Optical Characterization of Temperature- and Composition-Dependent Microstructure in Asphalt Binders

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Abstract. Asphalt pavement binders contain a rich variety of micrometer-sized inclusions of poorly understood origin that vary in size, shape and density depending on temperature and chemical composition, and that may be connected to the binder's strength and a pavement's durability. Atomic force microscopy (AFM) has been widely used to observe these microstructures, but is limited to surface structures near room temperature (RT). **Here we introduce noncontact optical microscopy and optical scatter to characterize asphalt binder microstructures at temperatures ranging from RT to 85 C for both low- and high-wax composition.** We benchmark optical measurements against conventional rheometric measurements of the temperature-dependent bulk shear modulus G*(T). **The main findings are: (1)** Elongated 5μm, striped microstructures (known from AFM studies as ``bees'' because they resemble bumble-bees), are resolved optically, found to reside primarily at the surface, and to disappear after a single heating-cooling cycle. **(2)** Smaller microstructures with no observable internal structure are found to reside primarily in the bulk, to persist after multiple thermal cycles and to scatter light strongly. Scatter from ``ants'' decreases monotonically to zero with heating and recovers almost completely upon cooling back to 15 C, albeit with distinct hysteresis. **(3)** Rheometric measurements of G(T) reveal hysteresis that resembles that observed by optical scatter, suggesting a possible connection between microstructure density and G(T). **The results demonstrate the versatility of optical measurements in characterizing functionally important binder properties over a wide temperature and composition range.**

