A new high-frequency torsional rheometer for bituminous binders

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ABSTRACT

When a rod is performing torsional vibrations at one of its resonance frequencies, any interaction with a viscoelastic medium will change both its resonance frequency and its damping characteristics. By measuring this change, modulus, phase angle, elastic and viscous constants of a bituminous binder can be obtained. The new High-Frequency Torsional resonance Rheometer (HFTR) developed at ETH Zürich and presented in this study is based on this principle.

In comparison to prevailing rheometers, the HFTR is inexpensive and has various technical advantages. It can be embedded in the binder or placed on its surface and is suited for laboratory and field measurements. High frequencies in the 1 to 60 kHz range allow the measurements to be unaffected by traffic induced low frequencies, which makes it ideal for field measurements. The HFTR can also be used for continuous monitoring of binder aging effects or as a portable fingerprinting tool for materials characterization in the laboratory. In addition, it allows continuous measurements during binder production.

This study focuses on the measurement technique using the HFTR for laboratory experiments involving conditioning of bitumen in a high temperature oven. Furthermore measurement uncertainties and practical examples are discussed.

RÉSUMÉ

Quand une tige entre en vibration torsionnelle à l'une de ses fréquences de résonance, toute interaction avec un matériau visco-élastique changera à la fois sa fréquence de résonance et ses caractéristiques d'amortissement. En mesurant cette variation, on peut obtenir le module, l'angle de phase, les constantes élastiques et visqueuses d'un liant bitumineux. Le nouveau Rhéomètre à résonance Torsionnelle Haute-Fréquence (HFTR en anglais) développé à l'ETH Zürich et présenté dans cette étude est basé sur ce principe.

En comparaison avec les rhéomètres actuellement prépondérants sur le marché, le HFTR est peu coûteux et a de nombreux avantages techniques. Il peut être plongé dans le liant ou placé sur sa surface et il est alors approprié aux mesures de laboratoire comme de terrain. La gamme de haute fréquences 1 à 60 kHz a pour conséquence que les mesures ne sont pas affectées par les basses fréquences liées au trafic, ce qui le rend idéal pour les mesures de terrain. Le HFTR peut aussi être utilisé pour la surveillance continue des effets de vieillissement des liants ou être utilisé comme un outil portable réalisant des empreintes destinées à la caractérisation du matériau au laboratoire. En outre, il permet des mesures continues pendant la production de liants bitumineux.

Cette étude est centrée sur les mesures techniques utilisant le HFTR pour des expériences de laboratoire impliquant le conditionnement du bitume dans des fours à haute température. De plus les incertitudes de mesure et des exemples pratiques sont discutés.

1. INTRODUCTION

This still ongoing study is part of a research co-operation

between EMPA Road Engineering/Sealing Components in Dübendorf and the Institute of Mechanical Systems of ETH in Zurich focussing on the continuous monitoring of bituminous

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Dr. Manfred N. Partl is a RILEM Fellow. He is the Chairman of RILEM TC 182-PEB 'Performance testing and evaluation of bituminous materials' and works at EMPA, Switzerland, a RILEM Titular Member. Dr. Partl was recently appointed as an expert in the RILEM Technical Activities Committee (TAC).

binders exposed to various high temperatures. Standard oven and PAV aging tests were the basis for the choice of temperatures and conditioning times. A new High-Frequency Torsional resonance Rheometer (HFTR) developed at ETH is used to characterize the material investigated by EMPA. The overall aim of the project is to determine whether there are differences in aging characteristics between the selected binders that could be detected using the HFTR.

The aging process in a pavement consists of continuous degradation and transformation of the components in bituminous binders under production and field conditions. This process is strongly related to the exposure of the binders to air and temperature and is therefore simulated by various accelerated aging laboratory tests like PAV, RTFOT and TFOT which involve exposing samples of thin binder films to heat, oxygen and pressure. After this treatment, these samples of thin films are usually mixed together in a homogenizing process and the resulting mixture is further used for material properties testing. The HFTR method eliminates the influence of this mixing of the material and allows testing of the thin layer of bitumen mechanically in a much more undisturbed way. During the aging process in the oven, the new device allows monitoring of the influence of binder properties changes in the surface zone of the film that is directly exposed to heat and oxygen.

