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ИЗУЧЕНИЕ ВЛИЯНИЯ НАНОРАЗМЕРНЫХ ДОБАВОК НА МЕХАНИЧЕСКОЕ ПОВЕДЕНИЕ ЦЕМЕНТНЫХ БЛОКОВ

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STUDY OF THE INFLUENCE OF NANO-SIZE ADDITIVES ON THE MECHANICAL BEHAVIOUR OF CEMENT STONE

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Introduction. The purpose of this study was to investigate the potential effects of adding carbon nanomaterials (CNM) to a cement paste using the experimental micromechanical equipment available at the Institute for Mechanics of Materials and Structures. Two samples were provided by Belarusian partners. One was control sample without CNM (sample named “K”), and the other sample was modified using CNM (sample named “O”). The CNM used in the experiments was obtained in the plasma of a high-voltage atmospheric-pressure discharge with the use of a methane – air mixture [1]. The CNM were added to the cement paste using preliminary prepared nano-paste consisting of nanotubes and a plasticizer. The water cement ratios and the amount of CNM are given in Table 1.

Table 1

Samples investigated in this study: water cement ratio and amount of carbon nanotubes

Sample	Water cement ratio	Amount of nanotubes, %
K (control sample)	0.29	0
O (modified sample)	0.22	0.05

The following equipment is used for characterization of the microstructure and micromechanical properties: X-ray Microtomography (Micro-CT); Atomic force microscopy (AFM); Nanoindentation (NI); Ultrasonic device (US).

The nanoindentation equipment and the ultrasonic device, on the other hand, are suitable test methods for investigation of cement stone. In the following, both methods are described and the corresponding test results are presented. At the end of the report, the test results are summarized and discussed with respect to the influence of the addition of CNM on the mechanical behaviour of cement stone.

Nanoindentation tests. During nanoindentation (NI), a tip with defined shape penetrates the sample surface, and in doing so the applied load and penetration depth are recorded continuously. Commonly each indent consists of a loading, holding (constant load), and unloading phase. Modulus E_r is calculated using the unloading part of the force-displacement curve.

One cement sample of about 2×2×2 cm, which contained 0.05 % mass fraction of CNM (abbreviated “O”), was tested, and one sample without addition (“K”).

The samples were glued onto steel plates. The sample surface was grinded and polished in order to obtain a sample roughness suitable for NI tests.

To ensure the basic applicability of this test method and to test if it is possible to detect the microstructure of the material in this way, at first on both samples a 10×10-grid of test points was exerted. This grid indentation should elucidate the spatial distribution and the corresponding elastic properties of the material phases at the micrometer scale. Figure 1 shows grid plots obtained by plotting the Young’s module E for all test points in a 2D-graphic. While many of the results are located in a plateau-like area at 20–30 GPa, several local maxima stick out, where Young’s modulus may increase up to 150 GPa. The stiffness differences can be related to the different material phases of cement (e.g., CSH and ettringite) showing the variation of mechanical properties between the phases. The (arithmetic) means of E for these tests were 30.9 (sample K) and 28.6 GPa (O). Figure 2 shows the statistical analysis of the results of E for this test series.

