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CNT-Distribution in Different Polyurethane Matrices

Introduction

Currently, carbon nanotubes (CNTs) are a major topic of research due to their extraordinary mechanical, thermal, electrical, optical and electrical conductivity properties. One of the most attractive applications is the incorporation of CNTs into polymer matrices to prepare high performance composites. In the present research we incorporate Multiwalled CNTs in solvent containing and water-based Polyurethane (PU) matrices and characterize their distribution in the mass by the use of an Atomic Force Microscope (AFM). The electric conductivity was measured on the polymer films.

Methods and Materials

POLYURETHANE (PU) AND CARBON NANOTUBES (CNTs)

It was focused on particles with a high aspect ratio to obtain awareness of synergistic effects of structurally different particles more easily. Commercially available MWCNT from Nanocyl SA (Belgium) were utilized. The MWCNT had a diameter range of 5 – 30 nm and a lengths of >1 µm according to the manufacturer's data. The nanoparticles were incorporated in coating masses based on PU by means of three roll mill (120 EH-250 of EXAKT). In order to obtain an optimal dispersion of the CNTs, different passages and operation modi were used. They were set to gap mode of 15 µm, 10 µm and 5 µm (process A, B and C) as well as force mode of 3 N and 8 N (process D and E). The CNT particle content varied from 0,5 % up to 6 %. Furthermore, the influence of a dispersing agent on the CNT-distribution was investigated.

ATOMIC FORCE MICROSCOPE (AFM) AND SURFACE RESISTANCE

To gain information on the nanometer-scale organization of the nanoparticle network in the PU/CNT matrix an AFM and Conductive-AFM (Type Nanowizard® 3 from JPK) were applied. The measurements with the AFM were performed in intermittent contact mode (AC Mode) with a NCH-Cantilever (uncoated Si-cantilever, length 125 µm, tip radius < 0,8 nm). In the AC mode the cantilever is usually driven close to the resonance of the system, to give a reasonable amplitude for the oscillation and also to provide phase information. The phase can be used to receive sample properties like stiffness. The C-AFM measurements are used to get topography and conductivity images simultaneously. Therefore, a conductive cantilever (Typ ContE-G, Cr/Pt coating, length 450 µm, tip radius < 25 nm) scans the sample surface in contact mode to generate a topography map. A bias voltage is applied between tip and sample and the tunneling current between the two is measured.

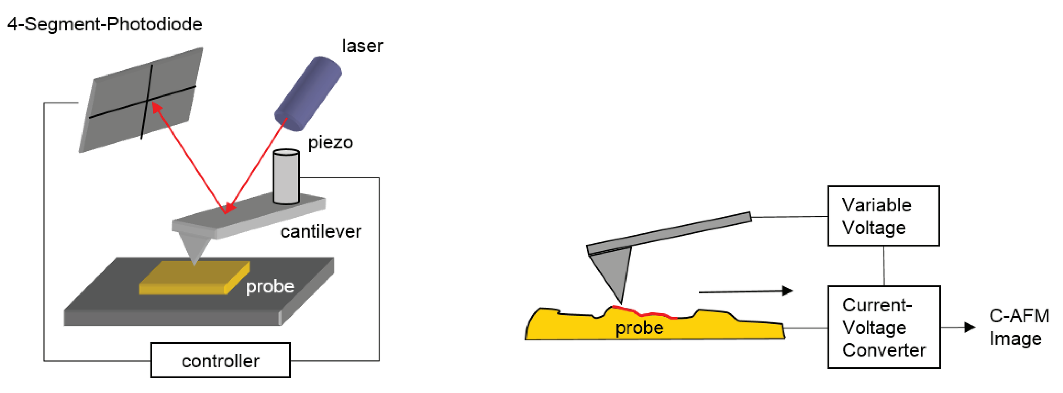

Fig. 1: Principle of AFM (left) and C-AFM (right)

According to DIN 54345 part 1 the surface resistance was measured as four terminal method (Milli-To2 of Fischer Elektronik GmbH & Co). After climate conditioning (24 h, 23 °C and 50 % rel. humidity) the samples (thickness 50 µm, 100 µm) were applied with a voltage of 750 mV for 60 s. The resistivity as well as the electrical conductivity were calculated from the measuring value of the square resistance by multiplying the thickness.

