FORMATION OF NANOCOMPOSITES BASED ON CARBON NANOTUBES AND SILICATES

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1. Introduction

Electrical, thermal, optical and mechanical properties of carbon nanotubes (CNTs) have been in the focus of interest since their discovery at the beginning of the nineties [1]. Nanocomposites with carbon nanotubes nowadays constitute one of the most rapidly developing areas of research of fibrous nanomaterials. Potential applications of nanocomposites based on CNTs and silicates include nanoporous filters, heat insulators, protecting materials for low-molecule gases, selective adsorbents or lubricants and light materials applicable for various purposes. The future prospects appear to be the so-called green nanocomposites, silicate nanocomposites [2] and biologically degradable carbon polymers in which both the catalytic and ion-exchange function of the silicate matrix can be utilized [3]. Highly prospective is creation of cross-linked CNTs/silicate materials or nanocomposites on the basis of CNTs meshes and of a polymer [4].

2. Experimental materials and conditions

Synthesis of CNTs can be performed on silicates with a layered and spatial structure [5-7]. We prepared nanocomposites based on CNTs+zeolite and CNTs+montmorillonite. The metal impregnated samples were prepared by immersing the silicates into an aqueous solution of $Fe(NO_3)_3 \cdot 9H_2O$ with different concentrations: 0.4 M, 0.04 M and 0.004 M/100 g of silicate. The suspensions were stirred for 12 hours, deposited on a polished Si wafer and allowed to dry quickly by an infrared lamp. In situ creation of nanocomposites and synthesis

of carbon nanotubes was carried out in the HF CVD reactor, where the precursors are activated by five tungsten filaments heated up to 2200°C. The working atmosphere was a mixture of methane and hydrogen. The pressure and temperature during deposition were 3000 Pa and ≈ 600°C, respectively. The synthesis time was 30 minutes.

3. Results

Nanocomposites based on carbon nanotubes and silicates were obtained on both types of substrates. The quality and nature of carbon deposited on the silicate surface and the growth of CNTs within the matrix were examined by scanning and transmission electron microscopies and Raman spectroscopy. SEM measurements showed images of carbon nanotube bridges and networks in nanocomposites. Figures 1 and 2 show CNTs grown on Fe-zeolite. SEM micrographs confirmed a high catalytic efficacy of the Fe-zeolite which is probably brought about by the structure of the zeolite that allows anchoring of Fe^{3+} catalytic particles in the pores and prevents their migration from the sample. High density of CNTs was observed in the whole volume of the zeolite. Figure 3 shows a TEM image of multiwalled CNTs grown on zeolite. One can see particles of the catalyst at the ends of the CNTs.

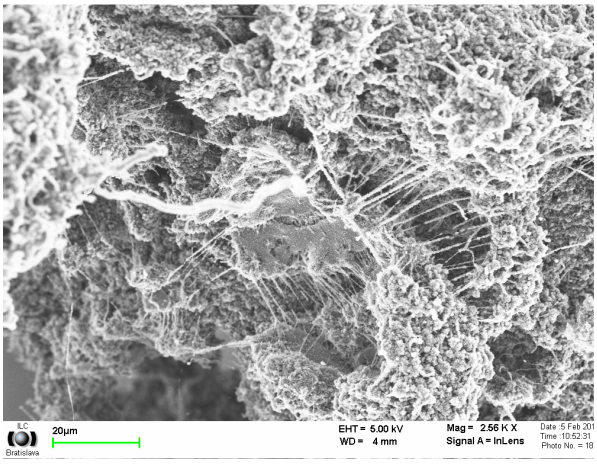

Fig. 1: *SEM image of CNTs grown on* Fe-*zeolite pretreated with* $Fe(NO_3)_3 \cdot 9H_2O$.

