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Study of corona discharge treated poly(ethylene terephthalate) films by atomic force microscopy

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Abstract. The paper studies poly(ethylene terephthalate) films modified in corona discharge. Surface topography and elastic properties of films dependence on the distance between needle and grounded substrate are obtained by atomic force microscopy.

Key words: corona discharge, poly(ethylene terephthalate) film, atomic force microscopy.

1. Introduction

Modification of polymer films in corona discharge is a well-known method of increasing their adhesive ability and imparting them electret properties [1]. Electrets characterized by surface potential, electric field intensity, effective surface charge density, relaxation time and time of life are widely used in electronics, bioengineering (implants coating), packaging, etc. Processes which take place on the surface under corona discharge treatment (CDT) identify chemical composition, topography and functional properties of the surface [2]. Study of surface characteristics is a topical issue [3-6]. It allows understanding the processes which lead to surface changes.

Work objective is to study surface characteristics of poly(ethylene terephthalate) (PET), modified by corona discharge, using atomic force microscopy (AFM).

2. Materials and methods

PET films were treated on Start-50 (OOO “Radar”) unit (figure 1) under following conditions: voltage on the needle $U = -9$ kV, lower electrode is grounded, treatment time $t = 1$ min, distance between electrodes h is 10 cm and 30 cm, room temperature and pressure, plasma-forming gas – air. Samples of sizes 75 mm x 75 mm x 110 μ m were placed on the grounded electrode 3 inner side of the drum up.

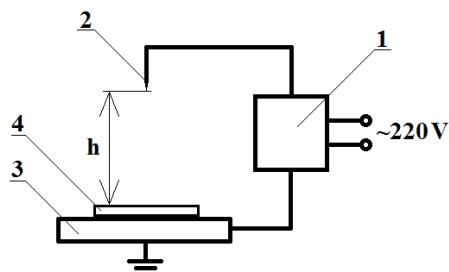


Figure 1. Schematic diagram of Start-50 corona unit. 1 – power supply unit, 2 – needle electrode, 3 – grounded electrode, 4 – sample.

For AFM method Solver P47 PRO (ZAO “NT-MDT”) microscope was used. Scanning proceeded in tapping mode. Surface topography and phase contrast of probe oscillation which is proportional to change of sample elastic properties were observed [7, 8].

3. Results and discussion

According to AFM scanning surface of the untreated film consists of macromolecular globular formations of size 50 nm x 50 nm and ~10 nm in height ($R_q = 3$ nm) (figure 2 (a), (b), (c)). Phase contrast map, obtained from 15 μ m x 15 μ m area (figure 2 (d)) allows to assume the presence of not less than 2 phases with different elastic properties. It can be proved by the shape of phase contrast distribution curve, which has an asymmetry towards positive values while maximum is at -0.3 nA (figure 2 (e)).

