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Nanocomposite thin film structures based on polyarylenephthalide with SWCNT and graphene oxide fillers

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Cyclic voltammograms (CV) and electrochemical impedance spectra were recorded using an AutoLab PGSTAT 204 potentiostat/galvanostat equipped with a FRA 32M module (Metrohm AutoLab, the Netherlands) with Nova software. Electrochemical measurements were carried out in a three-electrode cell consisting of a modified glass-carbon electrode (GCE) with a diameter of 3 mm as a working electrode, platinum wire as a counter-electrode and Ag/AgCl (saturated KCl) as a reference electrode. All the measurements were carried out at room temperature, 22 ± 5 °C. A Nanoeducator II NT-MDT atomic force microscope was used to study the surface morphology of composite films and PAP films.

PAP polymer samples (≥99%) were provided by the Ufa Institute of Chemistry, Russian Academy of Sciences (Ufa, Russia). PAP film was modified with SWCNT (diameter: 0.7 - 1.1 nm) (Sigma-Aldrich, USA) and GO (powder: 15-20 sheets, 4-10% edge-oxidized) (Sigma-Aldrich, USA). The reagents and solvents used were pure and were used without additional purification. To modify the electrodes and create field-effect transistors, solutions of the PAP polymer in chloroform prepared by dissolving 20 mg of PAP in 200 μl of chloroform were used. PAP/GO and PAP/SWCNT composites were obtained by adding 5 mg of GO and 5 mg of SWCNT, respectively, to the resulting solution. To improve dispersion, the solutions of the studied composites were kept for 20 minutes in an ultrasonic bath (Elmasonic One, operating frequency 35 kHz). The 5.0 mM solution of the $[Fe(CN)_6]^{4-/3-}$ redox couple (1:1) was prepared using 0.1 M aqueous solution of KCl as a background electrolyte. Aqueous solutions were prepared using deionized water (conductivity 0.1 μS).

Using an automatic pipette, 10 μl of the prepared PAP, PAP/GO or PAP/SWCNT solution was taken and applied to the surface of the pre-polished GCE, then the electrode was dried for 6 min under an infrared lamp and allowed to cool for 3 min. Before each measurement, the GCE surface was polished for 1 min using a deagglomerated suspension based on Al_2O_3 (0.3 microns) and a Spec-Cloth Adhesive black disk 200 mm polishing material purchased from Allied High Tech Products Inc. (USA) followed by repeated washing of the electrode with deionized water and air drying at room temperature.

The voltammetric and electrochemical impedance characteristics of GCE /PAP, GCE /PAP/GO, and GCE /PAP/SWCNT electrodes were studied in a standard 5 mM $[Fe(CN)_6]^{4-/3-}$ solution in 0.1 M KCl background electrolyte solution. The $[Fe(CN)_6]^{4-/3-}$ solution (20 ml) was placed in an electrochemical cell. Impedance spectra were recorded in the AC frequency range from 50 kHz to 0.1 Hz with an amplitude of 5 mV. The CV were recorded in the potential range from 0.6 to 1.0 V at a scanning rate of 0.1 V s^{-1}. The data array for each sample comprised five parallel measurements, which was sufficient to obtain reproducible results. The voltage characteristics of field-effect transistors were measured using the following instruments: a Mastech HY3005D-2 power supply and a Tektronix DMM-4020 multimeter as an ammeter.

The morphology of the surfaces of the films was also studied. AFM images (Figure S1) were obtained using Nanoeducator II.

