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# **Electro Mechanical Scanning Probe Lithography of Carbon Nanostructures**

#### V D Frolov<sup>1</sup>, V I Konov<sup>1</sup>, S M Pimenov<sup>1</sup>, S V Kosheleva<sup>2</sup>

- <sup>1</sup> A.M. Prokhorov General Physics Institute of RAS, Vavilov str. 38, 119991 Moscow, Russia
- <sup>2</sup> Moscow State Institute of Radiotechnics, Electronics and Automation, Vernadsky pr-t 78, 119454 Moscow, Russia

E-mail: frolov@ran.gpi.ru

**Abstract**. Carbon nano-processing as one of the most promising methods for creation of nanostructures and various types of electronic and optical systems is analyzed. A new method of electro-induced scanning probe lithography (SPL) based on the formation of ordered carbon nanostructures by combining electrical and mechanical actions on the original material in a scanning probe microscope (SPM) is proposed and tested.

#### 1. Introduction

In recent years, many materials have been subjects for electric field-induced modification in a scanning probe microscope (SPM) (see for example the review [1]). The nanoprotrusion formation was demonstrated in experiments with silicon [2-5] and some metals [6-9] that was considered as a result of their local oxidation at positive voltages, i.e., anodization of the sample surface under the probe actions in the presence of adsorbed water vapors.

Scanning probe lithography (SPL) is highly effective and essentially the only method for precision processing of carbon compounds in the nanometer scale. However, in the case of carbon the surface modification shows more complex character compare to inorganic materials. For instance, either protrusions or cavities were resulted from the SPL of amorphous carbon films [10-13], mostly depending on the applied voltage and the environmental conditions (air or vacuum). For highly oriented pyrolytic graphite (HOPG), the only type of modification (nanocavity) was observed in similar nanoprocessing regimes. In the case of hydrogenated diamond surfaces it was found that the SPL induced protrusions as well as cavities, the formation (and registration) of which depends not only on the bias voltage but also on the mode (contact or non-contact) of registration in SPM [15-18].

Experimental evidences were done about local oxidation of carbon films whilst prevailing the SPM-actions threshold that lead to the removal of the uppermost surface layers (i.e. the appearance of nanocavities). In order to explain the formation of nanoprotrusions on diamond-like carbon (DLC) films, it was proposed that the carbon phase transitions occur in carbon, being the result of local heating of the material [11,13,18]. Another explanation related to the properties of hydrogenated diamond surface was suggested in works [15,16], where nanoprotrusions were described as artifacts of

SPM mapping, namely, the congestions of water adsorbate on the oxidized area which are manifested in the form of appropriate modifications of the surface topography.

Thus, a variety of reactions of carbon materials under the electric field actions is of great interest and importance. At the same time, such a variety causes considerable difficulties in the search of regimes and conditions, providing stable and reproducible modifications of carbon material.

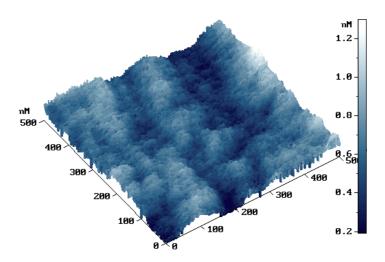
In this paper, a new method of electro-mechanical SPL of carbon materials is described. Experimentally, the SPL-based processing is explored for nanostructuring of thin graphite-like films.

### 2. Experimental

The origin samples were prepared by annealing the 10 nm thick diamond-like carbon films on Si substrates at 620° C under vacuum conditions. According to X-ray photoelectron spectroscopy, under these conditions there is a complete graphitization of the films with the formation of graphite nanoclusters, which is manifested in the considerable narrowing of the C 1s line to values of 1.0 - 1.1 eV [17]. Experiments were carried out in SPM Solver P47 (NT-MDT). To implement the mechanical effects (SPM-nanoengraving), the SPM contact mode was used. As instruments of nanoengraving, silicon cantilevers (force constant is ~ 10 N / m, tip radius is of 5-10 nm) were used. The reason is that such cantilevers have sufficient mechanical strength and toughness (~ 2.4 GN/m²) for the deformation of graphite layers (~ 0.4 GN/m²). The scan speed was 100 nm/sec. The designed pressure of the probe on the surface was 10  $\mu$ N. In order to realize electric field actions on the film surface, the voltage of positive polarity was additionally applied between the sample and the grounded probe.