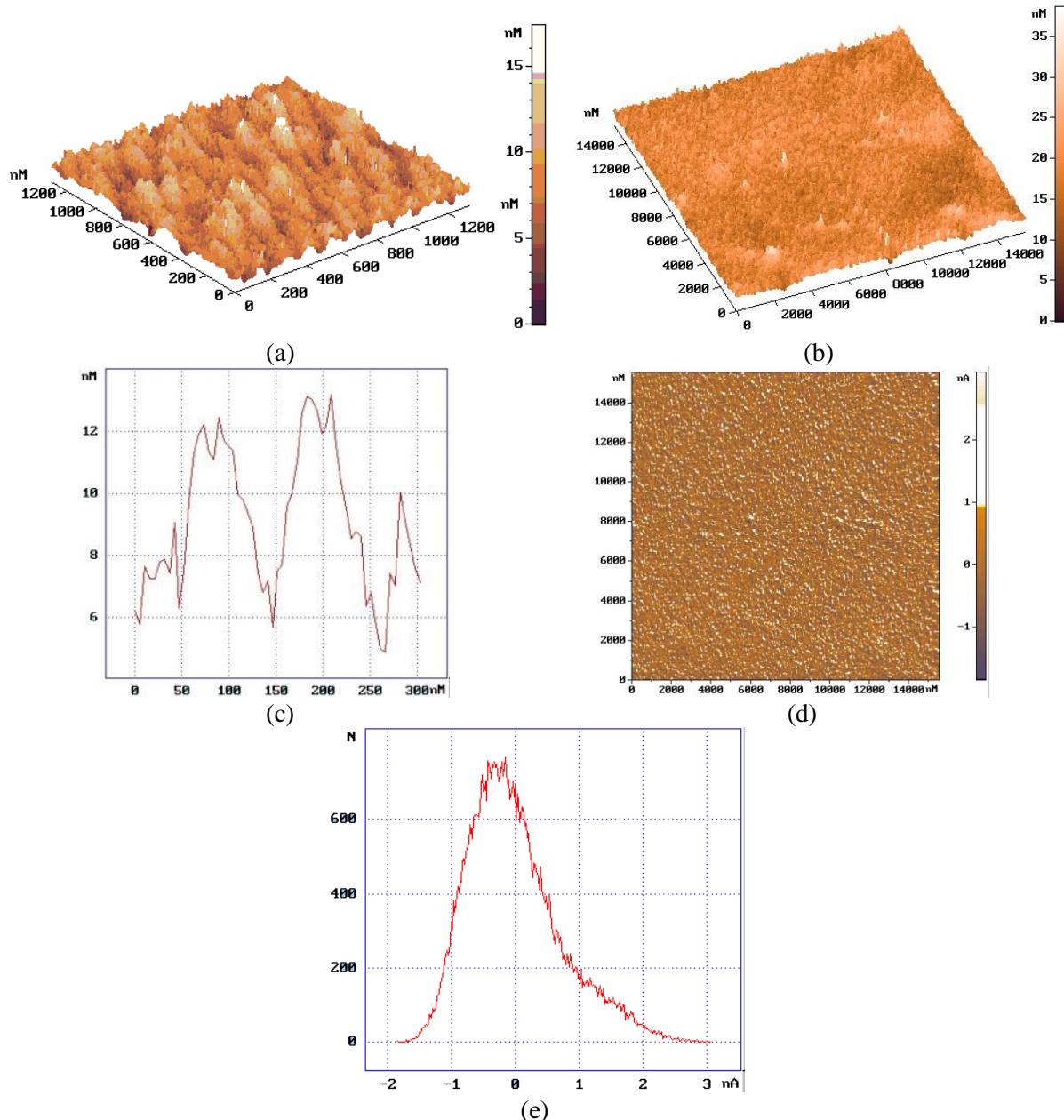


Figure 2. AFM-image of untreated sample: (a), (b) – surface topography, (c) – cross-section, (d) – phase contrast map, (e) – phase contrast distribution curve.

Study of the sample treated at $h = 10$ cm showed that some formations of size 250 nm x 250 nm on the surface reach 100 nm in height (figure 3 (c)). Size of other macromolecular formations increased up to 100 nm x 100 nm while roughness $R_q = 13$ nm. (figure 3 (a), (b)). Phase contrast map shows irregular distribution of phases on the surface (figure 3 (d)). Unlike the untreated film, phase contrast distribution curve of treated PET is asymmetrical towards negative values while maximum is at 0.5 nA (figure 3 (e)).