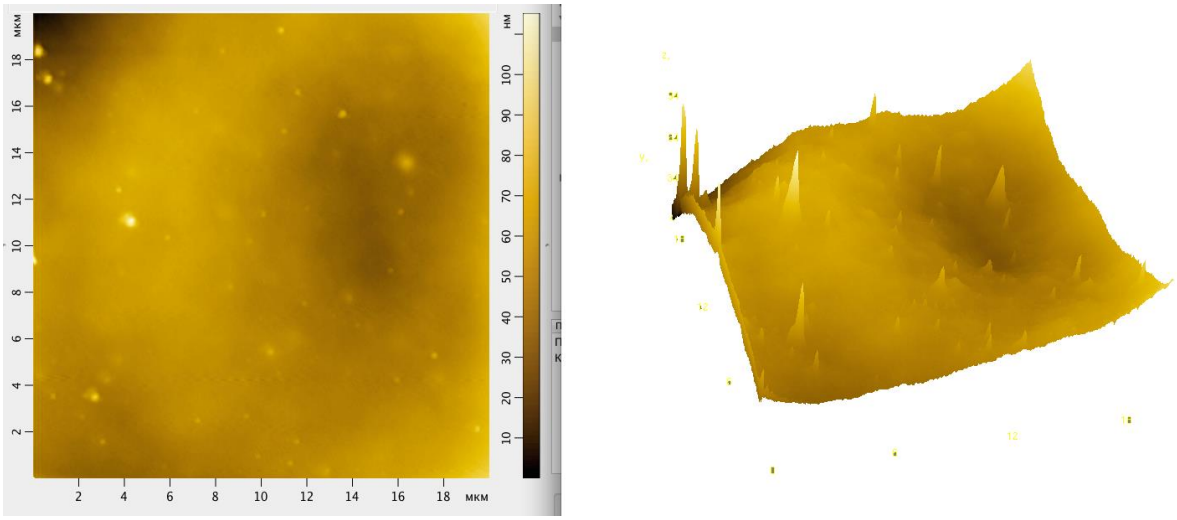

Figure S1a. AFM image of pure PAP; scan size = 20 by 20 μm.

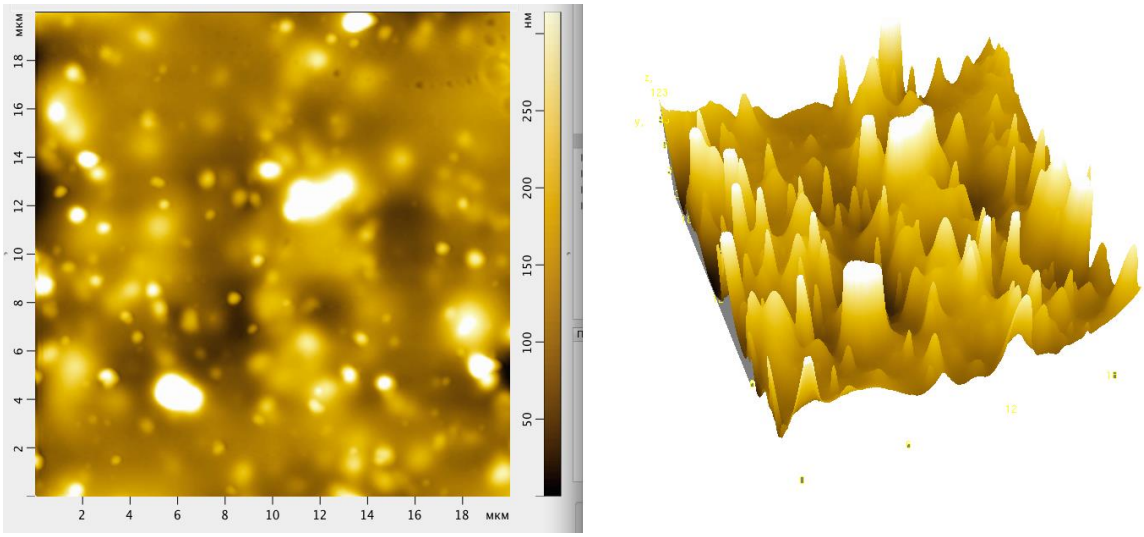

Figure S1b. AFM image of PAP/GO; scan size = 20 by 20 μm.

