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Rheological and Microstructural Assessment of Complex Bituminous Binders

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Abstract

In the past years, the use of liquid additives as bitumen modifiers has increased to enhance or adjust the properties of bitumen for wide range of applications. Their molecular composition and mutual interaction result in a specific phase morphology in the binders. Hence, there is a need to evaluate the phase and interphase framework and physical properties of complex binders. The RILEM Technical Committee 272-PIM 'Phase and Interphase behaviour of innovative bituminous Materials', Task Group TG1 investigates the characterization of assessing phase interphase properties of complex bituminous binders. In this framework, three liquid additives have been selected with different viscosity, nature and use. They were blended with base bitumen to achieve similar consistency and the blends were further aged. Physical properties were evaluated through rheology using dynamic shear rheometer (DSR) in a wide range of conditions. The phase morphology was addressed by atomic force microscopy (AFM), differential scanning calorimetry (DSC) techniques. AFM and DSC results, from fresh and aged binders, reveal that each binder display specific phase morphology and glass transition characteristics, manifesting mutual compatibility of the individual binder components. This approach of binder assessment: combining phase characteristics and rheological response can assist material selection to specific applications.

Key words: Rilem, complex bituminous binder, DSR, DSC, AFM.

Rheological and Microstructural Assessment of Complex Bituminous Binders

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TNO innovation for life KRATON TU Delft Université Gustave Eiffel

Rilem PIM

- Rilem TC 272 - Phase and Interphase in bituminous Materials
  - TG1 is about testing of complex bituminous binders
  - 17 labs participating from Europe and US
- Experiments
  - Physical properties and chemical structure
  - In a wide range of conditions

	Label	Description
Group 1	**Bit1**	Straight run bitumen 35/50 source Europe
	PmB1	*Commercial Polymer modified Bitumen as for TG2*
	PmB2	*Lab made highly modified bitumen with 7.5 % SBS (HiMA)*
Group 2	**Bit2**	Straight run bitumen 70/100 source Europe
	Blend1	Bit1 + asphalt reuse additive as for TC RAP TG3
	Blend2	Bit1 + REOB
	Blend3	Bit1 + paraffinic oil

Binders

Bit1 – 35/50 penetration grade bituminous binder [PG 70-22]

Bit2 – 70/100 penetration grade bituminous binder [PG 64-22]

Blend 1 – Bit1 + additive for asphalt recycling application also used for TC RAP TG3

Blend 2 – Bit1 + REOB

Blend 3 – Bit1 + paraffinic oil

Specific experimental plan

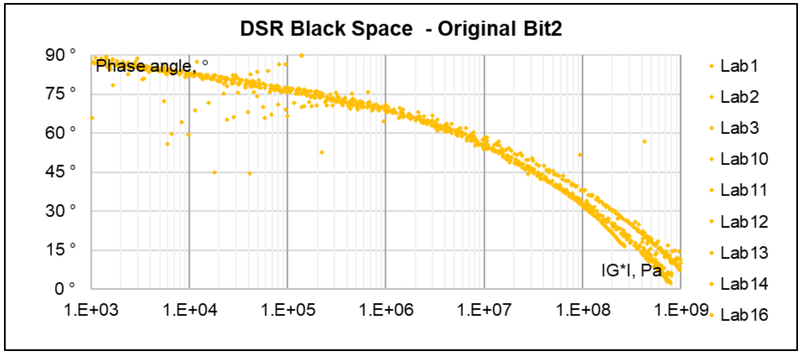
- Testing matrix
  - All labs did not perform all tests and not on all binders

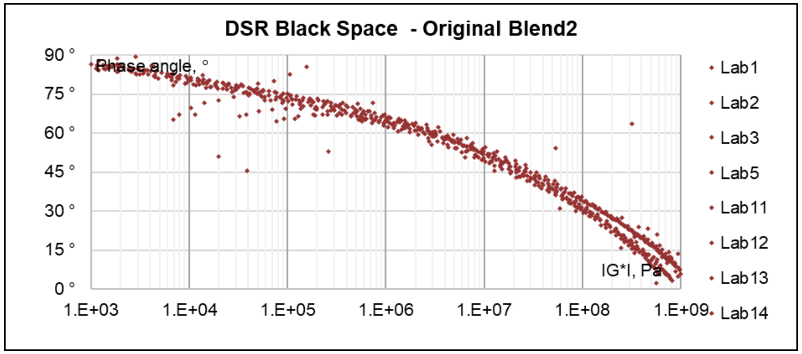
	RTFOT	PAV	Fraass	BBR	ABC	3 notch beam	4mm DSR	DSC	Penetration	Soft Point	Force ductility	Elastic recovery	**DSR**	MSCR	FTIR	**AFM**	Fluo microsco
Bit1	6	7	3	5	0	0	6	4	6	5	2	2	**11**	4	8	**1**	0
PmB1	6	7	4	6	0	0	7	4	7	6	2	2	**12**	4	8	**1**	0
PmB2	7	8	5	7	0	0	7	4	8	7	3	3	**13**	5	9	**1**	0
Bit2	9	10	3	8	1	0	6	4	7	6	3	3	**12**	7	10	**1**	0
Blend1	7	8	2	7	1	0	5	3	6	5		2	**10**	6	8	**1**	0
Blend2	8	9	2	7	1	0	6	4	6	5		2	**11**	6	9	**1**	0
Blend3	7	8	2	7	1	0	5	3	6	5		2	**10**	6	8	**1**	0

