

## НАНОТЕСТЫ БЕТОННЫХ ОБРАЗЦОВ С НАНОТРУБКАМИ И БЕЗ НИХ

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## NANO TESTS ON CONCRETE SAMPLES WITH AND WITHOUT NANOTUBES

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The concrete samples originate from fracture mechanics tests performed on February 19, 2012, at the Department of Geo-Engineering and Building Materials of Izhevsk State Technical University.

After the fracture mechanics tests three types of small specimens (marked with CNT, C3 and control sample) were prepared and tested by means of Nanoindentation (NI) and Atomic Force Microscopy (AFM) at the Institute for Mechanics of Materials and Structures and Institute of Automation and Control, respectively, of Vienna University of Technology.

**Testing Hardware.** High precision Nanoindenter (Hysitron).

*Technical Specifications:*

Maximum load: 12000  $\mu$ N.

Resolution: <1 nN.

Noise floor: 100 nN.

Resolution: 0,0002 nm.

Noise Floor: 0,2 nm.

Drift: <0,05 nm/s.

*Available Testing Modes:*

Quasistatic nanoindentation.

Feedback control-operate in closed loop load or displacement control.

Scratch testing.

nanoDMATM dynamic testing

Dimension Icon Atomic Force Microscopy (Bruker)

*Technical Specification:*

X–Y scan range 90×90  $\mu$ m typical, 85  $\mu$ m minimum.

Z range 10  $\mu$ m typical in imaging and force curve modes.

Vertical noise floor <30 pm RMS in appropriate environment typical imaging bandwidth (up to 625 Hz).

X–Y position noise –0,15 nm RMS typical imaging bandwidth.

Z sensor noise level 35 pm RMS typical imaging bandwidth.

Integral nonlinearity (X–Y–Z) <0,5 % typical.

*AFM Modes:*

Standard: ScanAsyst, TappingMode (air), Contact Mode, Lateral Force Microscopy, Phase Imaging, Lift Mode, MFM, Force Spectroscopy, PeakForce Tuna, Force Volume, EFM, Surface Potential, Piezoresponse Microscopy, Force Spectroscopy;

Optional: PeakForce QNM, HarmoniX, Nanoindentation, Nanomanipulation, Nanolithography, Force Modulation (air/fluid), Tapping Mode (fluid), Torsional Resonance Mode, Dark Lift, STM, SCM, C-AFM, SSRM, TUNA, TR-TUNA, VITA.

**Preparations.** From all three sample types (CNT, C3 and control sample, short K), two samples were glued to metal holders. One sample for each type was polished by a machine and by hand to produce a smooth surface, parallel to the holder. This procedure facilitates the measurements on the indenter and the microscope. The second set of samples was left in its raw state. Fig. 1 shows the six samples.

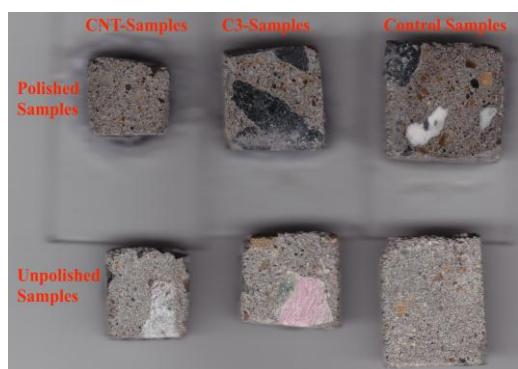


Fig. 1. Tested concrete samples

**Nanoindentation Tests.** On each of the three polished samples, four series of measurements were performed. The same test parameters were used as for earlier tests on concrete:

- Maximum force 1200  $\mu\text{N}$ ;
- 10 s linear increase of the force, 5 s constant force, 10 s decrease to 0 N;
- test grids of 12 $\times$ 12 indents, grid spacing 10 microns (test series 10a and 10b) and 5 microns (5a and 5b) for each sample.

This results in plots for the (reduced) modulus of elasticity as in Fig. 2.

In this example, the values for the reduced modulus were between 1 and 125 GPa. This test area was selected by the help of the optical images,

given by the microscope mounted on the indenter. A seemingly homogeneous area was chosen for each test series.