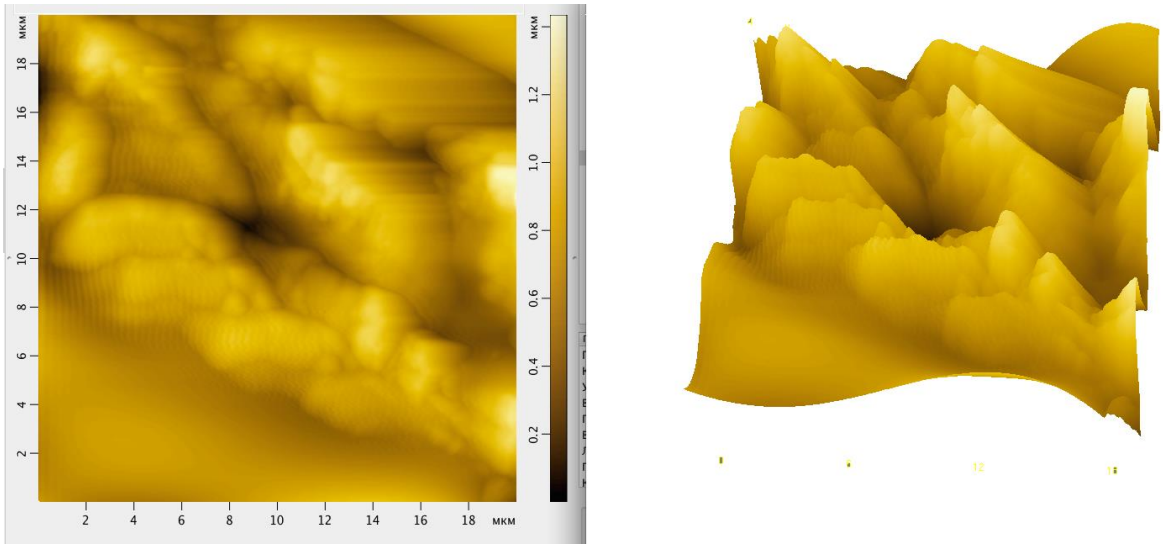

Figure S1c. AFM image of PAP/SWCNT; scan size = 20 by 20 μm.

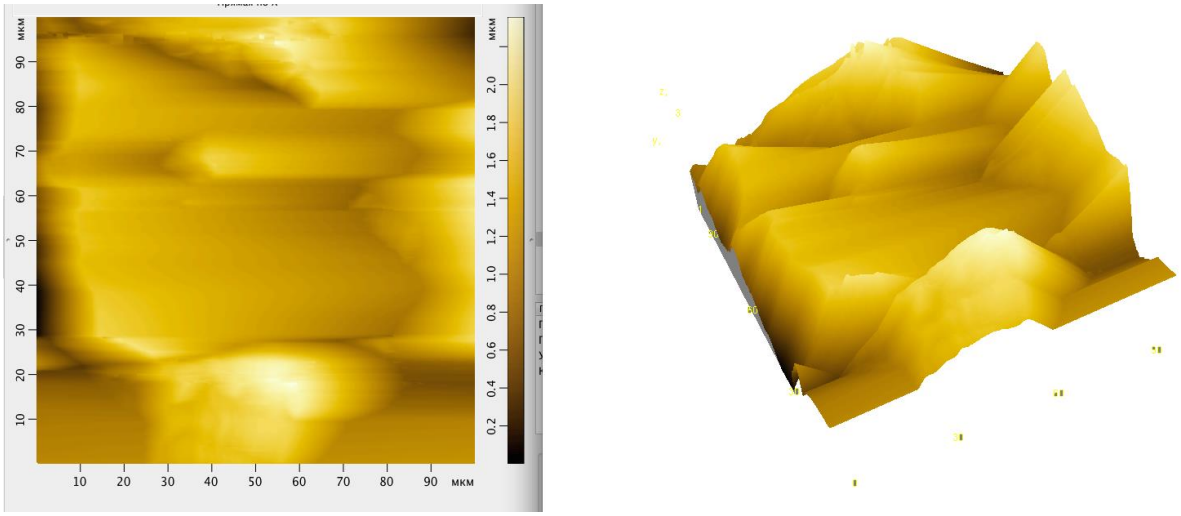

Figure S1d. AFM image of PAP/SWCNT in the gap area; scan size = 20 by 20 μm.