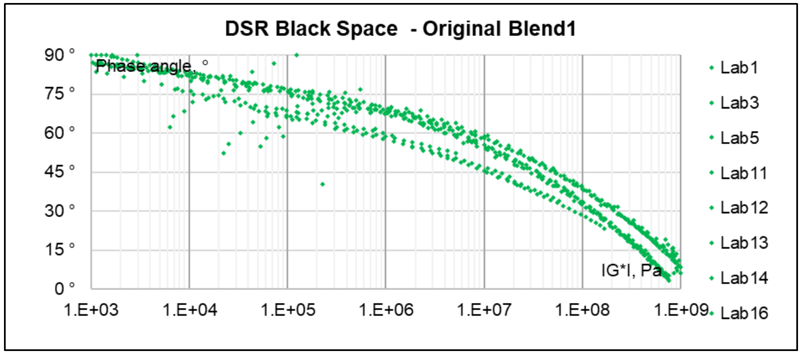
- In this scope of work-
  - Group-2 blends;
  - Characterization techniques: DSR and AFM

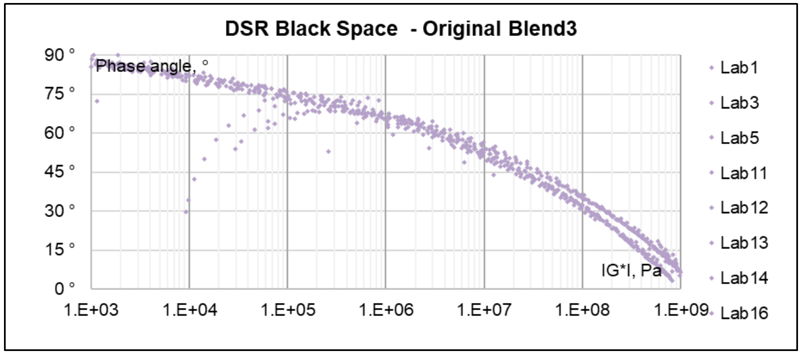
DSR results

- For each binder results are obtained from 10 to 12 labs
- Use of Black Space diagram to analyse validity of results
- Some (inevitable) variations → some data are not used later