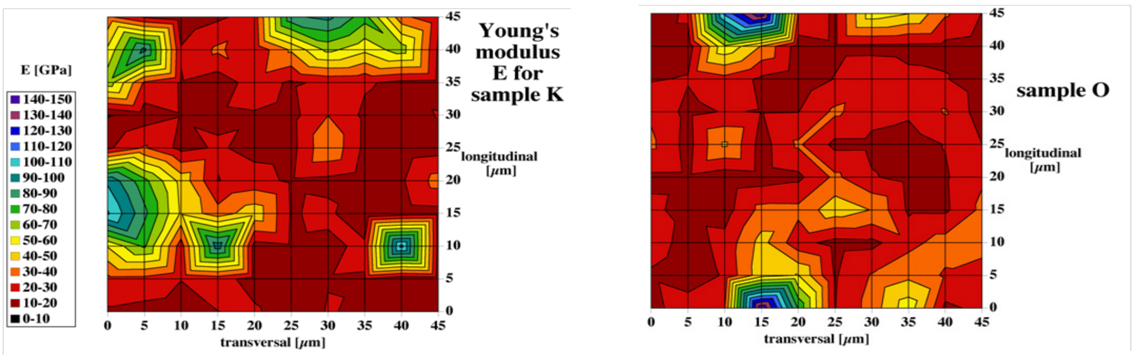
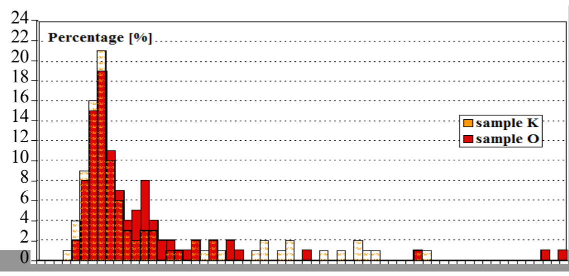


Fig. 1. Results for the 10×10-grid for “K” and “O” specimens



2,5 10,0 17,5 25,0 32,5 40,0 47,5 55,0 62,5 70,0 77,5 85,0 92,5 100,0 107,5 115,0 122,5 130,0 137,5 145,0 152,5

Interval limit [GPa]

Fig. 2. Statistical analysis of Young's moduli

There is a distinct maximum at 20–25 GPa for both samples and outliers up to 160 GPa. Also a local maximum at 30–35 GPa is observed for the sample with CNM.

Due to the high variation of the results another 20×20-grid was exerted on both samples (with otherwise unchanged parameters). As seen in Figure 3, this resulted in similar results as in Figure 1, although the level of the plateau was significantly higher for the K-sample. This might be due to the addition of CNM, but it appears doubtful because of the low weight proportion of the additive, especially since the outliers are also found in the additive-free sample.

To detect an effect of the additive, a higher penetration depth might be helpful, because by increasing the tested volume it should be possible to increase the probability to hit a nano-particle. Therefore on both samples a third test series with maximum force at 10 mN was carried out.

One can see the effects of small-scale variation (demonstrated by the standard deviations within the 3×3 grids, indicated by the error bars) as well as the large-scale variation (shown by the differences between the four test areas). The mean values for *E* over all four test areas were 43.4 (K) and 37.3 GPa (O). The average penetration depth was 569 nm, compared with 216 nm at 800 μN.

Ultrasonic tests. In this study, the ultrasonic pulse-transmission technique was employed for determination of stiffness properties of the cement stone samples [2]. Two transducers are used; one sending a signal into the specimen and another one receiving the sent signal at the opposite side of the specimen (see 4). An auxiliary testing device (see Fig) made of aluminium and steel was used to hold the two transducers in a parallel position and to apply a uniform pressure.

In total, 3 cubic specimens of the control sample, K-1 to K-3, and the sample modified using CNM, O-1 to O-3, were prepared. The dimensions of the samples were measured using a slide gauge. Special attention was paid on getting parallel side faces.

The ultrasonic tests were performed by using two different transducers with a nominal frequency of 100 kHz and 500 kHz.

