VISUALIZING CARBON NANOTUBES INSIDE POLYMER COMPOSITES BY SCANNING ELECTRON MICROSCOPY

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We present a method to visualize conductive filler particles in insulating polymer matrices with a scanning electron microscope (SEM). Carbon nanotubes (CNT) are used as fillers in an epoxy polymer based on bisphenol-A resin with an amine hardener. The SEM (LEO 1530) is equipped with a field emission cathode which allows a resolution of approximately 2 nm. The influence of several SEM and sample parameters is analyzed. We discuss the challenge of image interpretation in view of the apparent lack of appropriate information in literature [1][2].

The technique under investigation is called charge or voltage contrast imaging [3]. It is based on sensing potential variations on *uncoated* sample surfaces caused by inhomogeneous charging (due to conductivity differences of filler and matrix). The first attempts to explain such charge contrast images [4][5] do not necessarily apply to modern SEM and detector types and contain errors.

We point out that not every detector is sensitive to charge contrasts, as displayed in Fig. 1. Our Everhart-Thornley detector (ET) is located lateral to the sample and seems to detect topographic contrast only (Fig. 1a). Two ditches, but no nanotubes, are visible at the sample surface. Our InLens detector (located inside the focusing lens), however, monitors nanometer-sized charge contrasts attributed to nanotubes inside the polymer (Fig. 1b). Nevertheless, ET detectors in Environmental SEM seem to be sensitive to charges [5]. While Hitachi Inc. claims that their InLens detector (which works different than ours) is completely insensitive to sample charges, charge contrast images recorded with that detector were reported by other groups [6].

We demonstrate a new approach for analyzing the quality of nanotube dispersions over several length scales, from tens of nanometers to some hundred micrometers. Figure 2 illustrates a zooming out sequence of the same sample area displaying a homogeneous and dense nanotube layer.

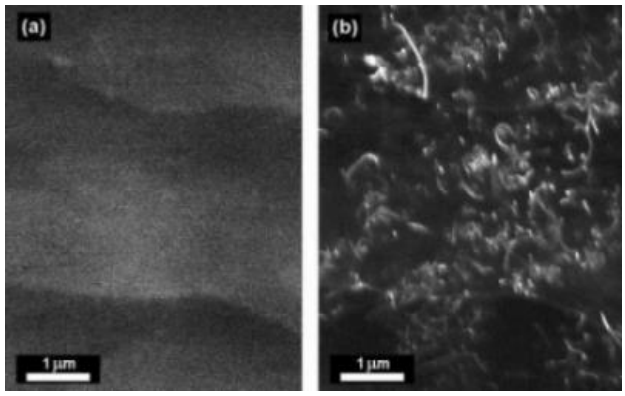

Fig. 1. Cryo fractured surface of a composite containing 1 wt% CNT recorded with two different detectors: (a) Everhart-Thornley, (b) InLens

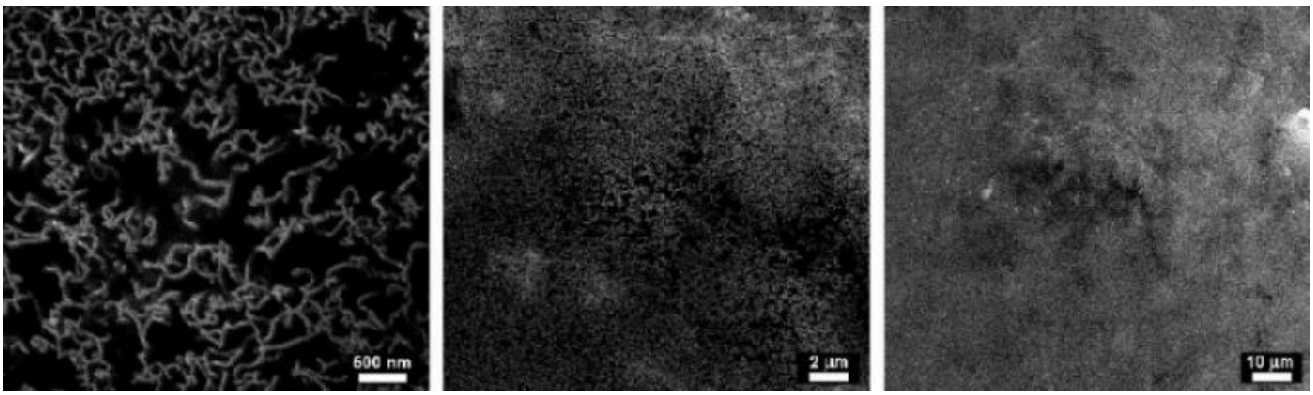

Fig. 2. Spin-coated composite containing 1 wt% CNT recorded at different magnifications and 10 kV acceleration voltage

We discuss the complex mechanism of sample charging which depends on the relationship of the electron dose to the discharging capability (conductivity) of individual sample regions. The dose itself depends on scanning density (magnification), scanning speed (beam dwell time per area) and acceleration voltage. The influence of the latter parameter on the image contrast is shown in Fig. 3. This also contradicts results reported in [5], since no additional nanotubes from deeper regions appear with increasing acceleration voltage. Thus, only limited information about a three-dimensional CNT network can be obtained this way.

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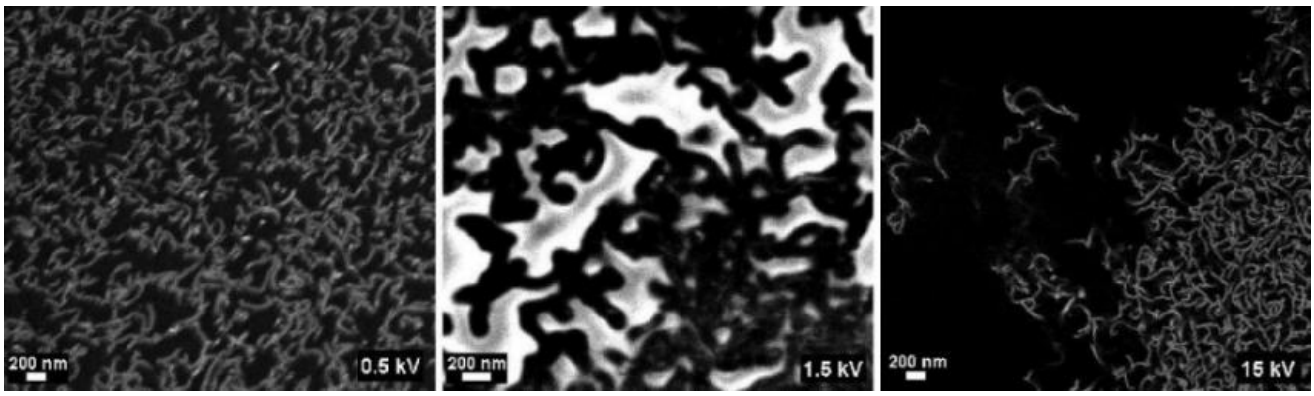

Fig. 3. The same sample as in Fig. 2 recorded at different acceleration voltages