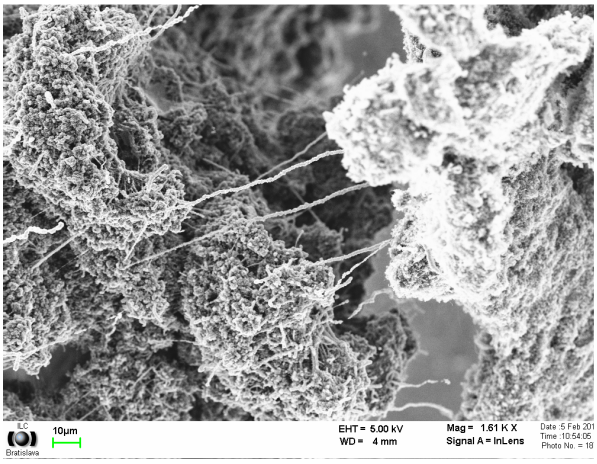

Fig. 2: *Detail, SEM image of CNT bridges.*

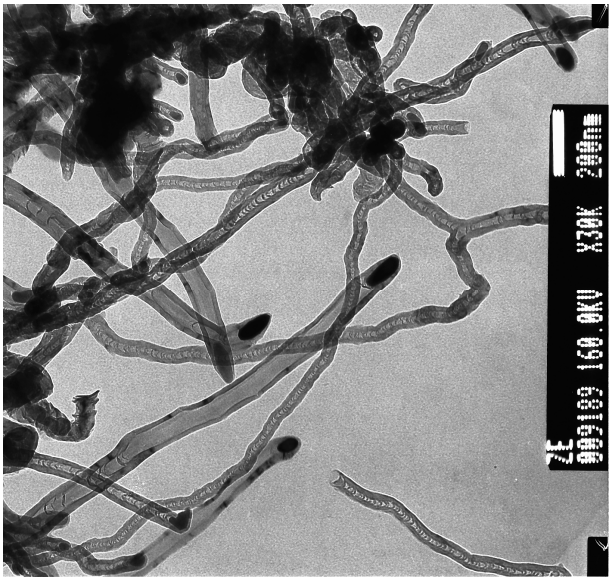

Fig. 3: *TEM image of CNTs grown on Fe-zeolite. At the ends of CNTs one can see particles of the catalyst.*

Figure 3 shows CNTs grown on Fe-montmorillonite. Since in the case of montmorillonite the catalyst particles can be located not only on the outer surface but also in the interlayer spaces of the mineral, its catalytic efficacy is enhanced.

Figure 4 shows the growth of amorphous carbon on the surface of CNTs. Amorphous carbon, detected by Raman spectroscopy, is an undesirable phase, on the other hand it proves the adsorption ability of the surface of CNTs, see Fig. 5.

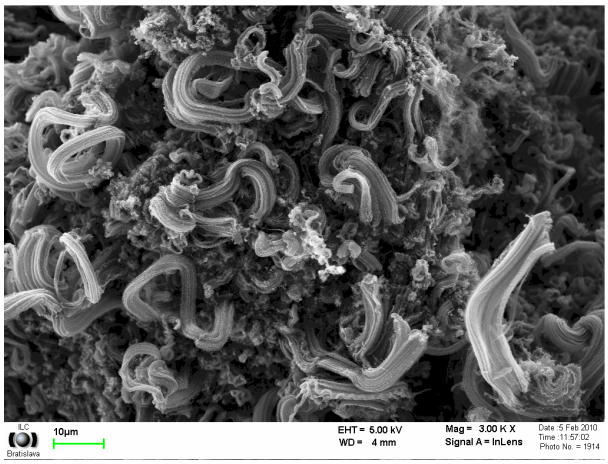

Fig. 4: *SEM image of the bundles of CNT grown on* Fe-*montmorillonite pretreated with* $Fe(NO_3)_3 \cdot 9H_2O$.

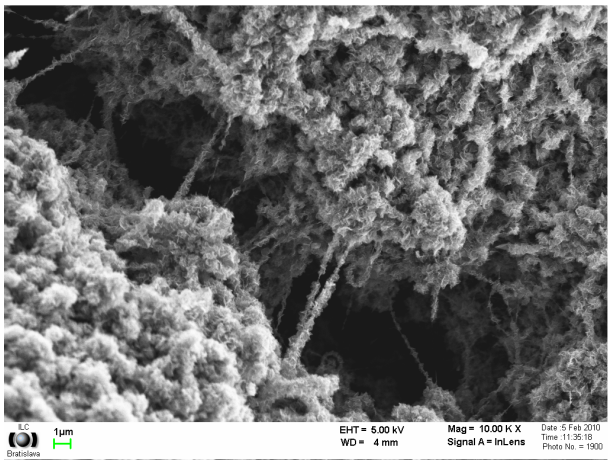

Fig. 5: *SEM image of the CNT bridges grown on* Fe-*montmorillonite pretreated with* $Fe(NO_3)_3 \cdot 9H_2O$. *One can see the growth of amorphous carbon on CNT bridges and on the nanotubes grown in the matrix.*

4. Conclusion

Nanocomposites based on CNTs+zeolite and CNTs+montmorillonite were produced *in situ* by HF CVD. Silicates – zeolite and montmorillonite – were used in the synthesis as substrates for immobilizing the catalytic particles of iron ions. We studied the mechanism of nanocomposite formation and CNTs growth on catalytic particles, the influence of the type catalytic support upon the morphology, quality and structure of the final nanocomposite, especially on the formation of carbon nanotube bridges. The CNTs formed a three-dimensional grid. The length of nanotube bridges was in a range from several nm to nearly 10 μm. Diameters of the grown MWNTs were in a narrow range but the nanotubes contain also other forms of carbon. TEM and Raman measurements showed the presence of amorphous carbon on the surface of MWNTs. Base growth and tip growth modes were observed on *in situ* created nanocomposites. Formation of carbon nanotube bridges and grids in the material can provide a promising way to produce new nanocomposites with improved mechanical and absorption properties.

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