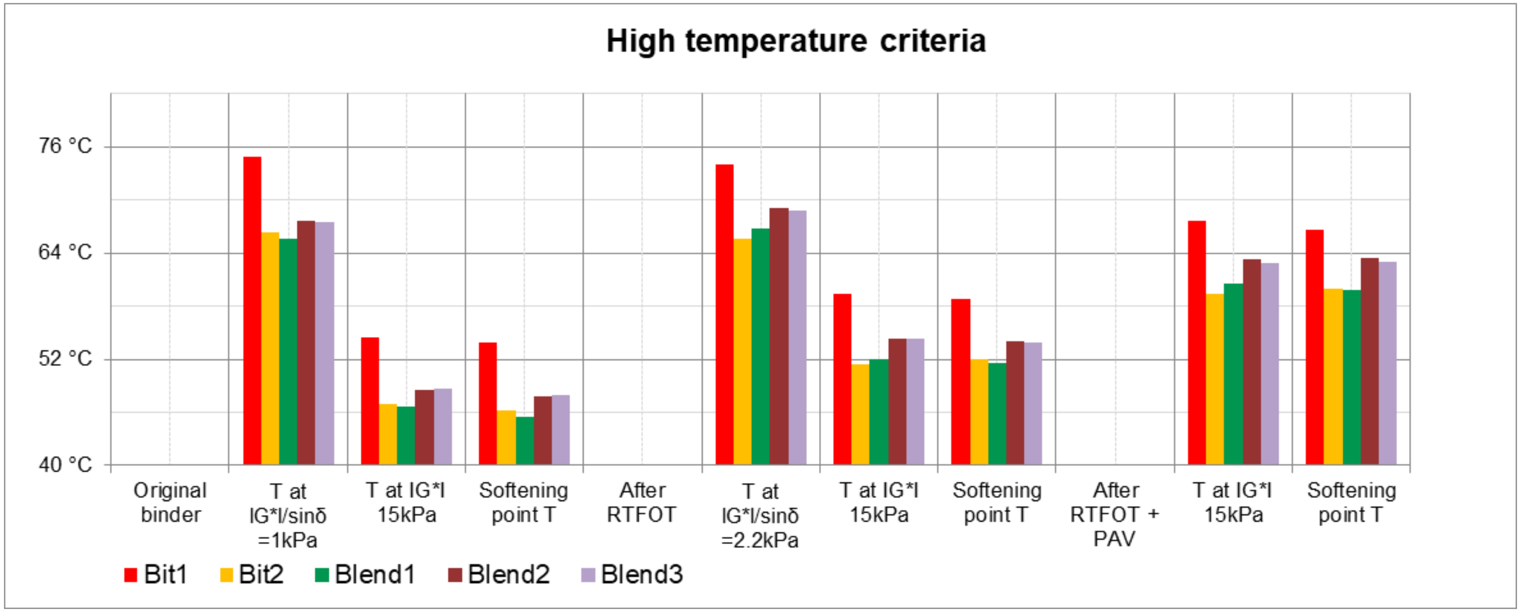




High temperature properties

Evaluation of binders using the high temperature assessment criteria

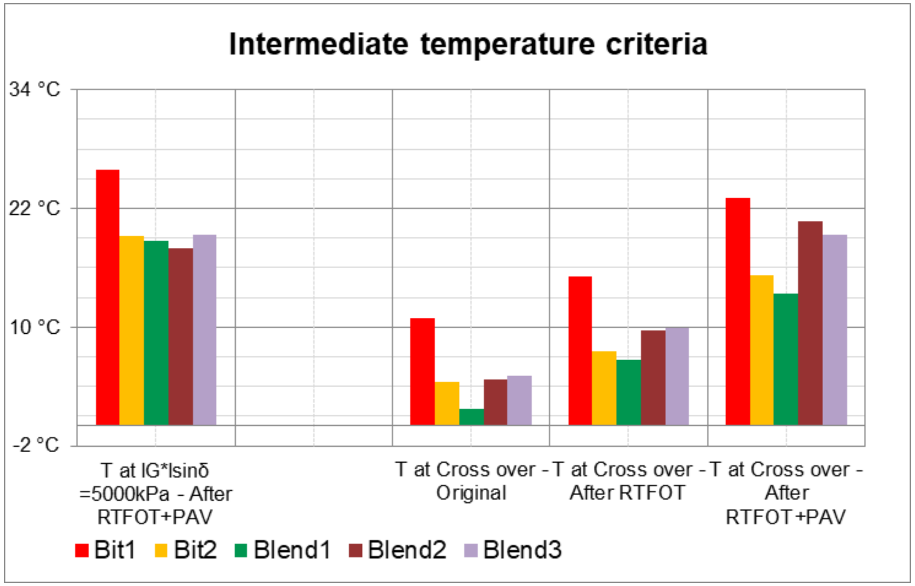
- On original, RTFOT, PAV aged binders
- Using high T criteria PG (US), softening point or equi-modulus temperatures (Europe)
- Similar values obtained independent of the blends as compared to Bit2
- Same trend is observed after aging



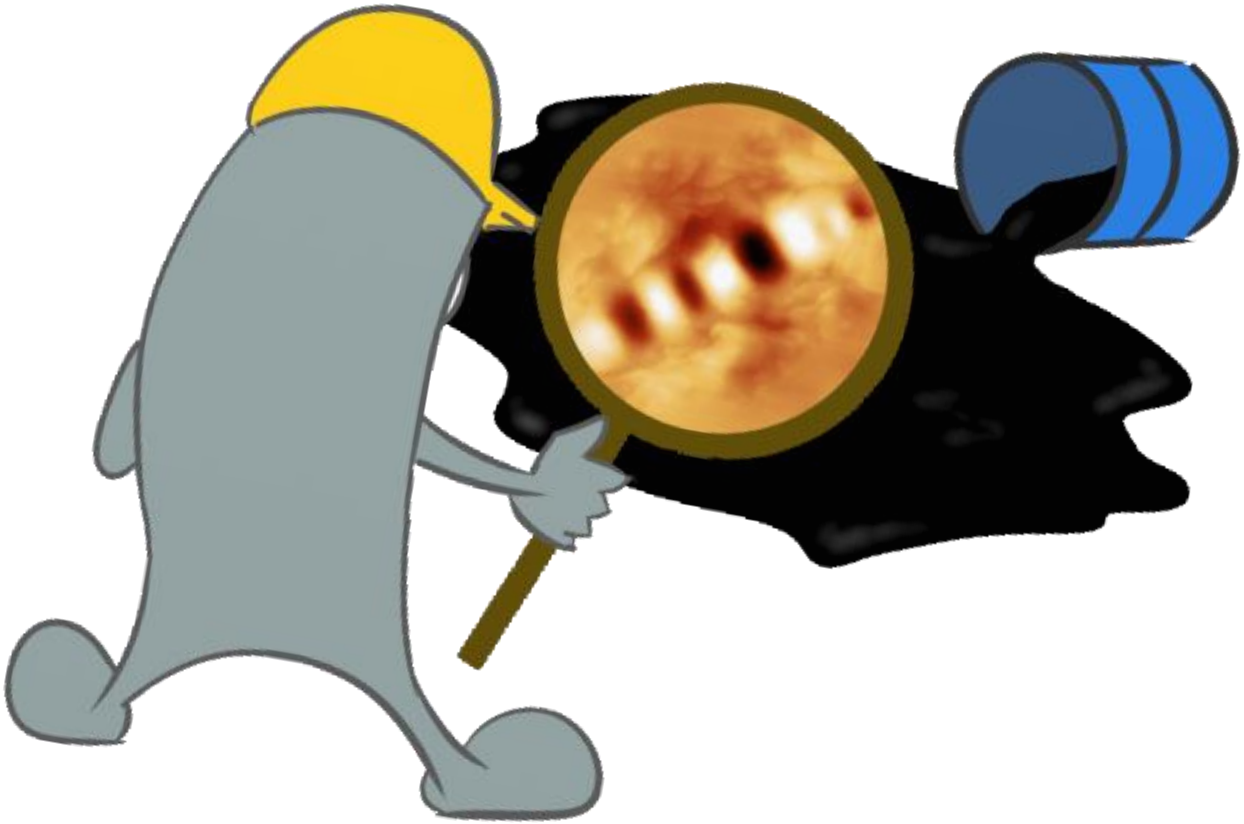
Intermediate temperature properties

- PG intermediate T after RTFOT+PAV loss modulus IG*Isinδ
