

## Rheological properties of mastic mortar depending on petrographic origin of filler particles

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### ABSTRACT

Mastic mortar, i.e. a mixture of bitumen and very fine filler particles (i.e. aggregate fine particles smaller than 0.063 mm), can significantly co-influence not only rheological properties of the processed asphalt mixture, but also the visco-elastic properties of compacted asphalt layers including resistance against crack formation. Nevertheless, the number of scientific studies dedicated to this topic in detail, is very limited. The aim of this work is to determine the basic rheological properties of these mastic materials using a dynamic shear rheometer, in particular concentrating on the petrographic origin of the filler particles. Fourteen filler types were selected and mixed with paving bitumen 70/100 in various ratios. Petrographic composition of the individual fillers was determined using powder X-ray diffraction analysis. Attention was given especially to the effect of the presence of mica on the filler and mastic properties. Filler properties were analyzed using the voids of dry compacted filler test so-called "Rigden voids" and the delta ring and ball test. Mastic mortars prepared in a laboratory were subjected to multiple stress creep and recovery test and oscillation measured at 60 °C in a dynamic shear rheometer. Results show that using dynamic shear rheometer to determine the properties of these materials is advantageous. Petrographic origin of the filler to a great extent affects the behavior and properties of the mastic mortar, especially its stiffening effect. Limestone filler stiffened the bituminous binder the least and reference materials, containing high ratio of mica, stiffened the binder the most.

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## 1. Introduction

Properties of the input materials, ingredients and their ratios have a major impact on the mechanical properties of the asphalt mixtures used for road construction (Ma et al., 2017; Poulikakos et al., 2014; Garcia et al., 2016; Androjić et al., 2013; Brown & Needham, 2000; Korayem et al., 2018; Fakhri et al., 2018, 2020; Ziari et al., 2020; Aliha et al. 2018, 2019a,b; Motamedi et al., 2020; Pour et al., 2018; Hagos, 2008; Liao et al., 2013; Bommavaram et al., 2009; Qiu et al., 2011; Wu et al., 2009; Arabani & Tahami, 2017; Ameri et al., 2016; Zarei et al., 2020; Shaker et al., 2020). When preparing these mixtures, the bitumen is mixed with aggregate fractions, which are coated by the bitumen. An important part of the aggregate is filler, which acts primarily to stiffen the binder. Filler is characterized by predominant content of fine particles of with size less than 0.063 mm (EN 2002). While passing through a drying drum of a batch asphalt mixing plant, these particles are mostly separated from the original aggregate and in a form of reclaimed dust returned separately to the asphalt mixture. The filler is very fine-grained and has

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the largest specific surface area of all the aggregate fractions, so it binds large amount of bituminous binder giving rise to mastic mortar (mixture of filler and bituminous binder) in the asphalt mixture. This material greatly impacts the properties of the asphalt mixtures.

Up until now, the binder component of asphalt mixtures was usually studied separately and its interactions with filler particles often neglected. Major reason for this was the lack of adequate methods for studying these composite materials. The rise of modern functional tests allows studying and describing the properties of the mastics and analyzing their effects on the properties of asphalt mixtures Micaelo et al. (2017). Some quarries produce aggregate, which negatively affects the rheological properties of the asphalt mixture during the process of compaction (rolling) of the laid asphalt mixture (Leichmann and Paták 2009). Waves form ahead of the rolling machine. Very often, cracks start to form parallel to the rolling machine, which cannot be fully removed by additional compaction. It was empirically determined that the source of this undesirable rheological behavior are often the finest fractions of the aggregate and the reclaimed dust. Problems are often thought to be caused by the presence of phyllosilicate particles, which are released during the process of crushed aggregate manufacturing from the parent rock and transferred to the filleric ratios (Micaelo et al.; 2017, Hakim and Said 2003; Miskovsky 2004; Kondelchuk 2008). Phyllosilicates are a group of materials characterized by very good basal separation. The most common representatives of phyllosilicates are mica materials (especially muscovite and biotite). They are relatively soft, but they have a surprisingly high bulk density. The aim of this paper was to present a topic, which is currently not well-studied in available literature for the description of mastic mortar properties. Another aim was to outline the relationship between the aggregate petrography and the results of selected laboratory tests. This also demonstrates the advantages of using a dynamic shear rheometer (DSR) for mastic properties analysis. The aim was to find such laboratory tests, which would allow determining problematic behavior of some types of fine aggregate during the processing of asphalt mixtures from them.