2. THE HIGH-FREQUENCY TORSIONAL DYNAMIC RESONANCE RHEOMETER (HFTR)

2.1 System specifications and description

The dynamic rheometer (material: 18-8 CrNi steel) consists of an outer tube rigidly joined at its end E to a cylindrical inner rod through an end plate (Fig. 1). The tube is free of loading along its lateral surface and attached at one end F to a thick plate PL of large diameter in comparison to the diameter of the tube. See Table 1 for the rheometer specifications.

Since the torsional rigidity of the plate is much larger than the tube, it acts as a decoupling mass enforcing a node of the torsional vibration mode in its immediate vicinity. The other

Fig. 1 - Scheme of the high-frequency torsional resonance rheometer (HFTR).

Table 1 - HFTR specifications	
Frequency range	1kHz to 60 kHz
Stability of resonance frequency	10^{-2} Hz at 10 kHz
Sensor amplitude	Less than 1 micrometer
Temperature range	-50 to 300°C

end E of the tube where it joins the internal rod is solidly closed with an end plate. With an electromagnetic transducer, fixed at the free end F of the internal rod, the system is forced to perform high frequency vibrations of very low amplitude at one of its torsional eigenmodes. The frequency (5.4 kHz in the present case) is stabilized within 0.01 Hz with the help of a phase-locked loop fixing the phase between the applied torque and the measured angle of rotation. Further details including those of the electronic control circuitry can be found in [1].

2.2 Measurement principle

Resonance frequency, f , corresponds to a phase angle of 90° . The damping (ϵ) is proportional to the frequency difference df_α of the frequencies f_α and $f_{-\alpha}$ at two values $90^\circ \pm \alpha$ of the phase angle in the vicinity of resonance (Fig. 2):

$$df_\alpha = f_{+\alpha} - f_{-\alpha} \quad (1)$$

$$f = (f_{+\alpha} + f_{-\alpha})/2 \quad (2)$$

When the end plate is placed on the surface of the

Fig. 2 - Phase spectrum.

viscoelastic bitumen, only a boundary layer of the binder in the immediate vicinity of the contact surface participates in the motion, provided that the frequency is sufficiently high. Nonetheless, the measured resonance frequency and the damping will change in comparison to similar measurements taken in air. Shear storage and loss moduli of the bituminous binder at driving frequency follow from the changes in df_α due to interaction with the viscoelastic medium.

In Ref. [2] similar experiments were performed with the HFTR embedded in an asphaltic plug joint material. In the current experiment the rheometer is placed at a controlled depth of approximately 7.4-mm (cf. Fig. 6).

3. MATERIALS AND TEST PROTOCOL

Two different types of straight-run bitumen have been used for this experiment with different penetration grade: 50/70, 180/220. The results of penetration tests at 25°C and

ring and ball softening point tests and viscosity for each binder are presented in Table 2. Since aging and the rheological properties of bitumen are highly dependent on temperature and exposure to heat, an attempt was made to control the exposure time and temperature of the samples.

Before placing the end plate of the HTFR on the surface of the bitumen film, the values in air (indicated by subscript A) of resonance frequency f_A (phase angle 90°) and frequency differences $df_{\alpha A}$ for phase angles $90 \pm \alpha$ (in the present case $\alpha = 22.5^\circ$) at varying temperatures are measured (cf. Fig. 3). In addition, these air values were periodically re-checked in order to verify that the rheometer is functioning properly. Temperature has a significant effect on the internal readings of the rheometer, therefore in order to record the changes in the binder only, the air values at relevant temperatures (in this case 90°C , 110°C and 135°C) have to be subtracted. At these test temperatures the scatter

Table 2 - Unaged penetration, softening point and viscosity data

Binder	Needle Penetration at 25°C [0.1 mm]	Ring and ball softening point [$^\circ\text{C}$]	Viscosity at 60°C [Pa.s.]
50/70	74	48.8	415.0
180/220	168	43.2	50.1

Fig. 3 - HTFR readings f_A , $df_{\alpha A}$ in air at various temperatures.

of df is comparatively small.

Subsequently, the values on bitumen (indicated by subscript B) of resonance frequency f_B and frequency differences $df_{\alpha B}$ are measured continuously. In the present case 7 measurements were taken every minute resulting in 420 measurements per hour.

References [3] and [4] were used as a guideline for sample preparation and testing. Test samples were placed on a TFOT pan and placed on a TFOT pan holder. The rheometer was supported by a specially designed support system on the TFOT pan holder. Figs. 4 and 5 show the rheometer during a test.