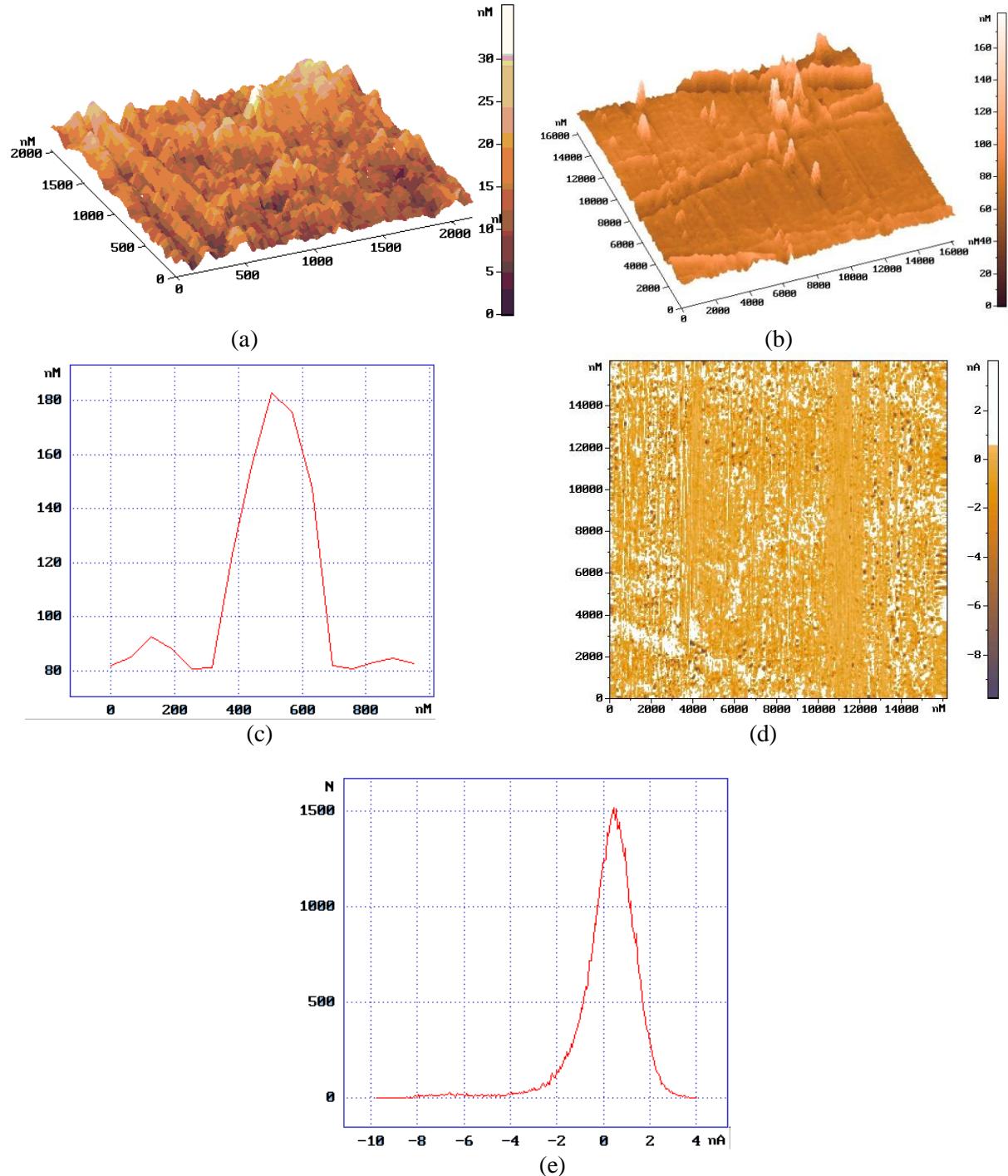


Figure 3. AFM-image of sample treated at $h = 10$ cm: (a), (b) – surface topography, (c) – cross-section, (d) – phase contrast map, (e) – phase contrast distribution curve.

AFM study of the sample modified at $h = 30$ cm allows to conclude that there is a big amount of macromolecular formations of size $500 \text{ nm} \times 500 \text{ nm}$ and $\sim 120 \text{ nm}$ in height. The rest of the surface is covered with smaller globulars of size $150 \text{ nm} \times 150 \text{ nm}$ and $\sim 25 \text{ nm}$ in height ($R_q = 17 \text{ nm}$) (figure 3 (a), (b), (c)). Analysis of phase contrast map and distribution curve leads to conclusion that surface structures of this sample differ stronger. Difference is characterized by presence of 2 peaks on the distribution curve, which values changed in comparison with previous samples (figure 4 (d), (e)).