## 2. Experiments and Methods

The voids of dry compacted filler test was used to evaluate the filler properties – the so-called Rigden voids, performed based on (EN 1097-4; 2008) and the delta ring and ball test described in (EN 13179-1; 2014). A DSR was used to study the rheological properties of mastic mortars, which meant measuring the complex shear moduli  $G^*$  and phase angles  $\delta$  according to (EN 14770; 2012). In addition the Multiple Stress Creep and Recovery Test (MSCR test) (EN 16659, 2016) was performed, which evaluates the creep and elastic response of the sample. The result is the value of non-recoverable creep compliance "Jnr", which also takes into account applied stress and induced strain (therefore a lower value of "Jnr" means higher stiffness and lower susceptibility to deformations) and percent recovery %R, which expresses the extent of elastic response. Oscillation and creep tests were performed at a temperature of 60 °C. In total 14 fillers gathered from different quarries were used in the tests, some of which showed non-standard behavior during compaction of asphalt mixture layer containing this particular aggregate. In order to perform a detailed petrographic identification, the composition of these rocks was determined using powder X-ray diffraction analysis. Three reference mica filler samples muscovite and biotite were also included. Composition of the reference mica materials is given in Table 1. It is apparent that the aggregate sample labeled "Biotite 1" has a higher concentration of biotite than the sample "Biotite 2".

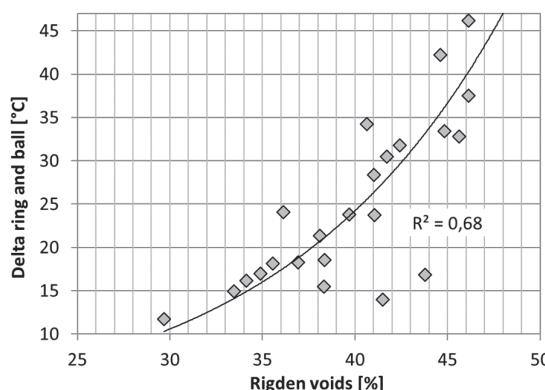
**Table 1.** Petrographic composition of reference mica minerals

Mineral [%]	Biotite	Muscovite	Chlorite	Quartz	Plagioclase	Alkali feldspar	Amphibolite
Muscovite	-	100	-	-	-	-	-
Biotite 1	60.8	-	14.9	7.2	13.4	0.9	2.7
Biotite 2	53.0	-	10.6	16.6	14.6	0.6	4.6

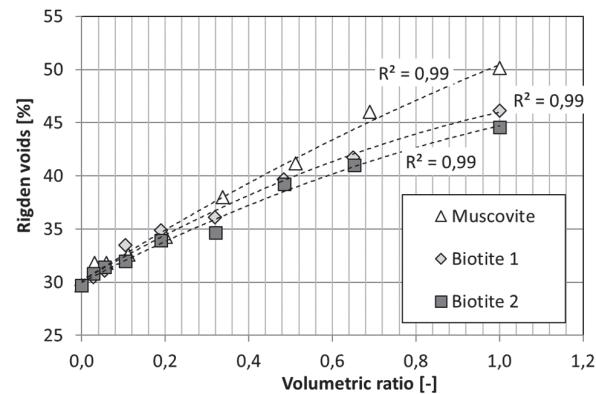
A non-modified paving bitumen with penetration gradation 70/100 was used as the binder in the mastic mortars. Their laboratory preparation was performed in accordance with the EN 13179-1 standard (EN 13179-1; 2014). The following concentration thresholds were used in concentration sets: 6.25 %, 12.5 %, 25.0 %, 37.5 % and 50.0 % of filler in paving bitumen (i.e. the volumetric amount of filler in the total volume of the mastic sample).

### 3. Results

Rigden voids according to (EN 1097-4; 2008) and the delta ring and ball (EN 13179-1; 2014) test turned out to be the most suitable for the description of properties and distinguishing the individual filler materials. Results from these methods show that different filler samples in their specific volumetric concentration differ – as expected – in their ability to stiffen the bituminous binder. Higher Rigden voids and higher value of the delta ring and ball test usually result in higher stiffening effect, which can be seen in Fig. 1.



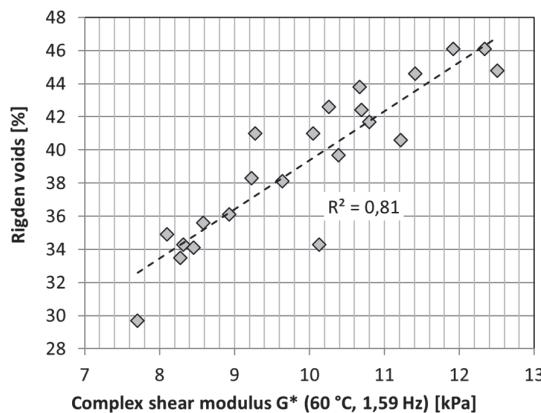
**Fig. 1.** Relationship between Rigden voids and values of delta ring and ball of the filler of fraction 0.000-0.063 mm