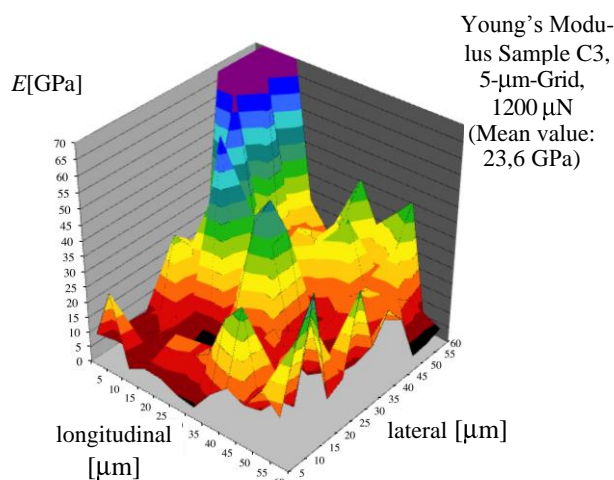


Fig. 2. Young's modulus for 12 $\times$ 12 5- $\mu\text{m}$  grid

In Fig. 3, the area of the 12 $\times$ 12-grid for the test series in Fig. 2 is marked by the white square. (The marks of the indents itself can't be seen in the optical image on this type of surface.)

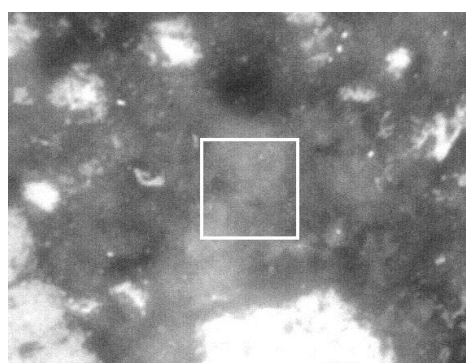


Fig. 3. Microscope snapshot of the test area

As can be seen in Fig. 2, with respect to Young's modulus the test area is actually highly inhomogeneous. In the optical image of the surface, there is hardly any evidence for this inhomogeneity.

Overall, for the six test series, we get the following results for the modulus and the hardness:

Modulus [GPa]	K	K	CNT
10a	19,00	10,41	35,81
10b	18,66	8,63	25,76
5a	17,73	23,58	24,68
5b	19,62	11,60	2,40
Mean value	<b>18,75</b>	<b>13,56</b>	<b>22,16</b>

Hardness [GPa]	K	C3	CNT
10a	1,150	0,457	2,930
10b	0,545	0,352	1,208
5a	0,518	0,968	0,651
5b	0,762	0,537	0,047
Mean value	<b>0,74</b>	<b>0,58</b>	<b>1,21.</b>

Therefore, on each of the three samples, 4 test series (Fig. 4) were performed (10a and 10b with a spacing of 10 microns, 5a and 5b with 5 microns) at 4 different positions.

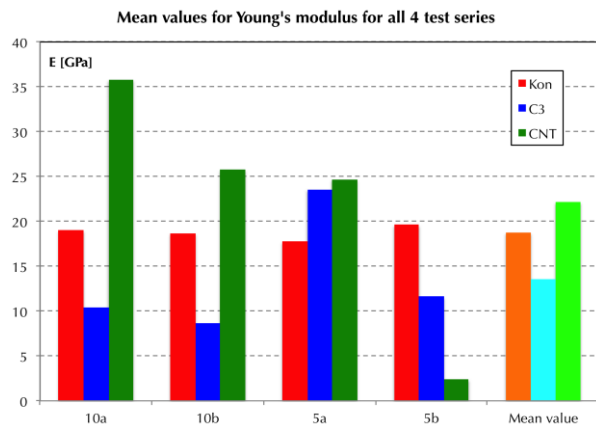


Fig. 4. Mean value for (reduced) Young's modulus

The standard deviations are not plotted, since they amount to 60–200 % of the mean values. As can be seen, only for the control sample there is a reasonable reproducibility for the mean value of the modulus. The fluctuations are especially high for the CNT sample. In the graphs for the modulus and hardness (see Fig. 2 as example), structures in many sizes can be recognized, but none can be attributed with certainty to nanotubes. Therefore, the same samples were analysed by means of an Atomic Force Microscope at the Institute of Automation and Control of Vienna University of Technology.