Results and Conclusions

MWCNT IN SOLVENT CONTAINING PU

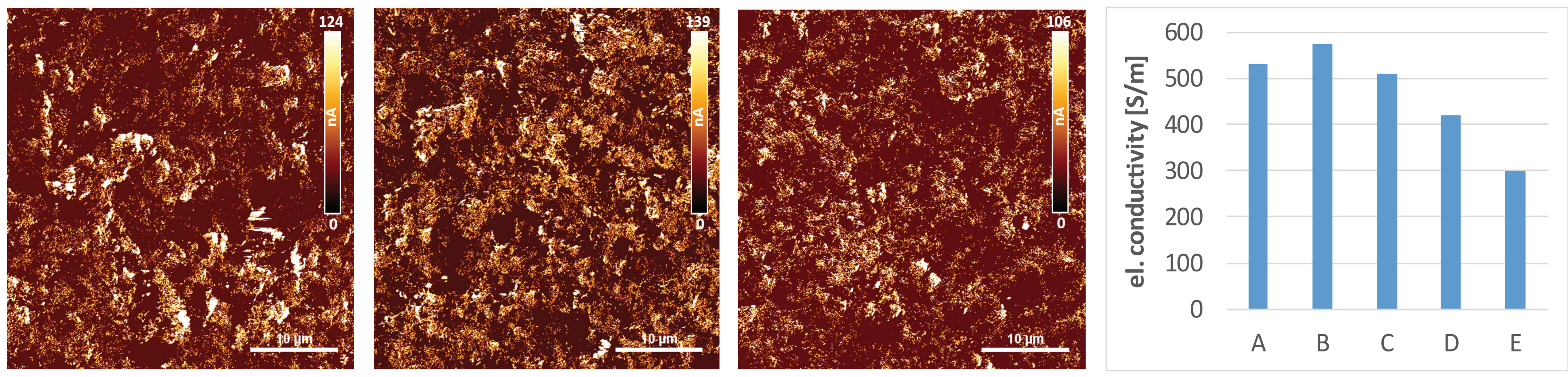

Fig. 2: C-AFM images (bios Voltage 2,5 V) of solvent containing PU-masses containing 3 % CNT prepared by different operation modi (from left to right A, C, D); electrical conductivity

- decrease of the particulate structures from A to E and separation of CNT bundles from the agglomerates
- connecting pathway between the CNT agglomerates
- application of force at the roller gap causes a break of the CNT into small fibrous segments