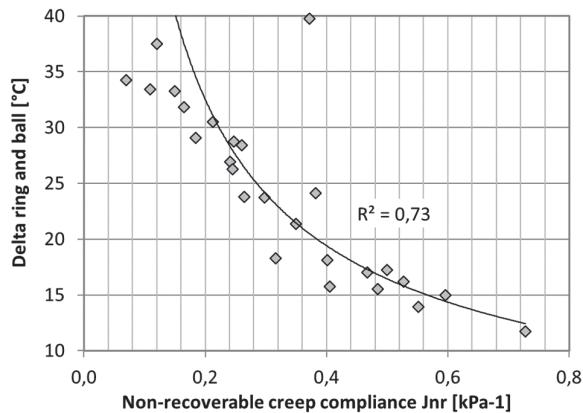
**Fig. 2.** Concentration sets – relationship between Rigden voids and the content of reference mica material in limestone filler of fraction 0.000-0.063 mm

The default filler reference materials “Muscovite”, “Biotite 1” and “Biotite 2” with high ratio of mica (see Table 1) were prepared by mixing with limestone filler which gave result to three sets of fillers with exactly defined ratio of reference material and limestone. Fig. 2 shows the relationship between Rigden voids and content of reference mica filler in the limestone filler.

However, in case of commonly available rocks, the value of Rigden voids usually rather decreases with increasing amount of mica. The relationship is therefore surprisingly the complete opposite compared to the reference concentration sets, which were prepared by mixing mica minerals with limestone filler in a laboratory. Similar results can be observed when studying the stiffening properties of the fillers using the delta ring and ball test. Basic parameters of oscillation and creep tests determined using DSR at a temperature of 60 °C correlate with values of parameters obtained by tests performed using the actual filler, which is obvious in Figs. 3 and 4. This suggests that for basic operational control of properties of fine aggregate it is possible to use the currently used tests (Rigden voids of dry compacted filler and delta ring and ball test).



**Fig. 3.** Relationship between Rigden voids and complex shear modulus for filler samples of 0.000-0.063 mm fraction with various petrographic origins



**Fig. 4.** Relationship between delta ring and ball and  $J_{nr}$  for shear stress 6.4 kPa for filler samples of 0.000-0.063 mm fraction with various petrographic origins

Mastics containing fillers with a high ratio of mica possess a better ability to recover (elasticity) especially at low shear stress (0.1 kPa). The parameter of average percent recovery %R is significantly higher for mica materials or aggregates, which show non-standard behavior. Lowest values of %R were usually observed for mastics containing limestone aggregate and %R values of mastics with standard aggregate were between these two extremes. This behavior, however, does not apply in general to all fillers with various mica content (probably because of the content of other minerals). Comparison of test results from DSR leads to similar conclusions as the results of empirical tests of filler materials. Petrographic origin using approximately similar grain size distribution determines the stiffening effect of the filler. Limestone filler is on one end of the scale (stiffens the bituminous binder the least) and reference material "Biotite 1" with high content of phyllosilicates on the other end. Stiffening ratio between these two materials in a classic delta ring and ball test is 3.6, for complex shear modulus 6.76 and for MSCR test parameters 5.59. Other fillers of various petrographic origins are somewhere between these two extremes. This means that the individual sensitivities and evaluating scales of test methods can lead to different results. One possible explanation could be various conditions during the individual tests. For oscillation tests performed in DSR the strain is within the visco-elastic region, while for other tests such assumption does not hold.

#### 4. Conclusion

The paper has focused on the topic of evaluating properties of mastic materials (bituminous binder mixed with mineral filler), a topic, which has not yet been thoroughly studied in scientific literature. Properties of mastics created by mixing paving bitumen with various filler types are evaluated using a dynamic shear rheometer. Properties of these fillers are analyzed using Rigden voids test (voids of dry compacted filler) and the delta ring and ball test with the aim of associating these results with petrographic composition of the individual fillers, which was determined using powder X-ray diffraction analysis. Attention was given especially to the effects of the presence of mica on the filler and mastics properties. The results have shown that using a DSR is quite advantageous for evaluating the properties of these materials. Petrographic origin of the fillers to a great extent affected the behavior and properties of the mastic mortar, in particular its stiffening effect. Limestone filler stiffened the bituminous binder the least and reference materials containing high ratios of mica stiffened the bituminous binder the most. Behavior of the aggregate gathered from commercial quarries, however, is more complex than the behavior of fillers prepared by mixing mica with limestone filler in a laboratory.

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