- Similar values for blends as compared to Bit2
- With cross-over temperature as transition between elastic to viscous behavior
  - Bit1 displayed higher value
  - Blend 1 slightly lower value than Bit 2
  - Blend 2&3 increase over aging

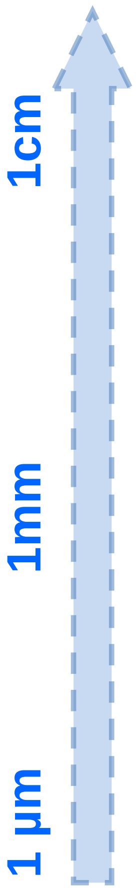
Cross-over T: an indicator to distinguish complex binder

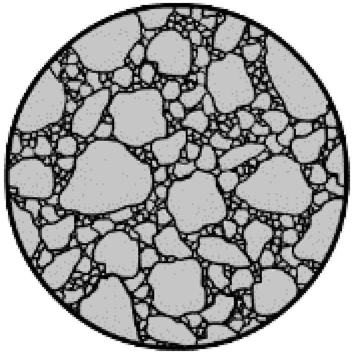


Bitumen at micro-meter length scale



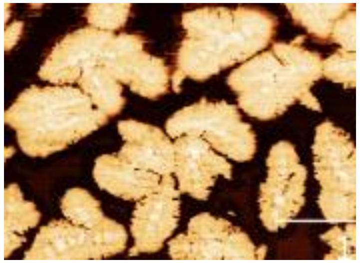
A bottom-up approach to understand the material behavior





