THE PULSE SHAPE EFFECT IN FIELD-INDUCED NANOMODIFICATIONS OF DIAMOND-LIKE CARBON FILMS

E.V. Zavedeev*, V.D. Frolov, V.I. Konov, S.M. Pimenov
Natural Sciences Center, General Physics Institute, Vavilova str. 38, Moscow, 119991, Russia
*E-mail: zavedeev@nsc.gpi.ru

Recent years, a number of materials were examined for the purpose of field-induced modifications using an air-operated scanning probe microscope (SPM). Local surface oxidation under the action of positive bias applied between the sample and the SPM tip, i. e. anodization, was demonstrated in the experiments with silicon and certain metals.

The same actions resulted in formation of nanocavities in highly-oriented pyrographite (HOPG), suggesting the anodization to be responsible for removing the uppermost surface layers from the substrate [1]. A more complex behavior was observed for amorphous carbon materials. Both graphite-like [2] and well-conducting diamond-like carbon (DLC) films [3] showed either nanoprotrusions or nanocavities inside exposed areas, depending on the voltage magnitude. Evidences were given that, above a threshold, the SPM actions led to the anodization. To explain patterning the nanoprotrusions, phase transitions in carbon due to local heating of the material were proposed [3]. However, the variety in reactions of carbon materials under the electrical field actions is still far from understanding, From this point of view, it seems important to compare the character of nanomodifications in the well-conducting and poorly conducting DLC films.

In the present work, we focus on the field-induced modifications of low-conducting hydrogenated DLC (a-C:H) films (sheet resistance R~ 10^6 Ohm/$\square$). The a-C:H films of 1 μm thickness were grown on Si substrates by means of r.f. plasma chemical vapor deposition in the C_2H_2 atmosphere [4]. To create patterns, the SPM probe coated with conducting layers (TiN, TiO, Pt) was scanned along a given line in a contact mode, and, during the scanning, a series (N= 1 ÷ 5000) of rectangular voltage pulses, either positive or negative, or bipolar, with the magnitude U in the range from -10 V to +10 V and duration T= 0.1 ÷ 1000 ms was consequently applied between the sample and the probe in some points distanced at Δ= 6 ÷ 60 nm each from other. Before and after the action the surface relief was examined in a tapping mode. Spreading resistance imaging via current-voltage (I-V) characterizations in numerous (up to 64×64) points of the scan area was realized as well.

It was generally established that both geometrical and electrical properties of the obtained nano-objects strongly depended on the pulse shape.

In the case of monopolar (i.e. positive and negative) pulses, the actions become to influence the exposed area when the magnitude $|U|$ exceeds the value of ~ 5 V that mostly leads to nanoprotrusions. Near the threshold, the nanoprotrusion width is ~ 50 nm, and it is widened to 150 ÷ 250 nm when $|U| \geq 7$ V. If the parameters N, T, Δ are kept constant, the nanoprotrusion is monotonically rising with increasing pulse magnitude, and reaches the maximum height of about 10 nm. Drastic changes in the pattern are registered after scanning the nanoprotrusion region in the contact mode. We consider that scratching the surface by the probe most likely leads to pitting the mechanically unstable material from the substrate.

On the contrary to the previous case, nanocavities in the form of grooves can be effectively patterned using the bipolar pulse actions (Fig. 1). The nanocavity width is found to be similar to that of nanoprotrusions, and the maximum depth of 30 nm is registered. It is distinctive that the groove appears only if the positive bias exceeds the value $U_+ \sim 5$ V, that is practically coincident with the threshold for patterning the nanoprotrusions under monopolar actions. The

dependence of height deviation δH inside exposed areas ($\delta H > 0$ for nanoprotrusions, and $\delta H < 0$ for nanocavities) on the negative pulse magnitude U_- at the positive bias $U_+ = 10$ V is displayed in Fig. 2.

Spreading resistance imaging shows the nanoprotrusions to be characterized by their conductivity close to that of the untreated film area whereas the nanocavities exhibit the well-pronounced increase in conductivity at the bottom.

Based in the obtained results, we come to conclusion that the nanoprotrusions appear due to local heating stipulated by Joule's losses in the subsurface layer underneath the probe. This process leads to phase transitions in carbon accompanying by a density decrease within the exposed area. The validity of such assumption is confirmed by the experimental fact that the nanoprotrusions appear at both pulse polarities. Also, it should be noted that no anodization is observed under monopolar actions, being inspected over available exposures.

It seems reasonable to suppose that the process of nanocavity formation develops in two stages: (1) during the positive biasing electrons are injected into the film, being accompanied by their trapping within the modified surface layer; (2) during the negative biasing the charged material, due to its electrostatic attraction to the probe, effectively removes from the film. Naturally, more detailed investigations should be done for more complete understanding of this phenomenon.

References:

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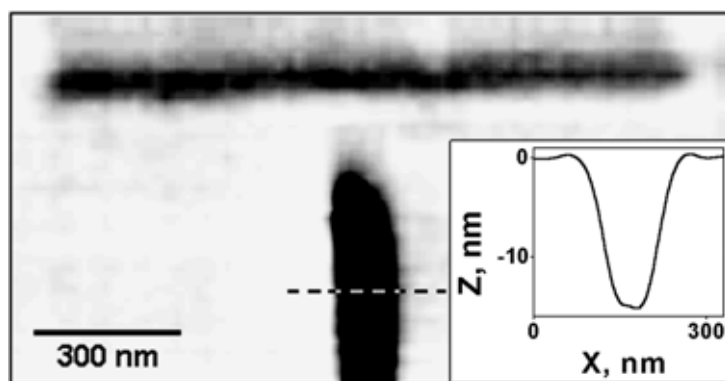[4] V.F. Dorfman and B.N. Pypkin, Surf. Coat. Technol. **48** (1991)193.

Figures:

Fig. 1. The a-C:H film topography after the bipolar pulse actions along two lines ($U_+=|U_-|=10$ V, $T_+=T_-=1$ ms, $N=1000$, $\Delta=20$ nm). The insert shows the nanocavity profile.

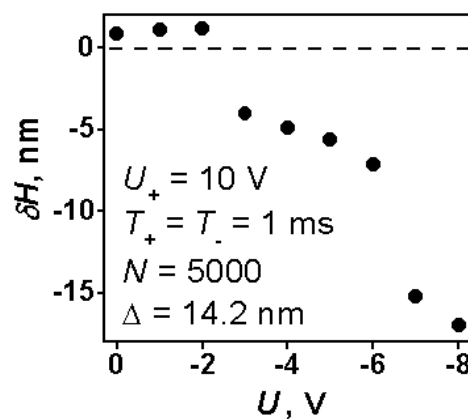Fig. 2. The δH vs. U_- plot at $U_+=$const.