Vienna University of Technology – Faculty of Civil Engineering

Christian Doppler Laboratory for Performance Based Optimization of Flexible Pavements

Advanced Test Methods for Characterization of Bitumen Chemistry, Microstructure, and Its Micromechanical Properties

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FROM nano TO macro-SCALE - MULTISCALE MODEL FOR ASPHALT

Recent progress in both experimentation and the micromechanical description of the behavior of multi-composed and hierarchical materials provided the basis for the development of so-called multiscale models for both man-made and biological materials. As regards the description of the complex thermo-rheological properties of asphalt concrete, a multiscale model for bituminous mixtures is currently developed at the Christian Doppler Laboratory for "Performance-based Optimization of Flexible Pavements" at Vienna University of Technology (see Figure 1). This model allows us to relate macroscopically observable asphalt properties to finer-scale information and, basically, to the performance of the underlying binder material (bitumen). Within each observation scale, the characteristics (such as structure and material properties) of the constituents present at this scale are taken into account. Moreover, changes in scale characteristics, resulting from mechanical loading and/or environmental conditions may be considered at the respective scale of observation.

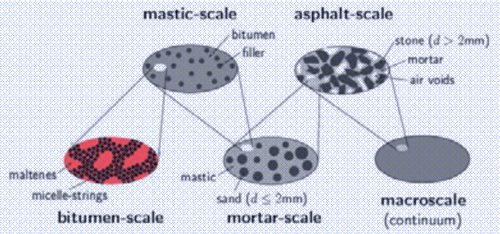


Figure 1: Observation scales introduced for multiscale modeling of asphalt.

Since bitumen mainly governs the thermorheological behavior of asphalt, its characterization at the bitumen-scale (see Figure 1) and, moreover, the link between bitumen chemistry and bitumen performance are essential for the predictive capability of the anticipated multiscale model. Hence, advanced chemical, microscopical, and micromechanical test methods are employed for the identification of input parameters for the proposed multiscale model at the lowest scale of observation (see Figure 2).

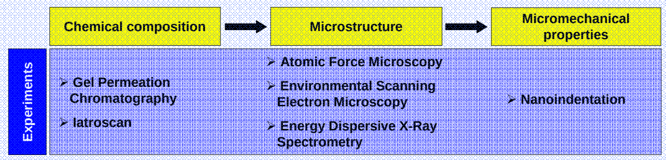


Figure 2: Identification of input parameters at the bitumen-scale.

HIGHLIGHTING THE MOLECULAR COCKTAIL OF BITUMEN

Bitumen is composed of a wide range of hydrocarbon molecules, ranging from low-molecular weight compounds, such as saturates and aromatics, to highly polar large-size asphaltenes (see Figure 3). In order to determine this complex chemical composition of bitumen, GPC and the Iatroscan method are employed.

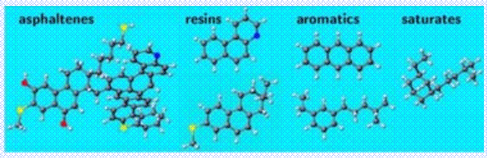


Figure 3: 'Molecular cocktail' present in bitumen.

GEL PERMEATION CHROMATOGRAPHY (GPC)

GPC allows determination of the molecular-size distribution of bitumen, which is shown in Figure 4 for a polymer-modified bitumen in original and aged condition. With increasing ageing (A→B→C) the distribution shifts to higher molar masses. Moreover, a small amount of large-weight molecules corresponding to the polymer content is visible as a separate peak.

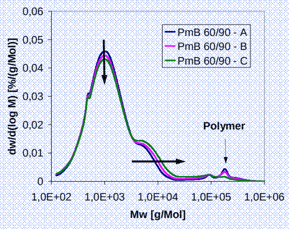


Figure 4: GPC results of bitumen.

IATROSCAN

In order to determine the molecular fractions, such as saturates, aromatics, resins, and asphaltenes (see Figure 3), the **Iatroscan** method is employed. A typical chromatogram of bitumen can be seen in Figure 5. The influence of ageing on the molecular fractions of bitumen is illustrated in Figure 6.

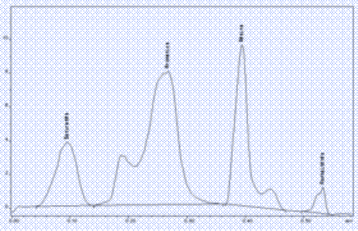


Figure 5: Typical Iatroscan chromatogram.

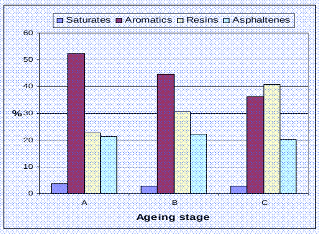


Figure 6: Generic fractions of bitumen.