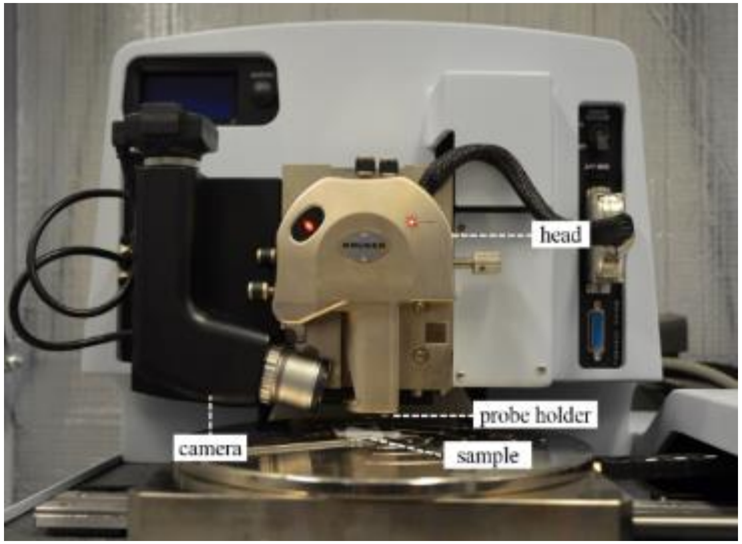
asphalt concrete

bitumen



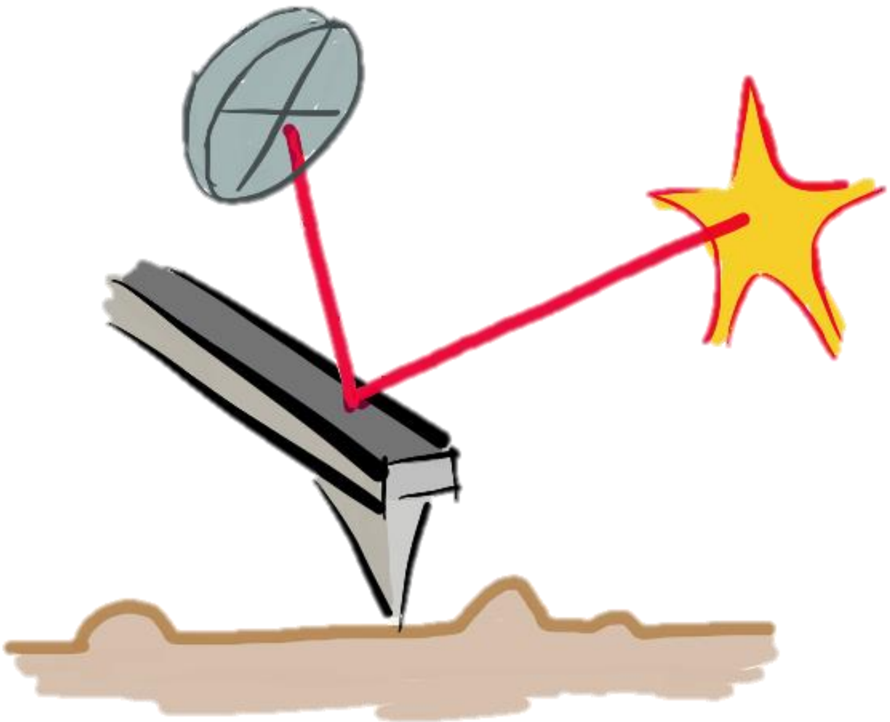
microstructure /
molecular architecture

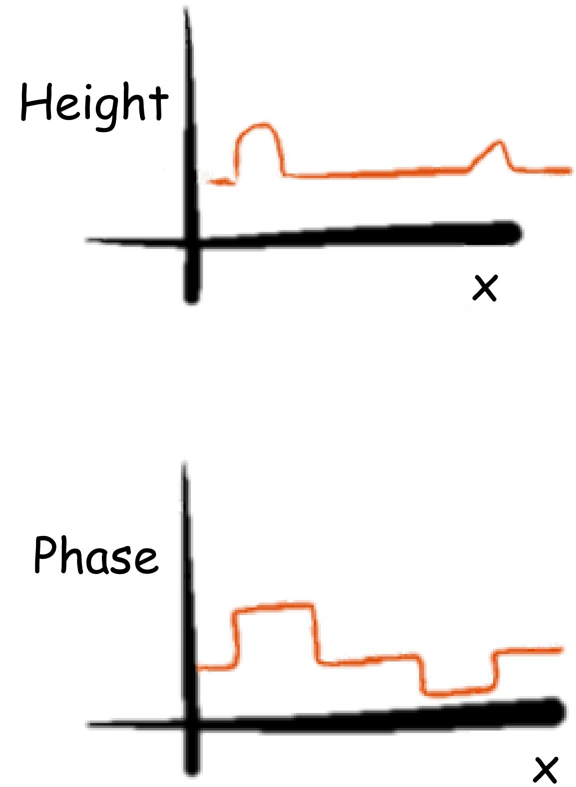
atomic force microscope



Dimension Icon

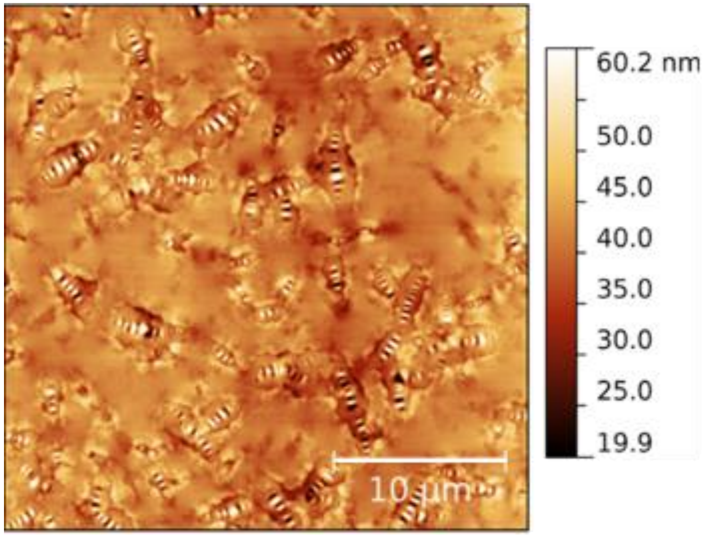
Atomic Force Microscopy: a tool to access phase-interphase properties of bitumen



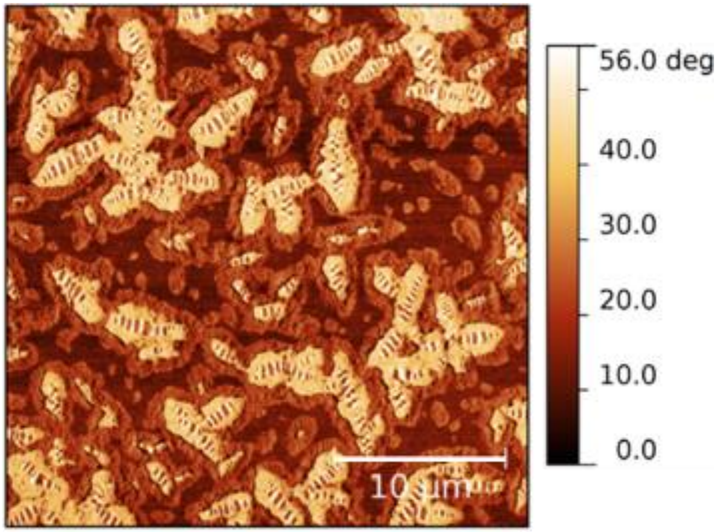


Bitumen microstructure observed by AFM

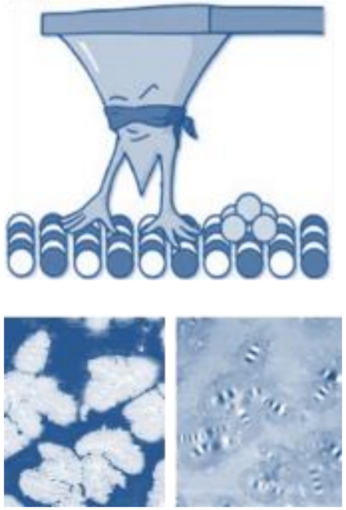
- Bitumen exhibits discontinuities at micrometer scale;
  - 2/3-phase morphology → microstructure
- Microstructure implies : stiffness discontinuities, i.e. stress inducing sites
- Microstructural details depend on:
  - molecular composition (crude origin, production parameters)
  - thermodynamic history, environmental conditions (aging)