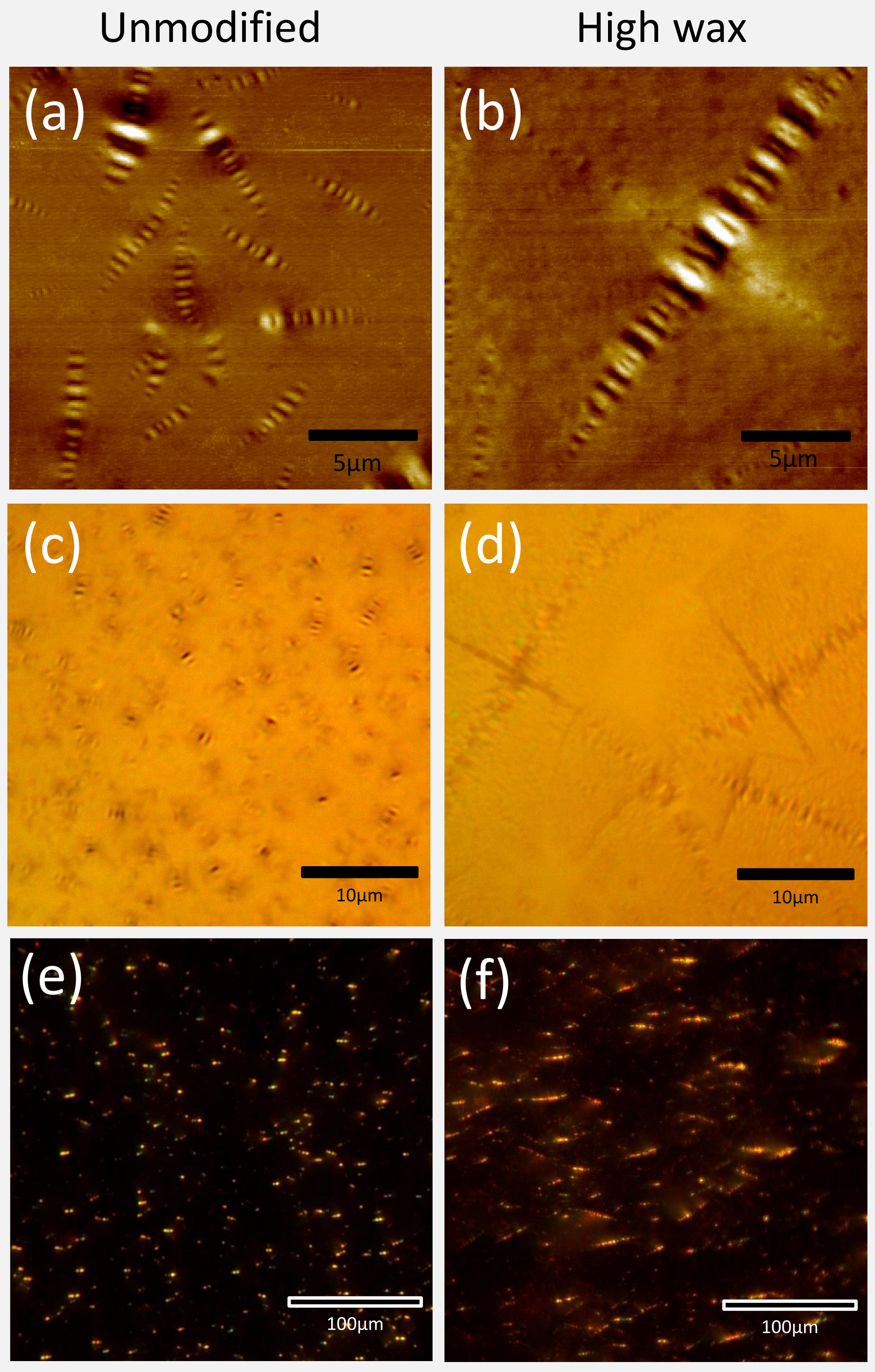
Overview

The majority of paved roads across the world are built using asphalt mixtures composed of approximately 5% bitumen binder and 95% stone, gravel and sand aggregate. Though only a small weight fraction, the bitumen is primarily responsible for a road's mechanical properties. Ideal binders possess high stiffness at high temperatures, low stiffness and high relaxation rates at low temperatures, and high resistance to fatigue cracking at intermediate temperatures. In this work we explore possible connections between the binder's bulk shear modulus and internal microstructure.

