure for long-term oxidative ageing of asphalt mix specimen, International Journal of Pavement Engineering, 17:6, 471-477. DOI: 10.1080/10298436.2014.993204

[2] Florian Handle, Mourad Harir, Josef Füssl, Ayşe N. Koyun, Daniel Grossegger, Norbert Hertkorn, Lukas Eberhardsteiner, Bernhard Hofko, Markus Hospodka, Ronald Blab, Philippe Schmitt-Kopplin, and Hinrich Grothe (2015)Tracking Aging of Bitumen and Its Saturate, Aromatic, Resin, and Asphaltene Fractions Using High-Field Fourier Transform Ion Cyclotron Resonance Mass Spectrometry. Energy & Fuels 2017 31 (5), 4771-4779. DOI: 10.1021/acs.energyfuels.6b03396