EXPLORING THE FINER SCALES - BITUMEN MICROSTRUCTURE & MICROMECHANICAL PROPERTIES

ATOMIC FORCE MICROSCOPY (AFM)

AFM provides insight into the surface topography and, to a certain extent, into the mechanical properties of bitumen. The results from AFM measurements allow identification of four material phases with different mechanical properties at the bitumen surface. Two of these phases form so-called "bee-shaped" structures surrounded by stiffer crystalline-like structures (Figures 7 & 8). The latter is again embedded into a softer matrix phase. By increasing the penetration depth the stiffer parts seems to become interconnected, building a string-like structure, emerging and immersing at the bitumen surface.

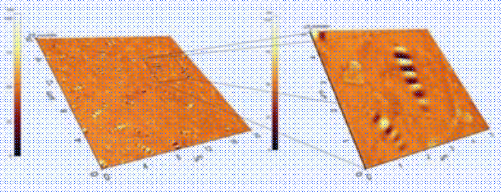


Figure 7: "Bee-shaped" microstructure at bitumen surface identified by AFM measurements.

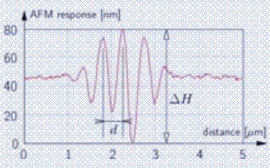


Figure 8: Section through a „bee".

ENVIRONMENTAL SCANNING ELECTRON MICROSCOPY (ESEM)

ESEM gives insight into bitumen microstructure, appearing as two-phase system in form of string-like network structures embedded into a matrix material. This network structure is presented in Figure 9 (a) as well as the influence of ageing on the network structure (b, c).

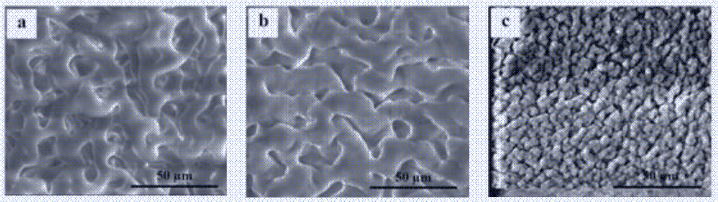


Figure 9: ESEM images of bitumen - original (a) and aged (b, c).

NANOINDENTATION (NI)

In order to identify the micromechanical properties of bitumen, nanoindentation (NI) is employed. Hereby, the measured penetration history allows back calculation of viscoelastic model parameters (see Figure 10). Additionally, the application of the so-called grid-indentation technique gives access to the mean values and the spatial distribution of the model parameters which are related to the different bitumen phases. The results depicted in Figure 10 confirm the microstructure already observed by ESEM testing and, even more important, show that the different phases exhibit different material behavior.

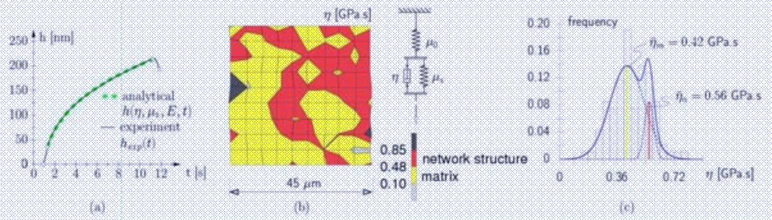
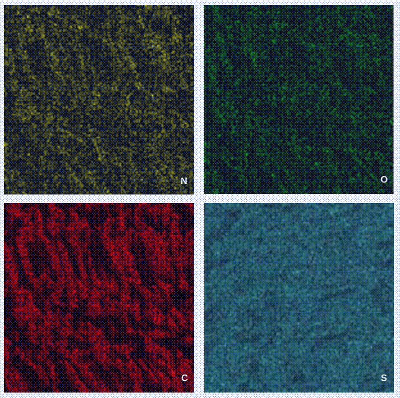


Figure 10: (a) Typical penetration history, (b) surface plot of viscosity η, and (c) corresponding frequency plot.

ENERGY DISPERSIVE X-RAY SPECTROMETRY (EDX)

ESEM in connection with EDX-spectrometry enables the determination of both the geometrical parameters *and* the chemical composition of bitumen. Consequently, correlations between particle size, chemical composition, and their contribution to the overall concentration can be established. Figure 11 shows an ESEM image of bitumen and the corresponding EDX elemental maps for sulfur, oxygen, nitrogen, and carbon.



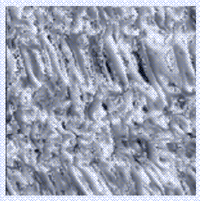


Figure 11: Elemental maps for nitrogen (N), oxygen (O), carbon (C), and sulfur (S).

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