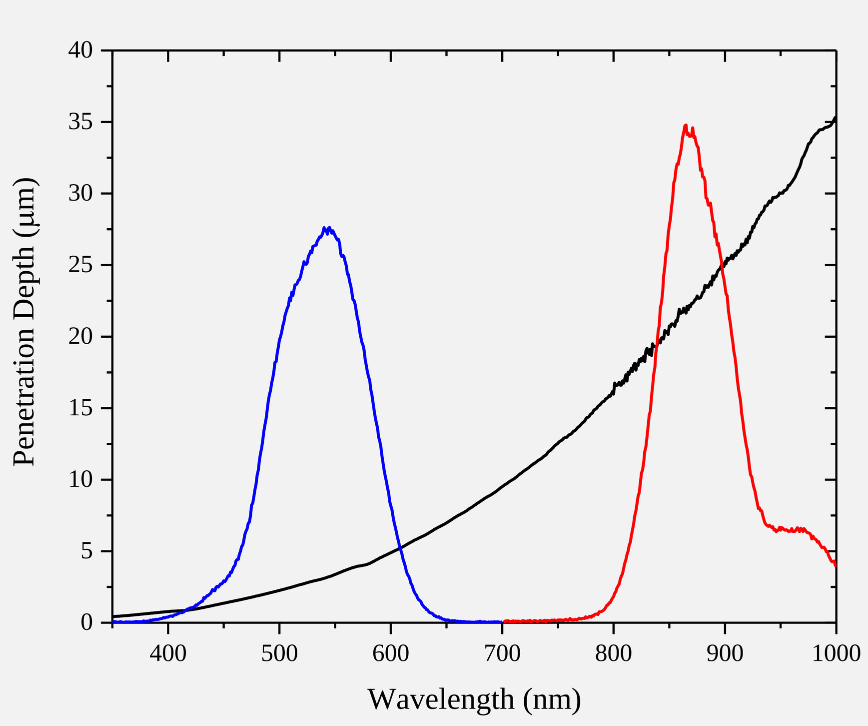
Methodology

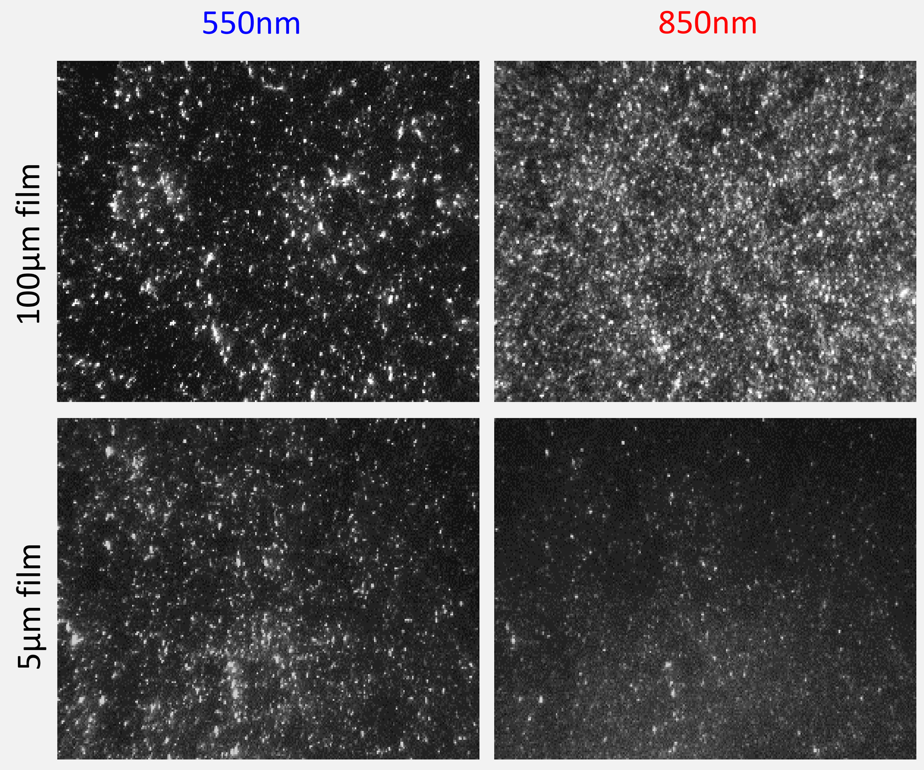
Samples: an unmodified binder (PG 64-22) and the same with a 1% (by weight) wax additive were compared in this study.

Optical Microscopy and Scatter: AFM images (a,b) show elongated, rippled microstructures that are widely observed on the surfaces of room temperature binders. In this work we use optics to overcome the limitations of AFM: (1) It can only image surface structures. (2) Scans are slow and limited to small areas. (3) Because contact is required, it has difficulty imaging structures at elevated temperatures. As seen in (c,d), optical microscopy rivals AFM in its ability to resolve microstructures of interest. Using darkfield microscopy, linear scatter can be observed over a larger area of the binder (e,f).



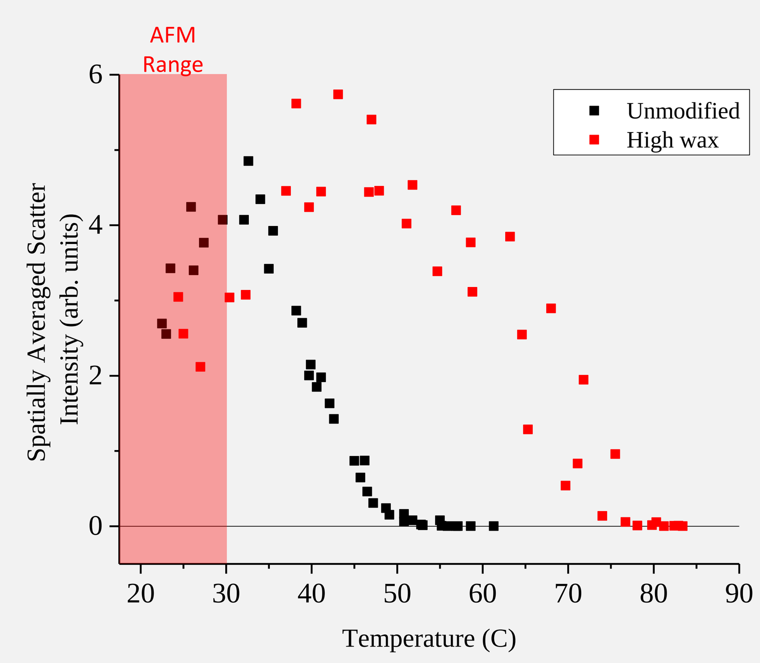
Frequency Dependence: Optical measurements were conducted in two wavelength bands with contrasting penetration depths: (1) visible (540 ± 50 nm) to probe near-surface microstructures; (2) near-IR (870 ± 30 nm) to probe deeper bulk microstructures.