**Results of AFM Tests.** First, the polished samples were examined, because it is also useful for microscopy, if the surface is smooth and only slightly tilted. The maximum size of the area, which can be scanned in one pass, is about 100 microns. Investigating the CNT sample, in the resulting images structures with a size in the order of 15–25 microns can be seen (albeit blurry), see Fig. 5, 6.

In the polished control sample (without nanotubes), no corresponding structures were visible; see Fig. 7.

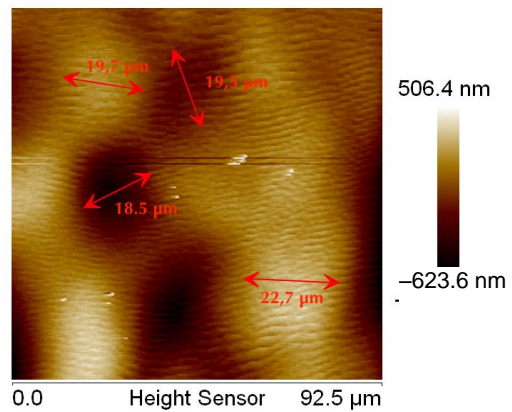


Fig. 5. Polished CNT-sample, position 1, height graphic

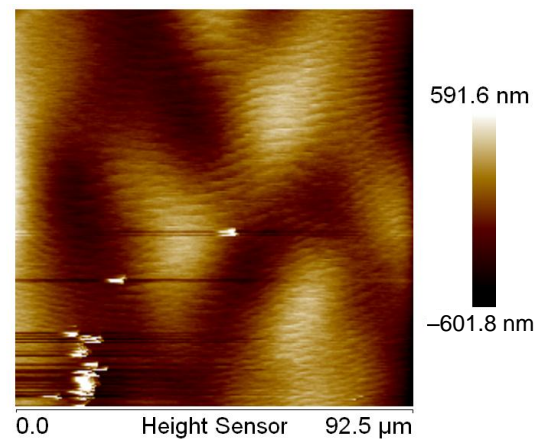


Fig. 6. Polished CNT-sample, position 2, height graphic

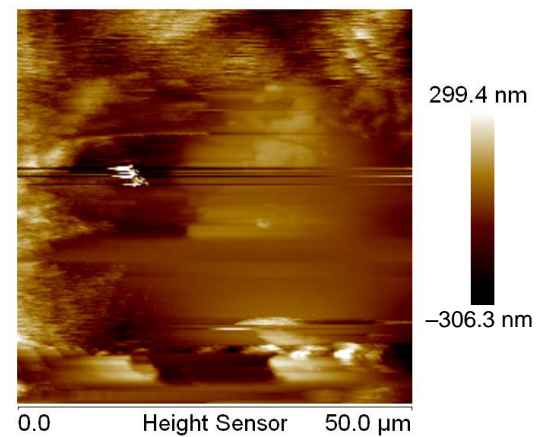


Fig. 7. Polished control sample, height graphic

Next, the unpolished samples were examined. Because of the roughness of the surface, only in certain regions it was possible to scan larger areas (up to 50×50 microns). In some of these areas, structures were visible on the surfaces, whose dimensions fit to the expected nanotubes; see Fig. 8.



Fig. 9 and 10 show the AFM images of a section, in whose upper half the bright areas from Fig. 8 were scanned. There is no significant contrast to the other areas, though.