Topography

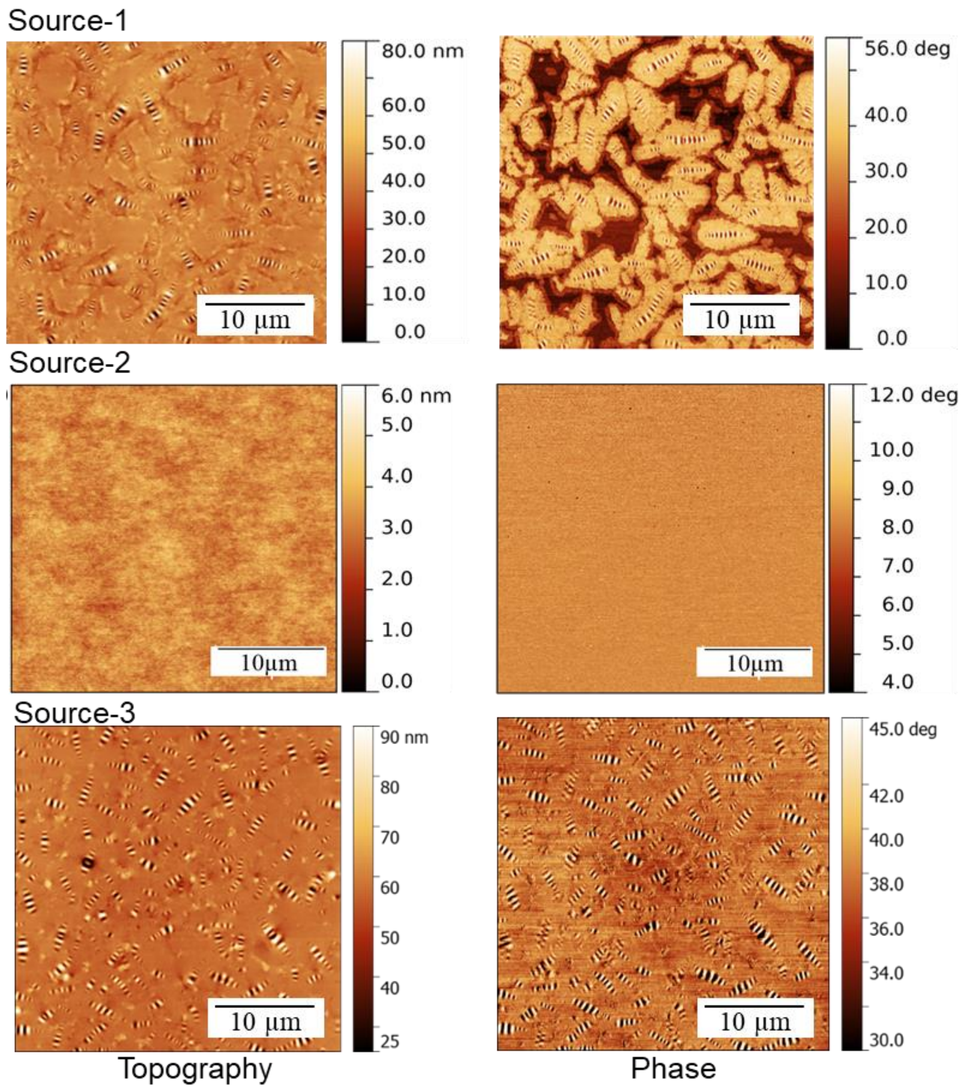


Phase



Source: PhD thesis- Sayeda Nahar: "Phase separation characteristics of bitumen and their relation to damage healing"

Phase behavior of PEN 70/100 bitumen from different sources

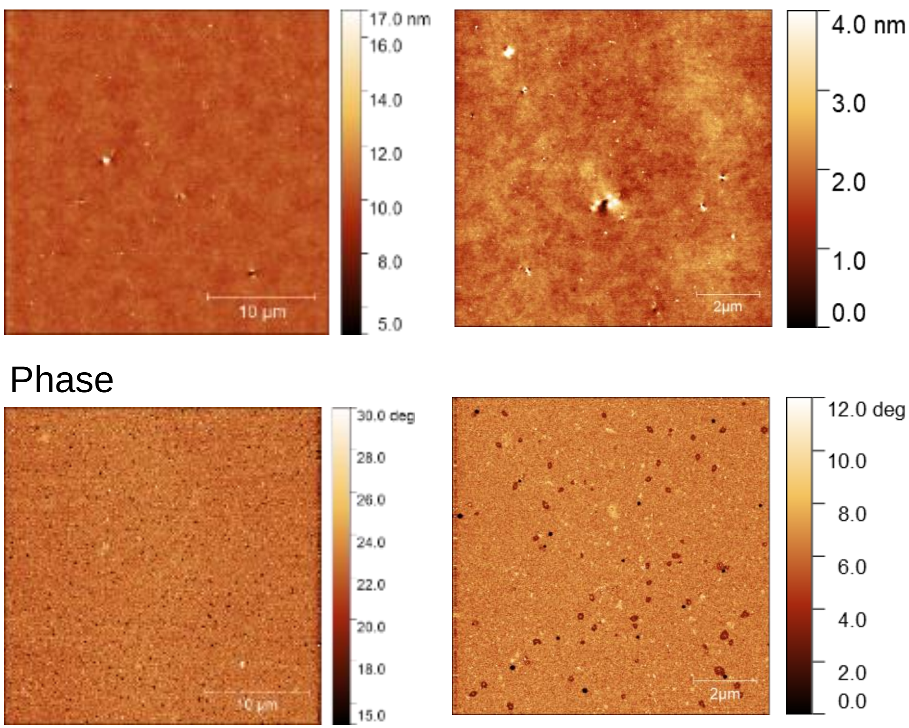


30 ×30 µm^2

Source: PhD thesis- Sayeda Nahar: “Phase separation characteristics of bitumen and their relation to damage healing”

Bitumen -1 (neat)

Topography



30 ×30 µm^2 10×10 µm^2

<u>AFM micrographs of Bitumen-1:</u>

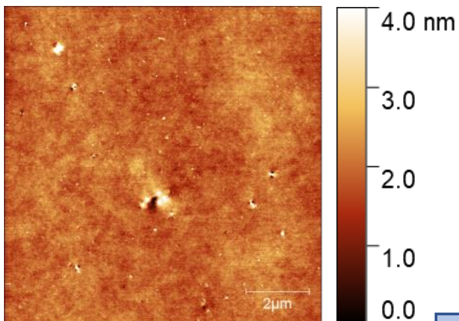
- Topography and phase images of 30×30 µm^2 and 10×10 µm^2 scans;
- Topography shows a flat morphology;
- It doesn’t show characteristic bee-structure and this morphology is commonly seen in bitumen from naphthenic origin (south-american crudes);
- Phase images confirm that the bitumen shows mostly continuous phase with softer dispersed submicron phase.