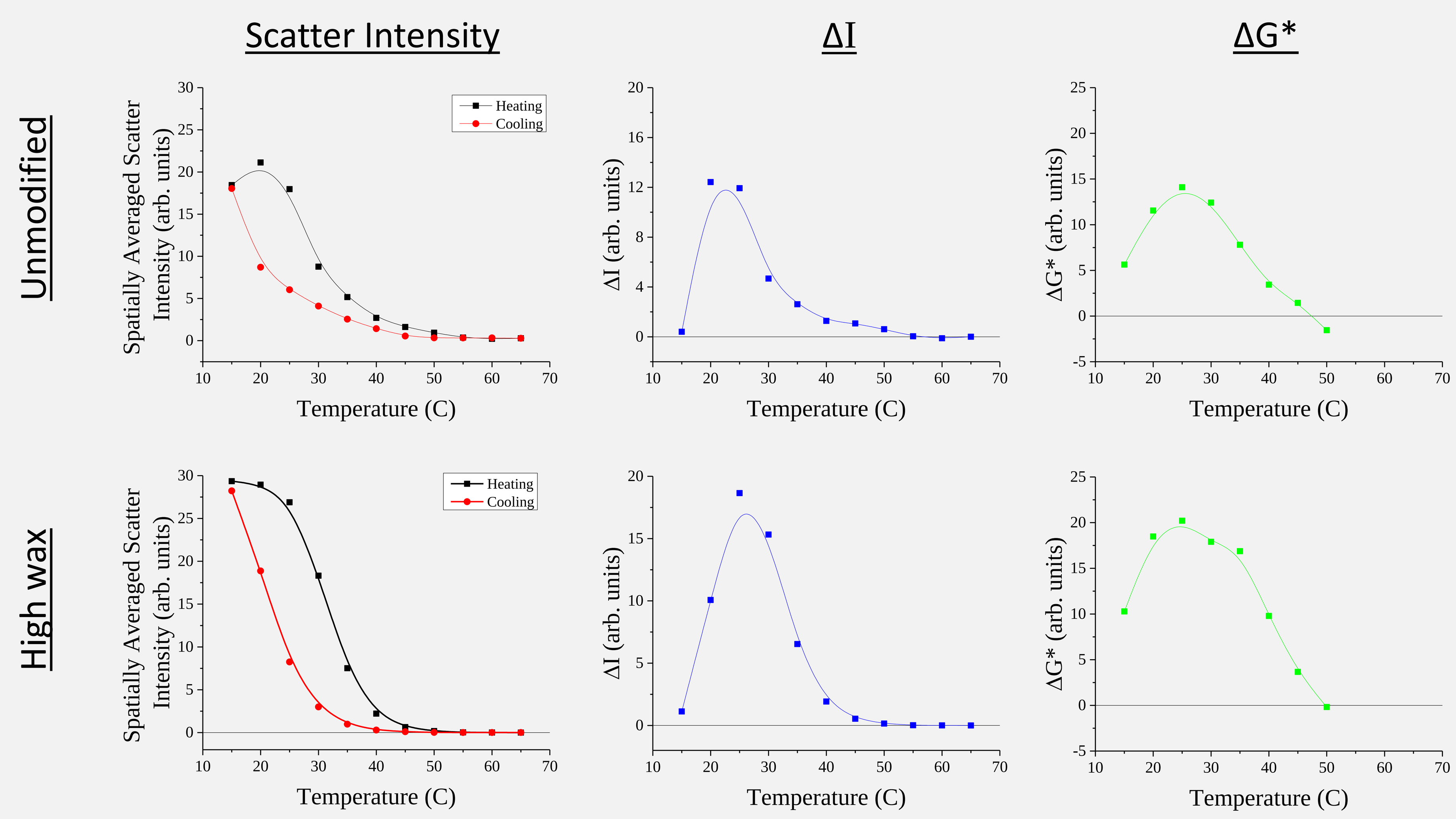
Dynamic Shear Rheometry: A small disk of binder is affixed between two parallel plates. An axial, oscillatory force is applied and the shear modulus (G*), a macroscopic property important to stiffness, is measured. G* is measured at 5 degree temperature increments in the range of 15 C to 65 C, both during heating as well as cooling.

Results



First Thermal Cycle: On first heating from RT to 85 C, scatter from both binders is dominated by large surface bee-like structures. Scattered intensity first increases then drops to zero. Scatter in the high-wax sample persists to higher temperature, because of larger, more robust bee structures. Upon cooling, scatter in both samples returns to a significantly reduced level, indicating that surface bee-like structures are eliminated.

Subsequent Thermal Cycles: Scatter on later thermal cycles is dominated by smaller bulk microstructures. Scattered intensity full recovers after each cycle. However, there is a distinct hysteresis. The difference ΔI between the heating and cooling half-cycles is plotted in blue. Shear rheometry yields a very similar hysteresis in the bulk shear modulus ΔG*, plotted in green, suggesting that bulk microstructure density is intimately related with bulk shear modulus.



Conclusions

1. Unlike AFM, optical microscopy and scatter distinguish surface from bulk microstructures in asphalt binders, and track changes in their size and density from RT to ~100 C.
2. Comparison of temperature-dependent optical scatter and shear rheometry reveals matching hysteresis behavior in microstructure density and bulk shear modulus, suggesting that bulk microstructures play a key role in determining mechanical properties.
3. Optical measurements provide a fast, non-invasive method for screening the internal microstructure of asphalt binders.

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