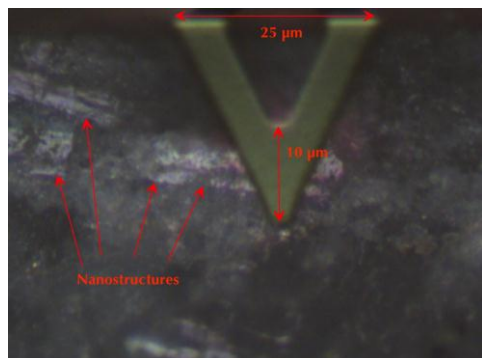


Fig. 8. Unpolished C3-probe, optical snapshot

The V-shaped structure in the upper half of the Fig. 8 is the cantilever, on which the tip is mounted, which is scanning the surface.

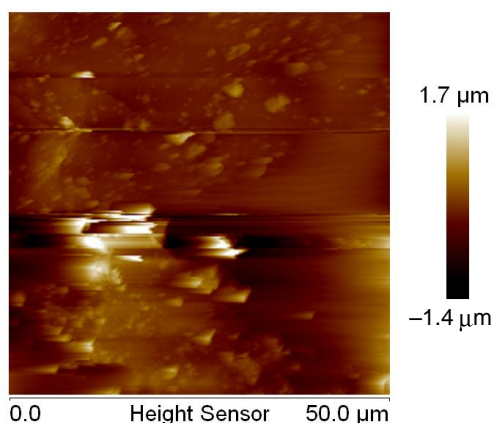


Fig. 9. Unpolished C3-sample, height graphic

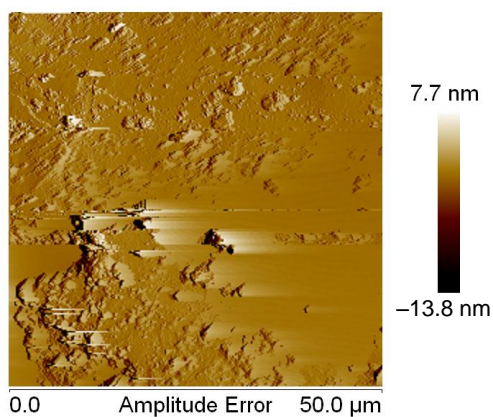


Fig. 10. Unpolished C3-sample, amplitude

On the same sample, another region was examined, because in the optical image there were par-

ticularly evident signs of a baton-shaped structure; see Fig. 11. This structure can also be seen easily in the upper half of Fig. 12 to 14.