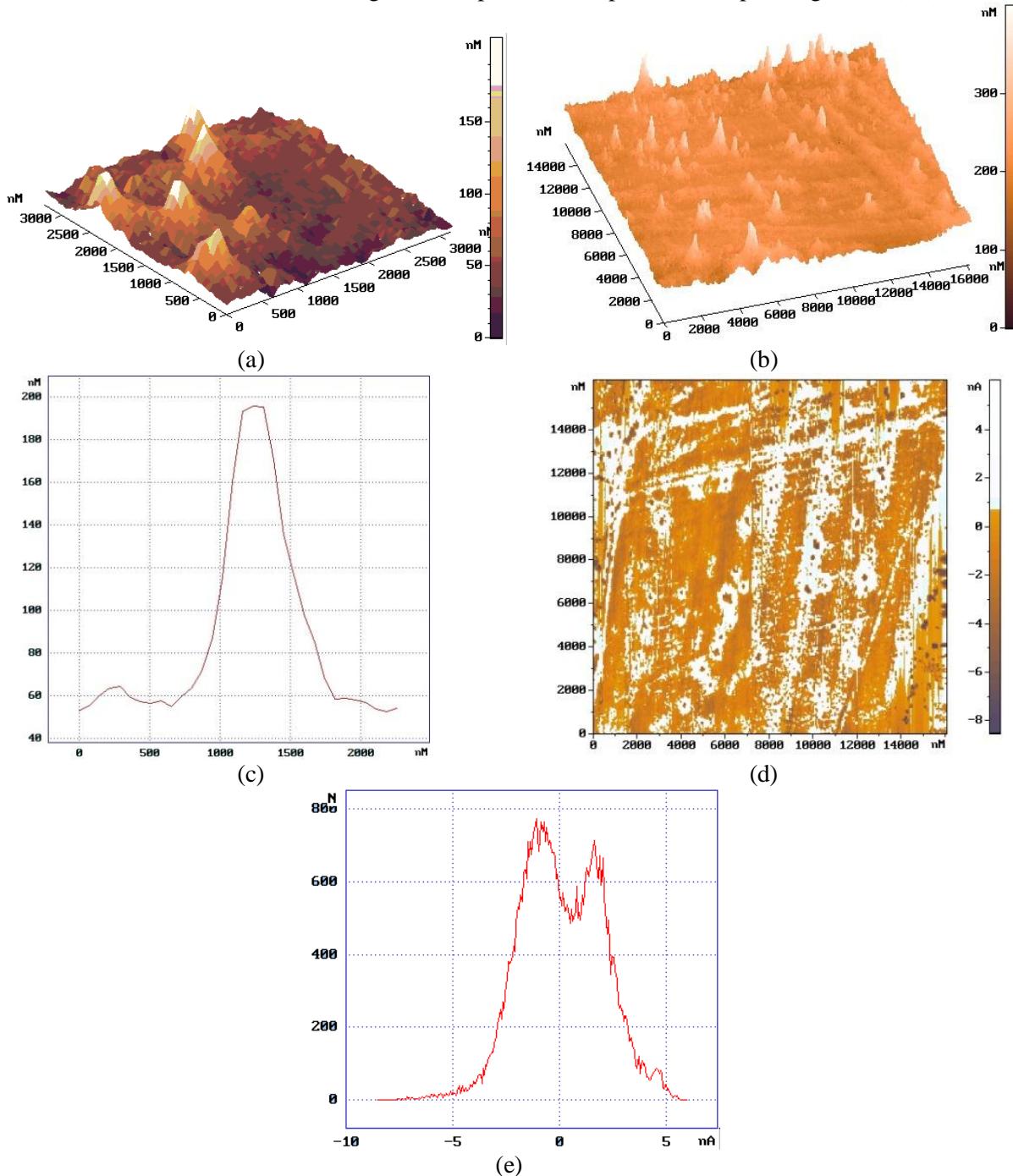


Figure 4. AFM-image of sample treated at $h = 30$ cm: (a), (b) – surface topography, (c) – cross-section, (d) – phase contrast map, (e) – phase contrast distribution curve.

4. Conclusion

Surface of untreated PET film contains a small amount of regularly distributed structures with elastic properties which are different from the main film. Corona discharge treatment of PET film surface leads to changes in surface topography, size of macromolecular formations and phase contrast distribution curve maximum, which witnesses the appearance of structures with elastic properties different from properties of untreated film. Changes in properties of film increases with distance between needle and film surface during CDT.

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