#### 3. Results and discussion

In Fig. 1, the original surface topology is presented. The right of Fig. 1 shows statistical data on the surface relief. As follows from the relief map, the film surface has a granular structure with a characteristic lateral size of grains ~50 nm. The surface roughness is of 0.15 nm, the relative spread of the film thickness is ~20%, thus indicating the films completely cover the substrate.

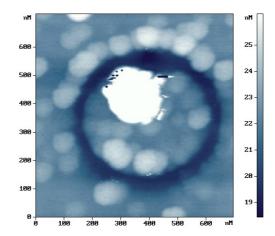


**Figure 1.** The original surface topology.

Fig. 2 illustrates the surface topography after 3 cycles of the mechanical actions of the probe (the cycle duration was -5 s).

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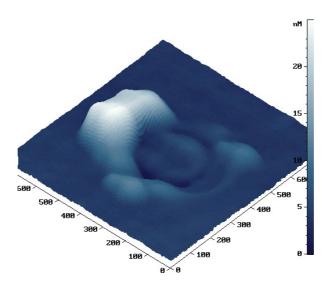
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**Figure 2.** Topography of the sample surface after the mechanical treatment along the circle.

As can be seen from Fig. 2, scanning the probe leads to the formation of cavities in the film surface. (the cutting speed is of 0.2 nm/s), Additionally, the ledges the film surface appears that can be obviously related to ejections of particles of matter from the area of the cut.

The surface topography after the combined electro-mechanical treatment is shown in Fig. 3. It is established that the cutting speed increases to ~0.5 nm/s, leading to the cut about 5 nm in depth. We propose that this process develops due to the transformation of graphite particles to the soot [18]. (The chemical content of soot in this study was not the subject of investigation, but we plan to continue to further explore this aspect).



**Figure 3.** Topography of the sample surface after the electro-mechanical treatment..

It is well known that the soot is rather soft material hence can be effectively removed by scratching. To confirm this circumstance, the treated film was scanned in a contact mode regime. The result is presented in Fig. 4. It is found that the nanoparticles are effectively removed that is clearly seen from comparison of Fig. 3 with Fig. 4.

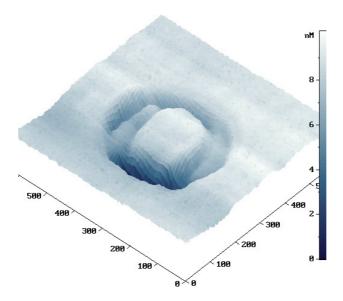
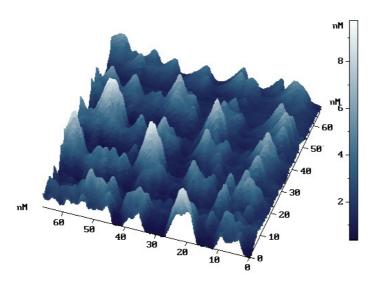


Figure 4. Topography of the sample surface after the electro-mechanical treatment..

It allowed us to form nanoobject arrays with the density of  $\sim 10^{12}$  elements/cm<sup>2</sup>.



**Figure 5.** The nano-object array with the density of  $\sim 10^{12}$  elements/cm<sup>2</sup>, formed on the graphite film surface.

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It is distinctive that the proposed method can be used for the formation of nanostructures in different carbon films, including mechanically strong and chemically resistant materials such as DLC films.

#### 4. Conclusion

The experience on electro-induced SPL of carbon nanostructures enables us to conclude that this process can be effectively realized in air. At once this process allows us to solve the problem on removing the remnants of reaction which during the only mechanical action become apparent as particles of matter removed from the cut, and during the additional electric actions are transformed to the soot. Owing to the small tip radius, it provides the formation of narrow grooves.

## Acknowledgement

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