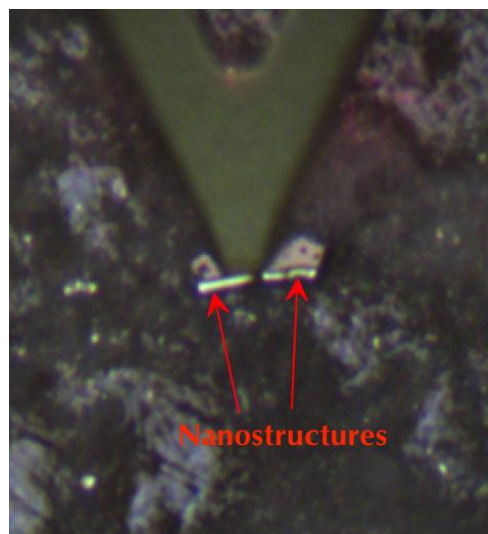


Fig. 11. Unpolished C3-sample, optical snapshot

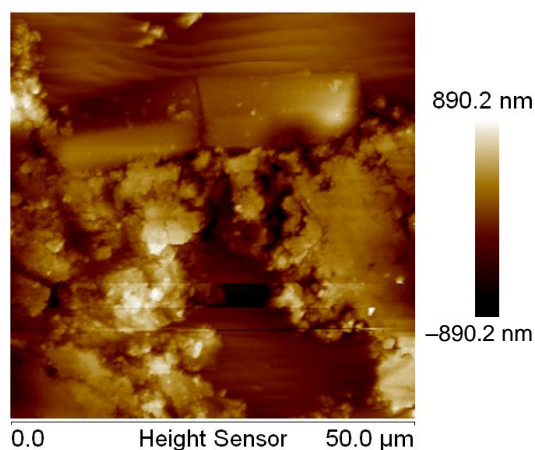


Fig. 12. Unpolished C3-sample, height graphic

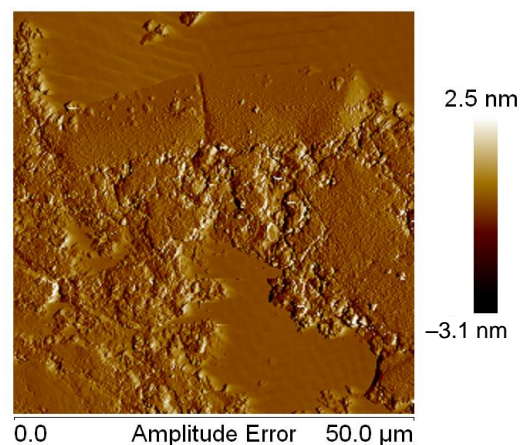


Fig. 13. Unpolished C3-Sample, amplitude

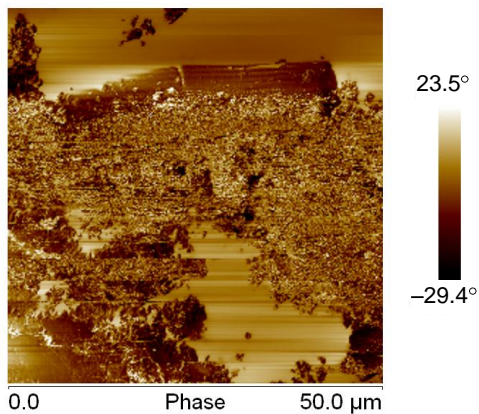


Fig. 14. Unpolished C3-Sample, phase

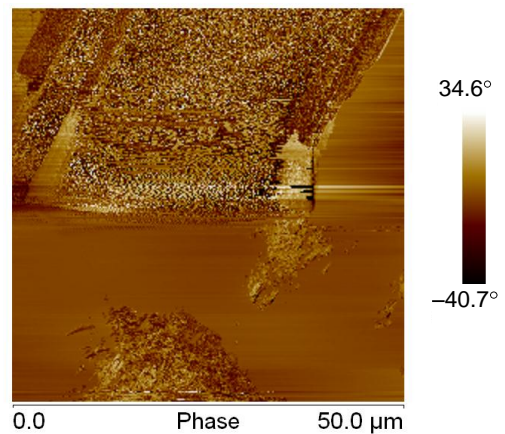


Fig. 16. Unpolished CNT-Sample, phase

Also the unpolished CNT-sample was examined. In one region, for example, an elongated structure was found, that is recognizable in the upper part of Fig. 15 and 16.

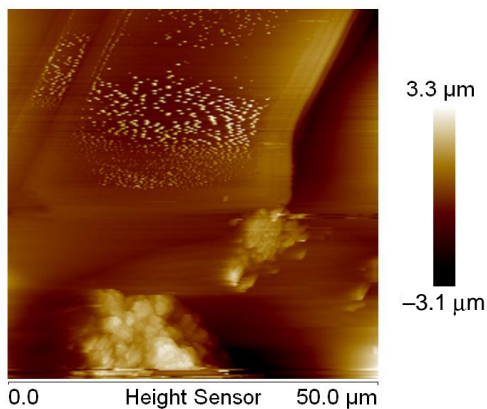


Fig. 15. Unpolished CNT-Sample, height graphic

### CONCLUSION

In the AFM images, structures of a size in the order of the nanotubes can be indicated. But due to the inhomogeneity of the material, a reliable identification does not seem possible. More or less the same applies for the test series with the Nanoindenter. A reliable statement of the effect of the Nanotubes on the elasticity and the hardness is not possible because of the high fluctuation of the elastic modulus and hardness.

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## ОБЕСПЕЧЕНИЕ УСТОЙЧИВОСТИ ОТКОСОВ ДАМБ ДЛЯ ЗАЩИТЫ ОТ НАВОДНЕНИЙ НА РЕКЕ ГОРЫНИ

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В Беларуси последовательно осуществляется программа защиты территорий от наводнений в бассейне рек Припяти и Горыни. Особую

опасность возникновения катастрофических наводнений создает река Горынь. Она принадлежит к типу равнинных рек с преобладанием