A 50 g sample of the bitumen being tested was cut out of the bitumen in the container and placed on a TFOT testing pan. The binder was warmed for one-half hour in the oven at test temperature in order to flatten in the pan. Thereafter the pan was placed on one of the TFOT shelves and the rheometer was lowered onto the

Fig. 4 - HTFR during a test.

Fig. 5 - Tip of HTFR rod during a test.

bitumen. The distance between the end of the HTFR and the bottom of the pan (h in Fig. 6) was controlled with a two-gage. This is important since the amount of penetration depth of the tip of the HTFR into the material influences the readings of the rheometer. Using the Boundary Layer Theory [5], and the solution for a rotating disk the boundary layer for each test temperature was estimated (Table 3). It was assumed that the

Table 3 - Result of boundary layer thickness estimate with η = dynamic viscosity; ρ = density; f = frequency; ω = angular frequency = $2\pi f$; δ_{90} = Thickness of boundary layer with fluid at 60°C ; δ_{110} = Thickness of boundary layer with fluid at 110°C ; δ_{135} = Thickness of boundary layer with fluid at 135°C

Material	η 90°C [Pas]	η 110°C [Pas]	η 135°C [Pas]	ρ [g/cm ³]	ω [Hz]	δ_{90} [mm]	δ_{110} [mm]	δ_{135} [mm]
50/70	22	3	0.28	1.028	31416	1.2	0.4	0.1
180/220	5	0.2	0.18	1.023	31416	0.6	0.1	0.1

Fig. 6 - Scheme of the HTFR dipped into the bitumen on the TFOT plate.

dynamic viscosity remains constant during the test and the rheometer tip radius is much larger than the boundary layer. As a result of the estimated boundary layer thickness calculated and shown in Table 3, a distance of $h=2\text{mm}$ from the bottom of the pan was determined to give reproducible results minimizing any interference resulting from the bottom of the pan (Fig. 6). Test temperatures of 90°C , 110°C and 135°C were selected in order to cover at least the range specified in [3].

4. DISCUSSION OF RESULTS

Aging of bituminous mixtures in the field occurs over time due to exposure to air and climatic loading, specifically heat. In [4] aging of a polymer modified asphaltic plug joint was monitored in situ for three years. The results indicated that at working temperatures (-10°C to 50°C) both df and f increased.

Figs. 7 and 8 show the fluctuations of resonance frequency (phase angle of 90°) f_B and $df_{\alpha B}$ while the HTFR is dipped into the bitumen at a constant temperature of 90°C , 110°C and 135°C for approximately a 24 hour periods. These conditioning temperatures and time showed that the resonance frequency has a tendency to increase however this increase is small compared to the stability of resonance frequency (Table 1), while $df_{\alpha B}$, after adjustment for air values (Fig. 3), increase about 4.6% (90°C) and 4.8% (110°C) for Bitumen 50/70 and 8.2% (110°C) and 26% (135°C) for Bitumen 180/220. However in absolute terms the rate of increase for various materials and temperatures is similar as seen in Fig. 7. It was established in [4] that the measured values of resonance frequency, f_B , are proportional to the elastic part of the complex modulus. It was also explained that the frequency

Fig. 7 - Changes in frequency difference $df_{\alpha B}$ for Bitumens 50/70 and 180/220 conditioned for 24 hours.

Fig. 8 - Change in resonance frequency f_B for Bitumens 50/70 and 180/220 conditioned for 24 hours.

difference, $df_{\alpha B}$, is proportional to the viscous part of the complex modulus. Therefore, Figs. 7 and 8 suggest that during a 24 hour conditioning time of a 50/70 and 180/220 straight-run bitumen the viscous part has significantly increased whereas the elastic part of the complex modulus has a tendency to increase a small amount and the rate of increase of the elastic component appears to become smaller during the heating process.

Furthermore, measurement uncertainties arise in this study since the penetration depth of the rheometer tip into the material cannot be precisely controlled.

5. CONCLUSIONS AND FURTHER WORK

This ongoing study has pointed out that as a result of high temperature conditioning the top layer of bituminous binders go through a significant increase in damping characteristics. Elastic characteristics quantified by resonance frequency increase however not significantly and conditioning time plays a direct role in this change.

The HTFR appears to be a promising tool to characterize the mechanical effects of aging, which occurs in the top layer up to an unknown depth, without the need for mixing and homogenizing.

Similar studies using a Pressurized Aging Vessel (PAV), as well as low temperature oven tests are planned. Furthermore the effects of increasing/decreasing the distance between the tip of rheometer and bottom of pan will be studied. Polymer modified binders will also be studied.

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