MWCNT IN WATER-BORN PU WITH AND WITHOUT DISPERSING AGENT

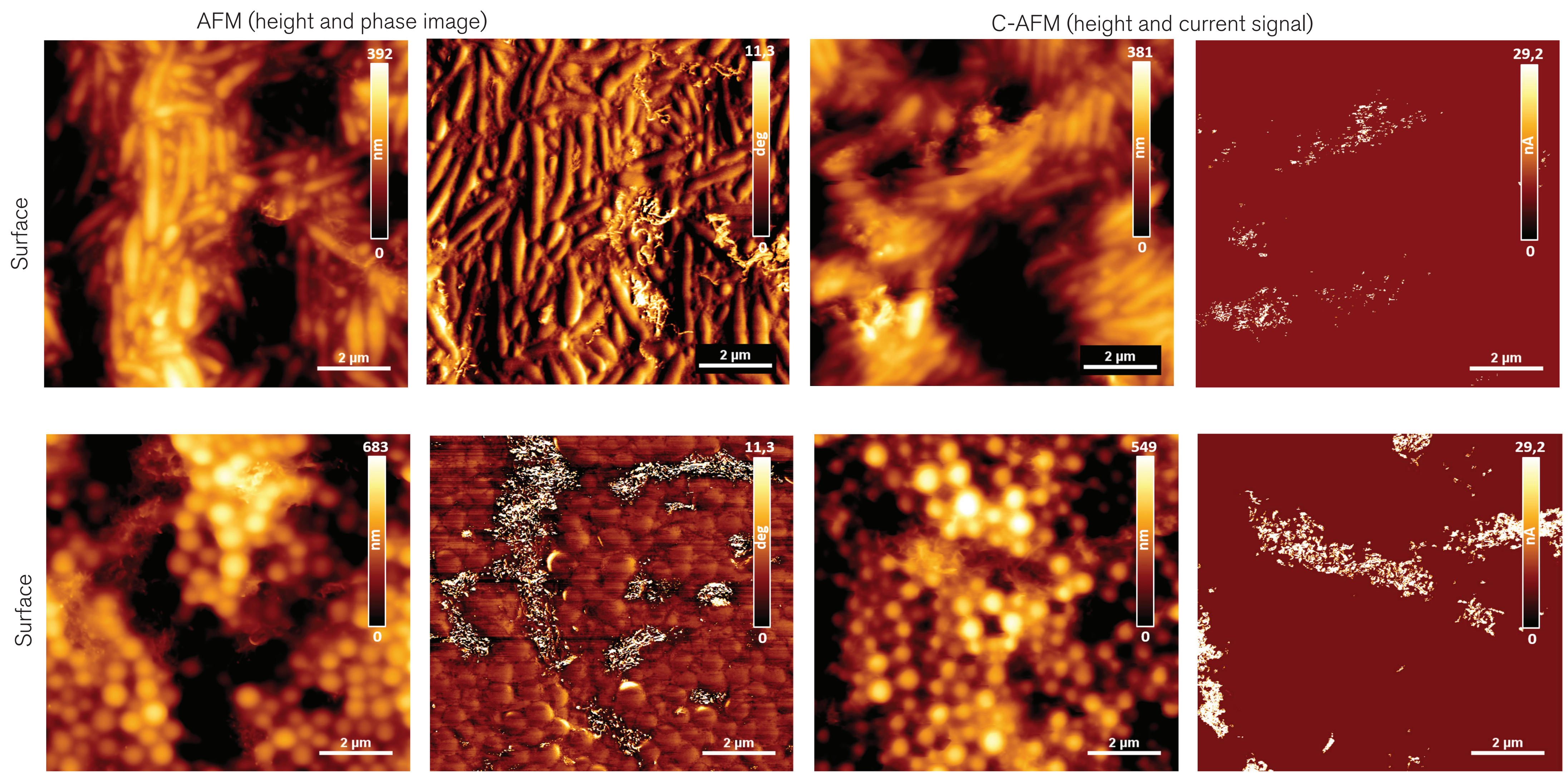

Fig. 3: AFM and C-AFM images (bios voltage 2,5 V), water-born PU-masses without (top) and with (bottom) dispersing agent, 4 % CNT

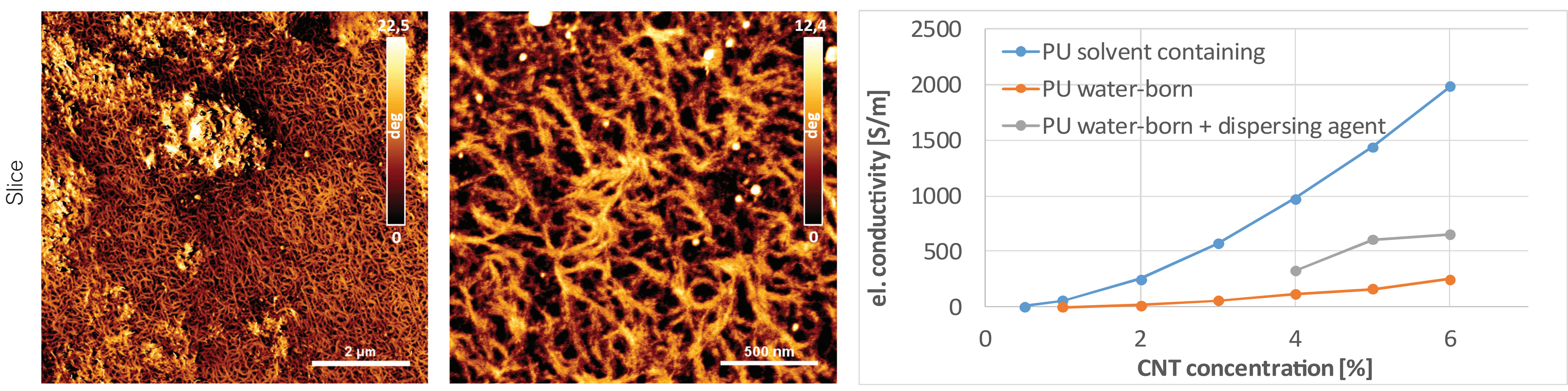

Fig. 4: AFM phase images of microtome slides water-born PU-foil with dispersing agent, 4 % CNT, conductivity of different PU-foils

- depending on the dispersing agent spherical or long shaped polymer surface structures are formed
- the visualization is limited because the CNTs are covered by the PU-polymer
- connecting pathways between the nanoparticles are visible in the microtome slides only

The influence of the mass preparation could be demonstrated macroscopically by the electric conductivity at the polymer films and also on the nanoscale with the AFM. The dispersing agent influences the surface structures. To see the connecting pathways of the CNTs microtome slides have to be prepared and analyzed. For the characterization of CNTs in different PU-matrices the AFM images of height, phase and conductive images have to be studied in context.

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