RILEM TC 231 NBM
RILEM TC CMB activities

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NTEC
Nottingham Transportation Engineering Centre

Overview

- End of RILEM TC 231 NBM
- Activities
- Conference
- Publications
- Start of RILEM TC CMB

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Round Robin

- 2 round robin activities
- 4 bitumens supplied courtesy of Nynas and Q8
- 1st activity – characterise using Differential Scanning Calorimetry
- 2nd activity – characterise using Atomic Force Microscopy

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Conference

- Multi-scale Modelling and Characterization of Infrastructure Materials
- 10-12th June 2013, KTH Royal Institute of Technology, Stockholm, Sweden
- 80 delegates from 13 countries
- 31 papers presented

Committee Publications

Challenges While Performing AFM on Bitumen

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Abstract. Using modern microscopic techniques such as atomic force microscopy (AFM) has added significant knowledge on the microstructure of bitumen. The advantages of AFM are that it requires relatively simple sample preparation and operates under ambient conditions. As the use of AFM is becoming more widespread and useful the RILEM technical committee (TC) on nano bituminous materials NBM 231 has conducted a round robin study on this method, the results with respect to reproducibility, repeatability or accuracy limits are presented elsewhere. However, the execution of good quality AFM experiments especially on bitumen is still a challenging task. Sample extraction and preparation are very crucial and attention should be paid to obtain homogenous samples with a sufficient thickness and no surface contamination. The preparation should include a high temperature treatment to provide a smooth homogenous surface. Annealing/resting of the sample has to be sufficiently long, at least 24 h under ambient temperatures to ensure the formation of a (meta)stable micro-structure. Imaging should be done using non-contact (Tapping) mode with stiff cantilevers (resonance frequency ~300 kHz) with a minimum amount of damping as possible.

1 Introduction and Motivation

Bitumen, the residue from the vacuum distillation of petroleum oil is a continuum and complex system of many different organic components such as conjugated polyaromatic and polynuclear ring systems, as well as saturated cyclic and aromatic hydrocarbons containing heteroatoms and linear or branched saturated hydrocarbons (wax) [1].

It is now widely accepted, that bitumen is not a homogenous, single phase system, but contains crystalline parts and displays also a partitioning into domains with a size of several microns to tenth of microns depending on the source of crude oil with different mechanical properties. The present knowledge on the

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Differential Scanning Calorimetry applied to bitumen: Results of the RILEM NBM TG1 Round Robin test.

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Abstract The application of Differential Scanning Calorimetry (DSC) has been proven useful in characterizing bituminous binders, distillates and crude oils. In this paper, results of the round robin test, organized by the Rilem TC 231 Nanotechnology-based Bituminous Materials (NBM) TG1 group are reported. The purpose is to investigate the repeatability and reproducibility of standard DSC measurements when applied to bituminous binders. In the full test program of the Rilem NBM group, DSC measurements are further compared to observations made in atomic force microscopy (AFM), AFM measurements are reported in a separate paper. Seven laboratories have participated in this round robin test. Four bituminous binders were investigated, containing various amounts of natural or added wax. The test program consisted of a well-defined isothermal annealing procedure, followed by a first heating and cooling scan, and afterwards followed by a second heating scan. At this stage, the data, as they were reported by the different participants, were compared. For the glass transition (Tg), mid temperatures, can be defined with a reasonable reproducibility, which improves if natural wax is not present. Regarding melting and crystallization, the shape of the melting curve is highly dependent on the thermal history of the samples. Peak temperatures of melting and crystallization phenomena were reported with a good reproducibility, while the reproducibility of melting enthalpies (or surface area's under the melting and crystallization signals) was not satisfactory. Different reasons for this and recommendations for improving the results are discussed in the paper.

Paper in Materials and Structures

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ORIGINAL ARTICLE

Laboratory investigation of bitumen based on round robin DSC and AFM tests

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Abstract In the past years a wide discussion has been held among asphalt researchers regarding the existence and interpretation of observed microstructures on bitumen surfaces. To investigate this, the RILEM technical committee on nano bituminous materials 231-NBM has conducted a round robin study combining differential scanning calorimetry (DSC) and Atomic Force Microscopy (AFM). From this, methods for performing DSC and AFM tests on bitumen samples and determination of the influence of wax on the observed phases, taking into account thermal history, sample preparation and annealing procedure, are presented and critically discussed. DSC is used to measure various properties and phenomena that indicate physical changes such as glass transition temperature (T_g) and phase transition such as melting and crystallization. In the case of existence of wax, either natural or synthetic, it can further indicate the melting point of wax, that could be used to determine wax content. The results from seven laboratories show

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New Committee TC CMB

- **C**hemo-**M**echanical Characterization of **B**ituminous Materials
- TG1: Chemo-Mechanical Coupling Identification
- TG2: Chemo-Mechanical Laboratory Characterization
- TG3: Environmental Susceptibility Investigation

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New Committee TC CMB

- **C**hemo-**M**echanical Characterization of **B**ituminous Materials
- Start up meeting at KTH 9th June 2013
- Next committee meeting planned for 3rd March 2014 in Delft
- Joint workshop on 4th March combining TC CMB and the Chemo-Mechanics Task Group of the ISAP TC on Modelling

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Workshop Agenda March 4th 2014

- 08:45 – 09:00 Workshop opening : chairs of ISAP and RILEM committees
- 09:00 – 10:30 SESSION I – RECENT DEVELOPMENTS IN BITUMEN CHEMISTRY
- 10:30 – 11:00 BREAK
- 11:00 – 12:30 SESSION II – BITUMEN MICROSTRUCTURE: CHARACTERIZATION AND INTERPRETATION
- 12:30 – 13:30 LUNCH
- 13:30 – 15:00 SESSION III – (MICRO)MECHANICS OF BITUMINOUS PHASES
- 15:00 – 15:30 BREAK
- 15:30 – 17:00 SESSION IV – STRUCTURE-PROPERTY RELATIONS: TOWARDS THE MASTIC SCALE

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