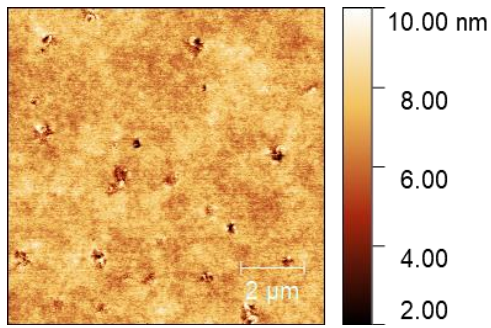
Bitumen-1 after PAV

Fresh

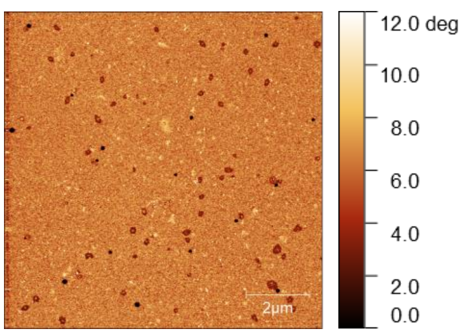
RTFOT+ PAV

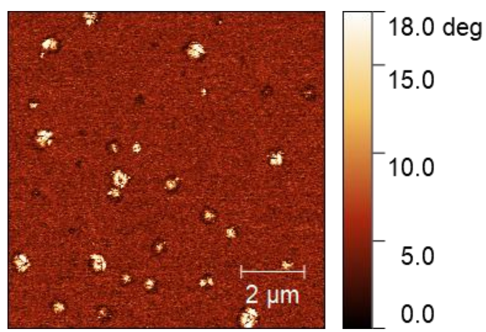
Topography





Phase





10 ×10 μm^2

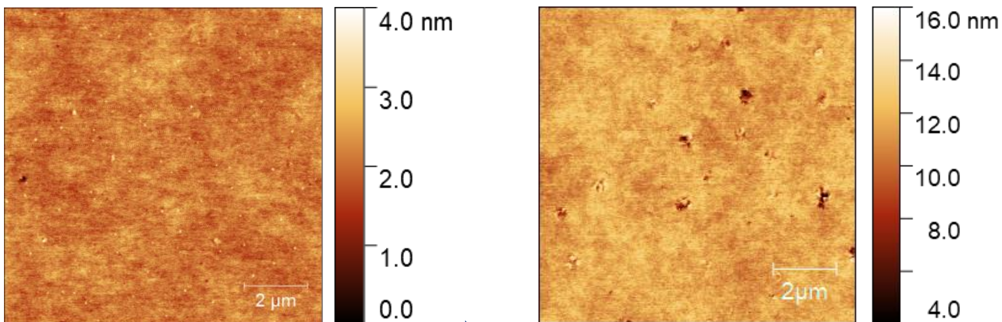
<u>AFM micrographs of Bitumen-1 after RTFOT+ PAV:</u>

- Phase images reveal development of a new phase that is higher in stiffness.

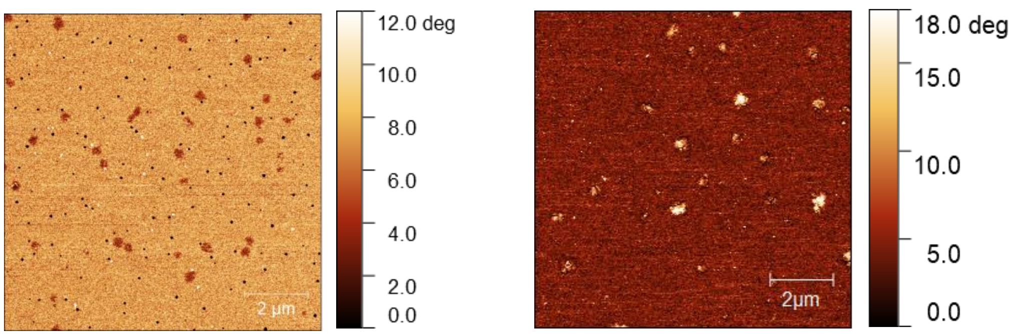
Bitumen -2

Fresh | **RTFOT+ PAV**

Topography



Phase



10×10 µm^2

<u>AFM micrographs of Bitumen-2:</u>

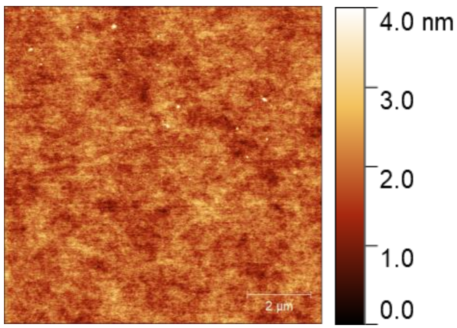
- Morphology of fresh bitumen-2 is comparable with bitumen-1 with an additional fine dispersed phase of 20-50 nm.
- After ageing, domains of higher stiffness emerges.