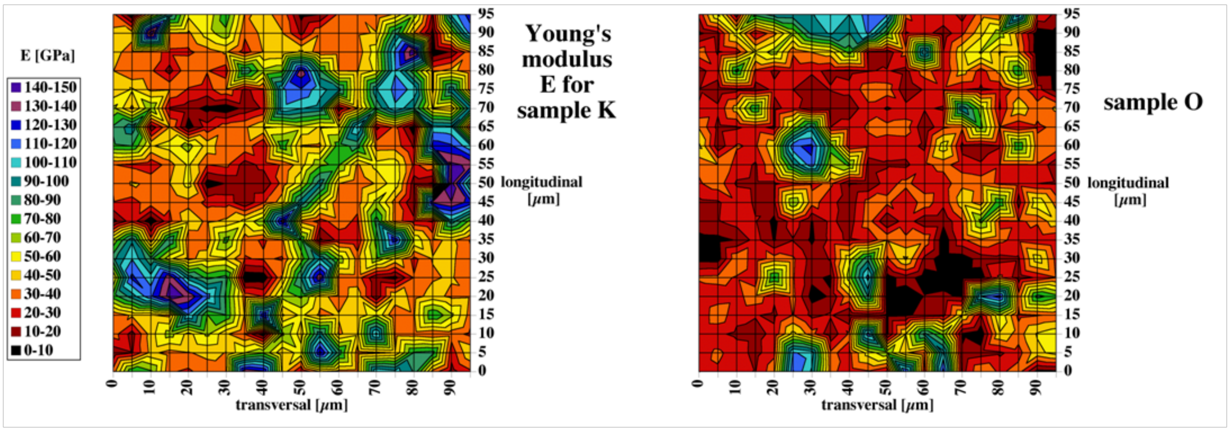


Fig. 3. Young's modules for 20×20-grids

- Transducer
- Honey
- Foil
- Sample
- Foil
- Honey
- Delaypart
- Honey
- Transducer

Fig. 4. Set-up of ultrasonic equipment for pulse transmission technique

The time of flight, *TOF*, was measured manually in each direction of the cubes using an oscilloscope and was corrected by the system transducer delay time (2 for test results). The wave speed and the stiffness component C_{1111} were computed based on the corrected time of flight and the sample density using Equations.

For the control samples by using the 100 kHz transducer, very small standard deviations are observed, whereas significant variations are found for the modified samples. The mean values of all three samples (K1-3 and O1-3) amounts to 27.42 GPa for sample “K” and to 29.47 GPa for sample “O”. The test results measured by using the 500 kHz transducer show a similar behaviour, with a mean value of sample “K” of 27.31 GPa and for sample “O” of 29.61 GPa.

Ultrasonic tests were performed on cement stone samples with and without CNM. The test results showed, that the stiffness of cement stone modified with CNM (C_{1111} = 29.47 GPa) was significantly higher (7.5 %) than the stiffness of the control sample without modification (C_{1111} = 27.42 GPa). For the modified sample a higher standard deviation is found, which might be explained by an inhomogeneous distribution of the CNM.

Table 2

Test results using 500 kHz transducer: dimensions, densities, measured times of flight, wave velocity, and computed stiffness component C_{1111}

Label	Mass wet [g]	side I h [mm]	side II a [mm]	side III b [mm]	density [g/cm³]	side I TOF [µs]	side II TOF [µs]	side III TOF [µs]	Delay	side I corr. TOF [µs]	side II corr. TOF [µs]	side III corr. TOF [µs]	side I v [km/s]	side II v [km/s]	side III v [km/s]	side I C_{1111} [GPa]	side II C_{1111} [GPa]	side III C_{1111} [GPa]
O-1	14.940	20.133	20.037	17.810	2.079	7.61	7.60	7.30	2.51	5.100	5.090	4.790	3.948	3.936	3.718	32.407	32.223	28.748
O-2	16.150	19.777	19.787	19.873	2.077	7.85	7.95	7.94	2.51	5.340	5.440	5.430	3.703	3.637	3.660	28.484	27.474	27.817
O-3	16.670	20.043	20.060	19.867	2.087	7.99	7.73	7.70	2.51	5.480	5.220	5.190	3.658	3.843	3.828	27.918	30.820	30.579
K-1	13.810	19.343	19.320	18.283	2.021		7.87	7.60	2.51		5.360	5.090		3.604	3.592		26.259	26.078
K-2	10.350	16.257	18.140	17.270	2.032	7.00	7.54	7.05	2.51	4.490	5.030	4.540	3.621	3.606	3.804	26.641	26.431	29.407
K-3	12.785	18.293	18.257	18.767	2.040	7.43	7.41	7.55	2.51	4.920	4.900	5.040	3.718	3.726	3.724	28.200	28.317	28.282

R E F E R E N C E S

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