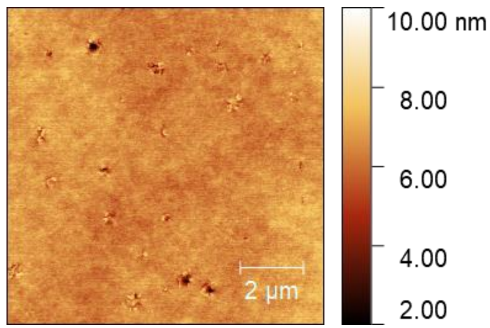
Blend-1

Fresh

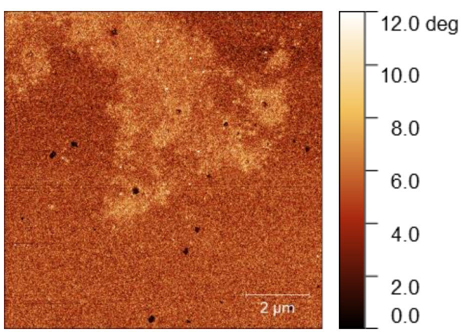
RTFOT+ PAV

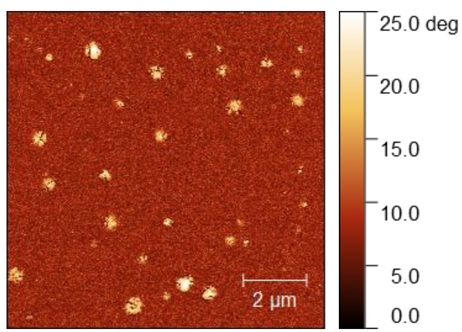
Topography





Phase





10 ×10 µm^2

<u>Micrographs of Blend-1:</u>

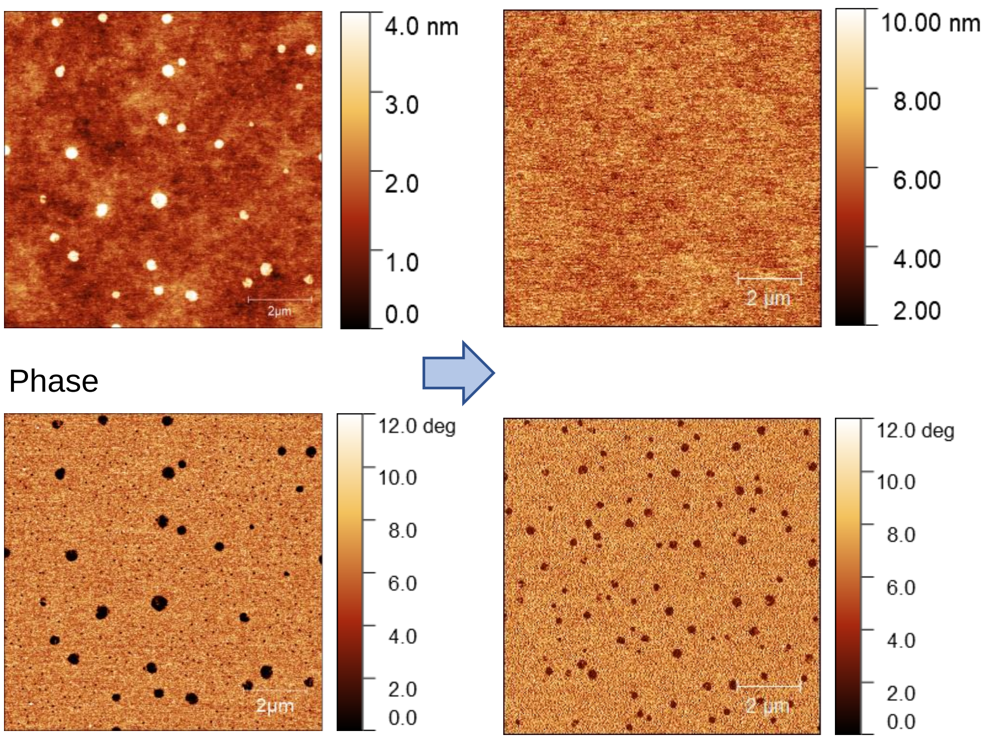
- Topography of fresh blend-1 shows flat morphology;
- Phase images mostly show the phase behavior of base bitumen-1;
- At some locations mutual immscibility/ incompatibility is apparant;
- After ageing, shows similar harder phase.

Blend-2

Fresh

RTFOT+ PAV

Topography



10 ×10 μm^2

<u>Micrographs of Blend-2:</u>

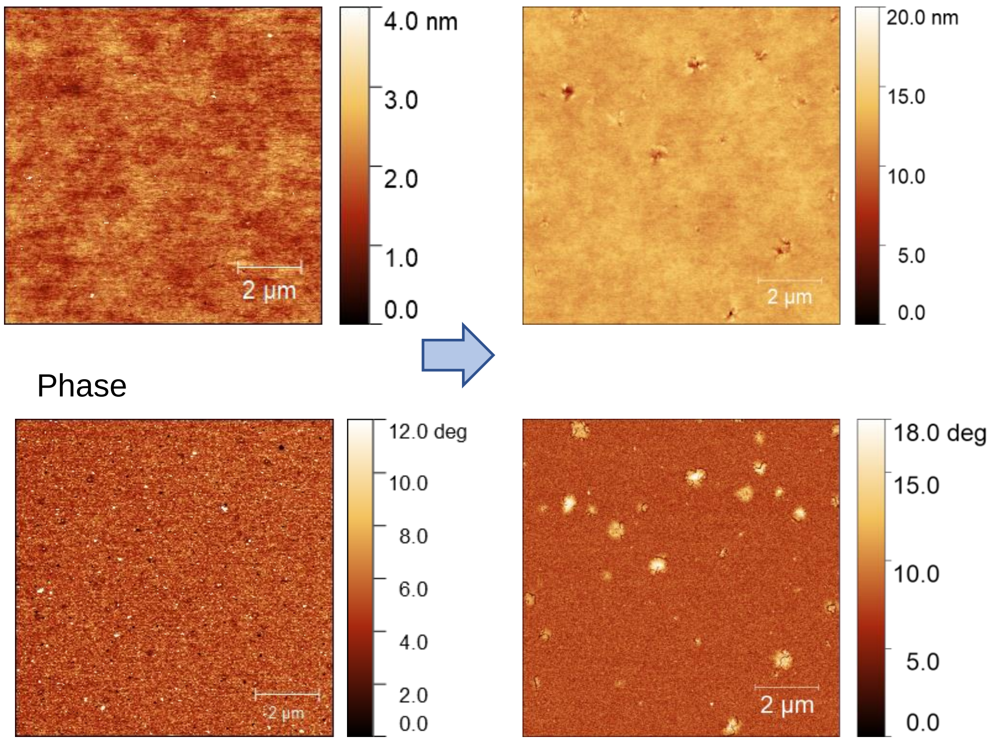
- Topography of fresh blend-2 shows submicron droplets that are protruded from the surface;
- Phase images confirm that these droplets are softer than the matrix;
- After ageing, this phase is found to be more dispersed within bitumen matrix but remains softer.

Blend-3

Fresh

RTFOT+ PAV

Topography



10 ×10 µm^2

<u>Micrographs of Blend-3:</u>

- Topography and phase images of blend 3 shows a homogeneous morphology;
- There are very fine particulates can be precipitated wax;
- Aged blend shows similar properties as bitumen-1 after ageing.

Conclusions

- Characterization of binders using DSR may not distinguish between different natures of complex asphalt binders;
- Intermediate temperature regime, cross over temperature obtained from DSR may show some distinguishing featurs between blends;
- From AFM micrographs it is revealed that all liquid additives show relatively good compatibility with the base binder;
- Blend-2 shows a distinct phase morphology– any ideas are welcome;
- AFM is a powerful tool to reveal phase-interphace characteristics of the binders at nano to micrometer length scales;
- By combining DSR and AFM we can establish the structure property relationship.