# **Investigation of Modification of the Internal Surface of Polysulfone Hollow Fiber Channels under Their Irradiation by a 10-keV Electron Beam**

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**Abstract**—The results of spectral analysis of the surface of polysulfone fiber (channel) after transmission of a 10-keV electron beam through it are presented in this work. This work is a continuation of the study of the dependence of the quantity of electrons transmitted without loss of energy through the array formed of polysulfone hollow fibers, on the angle of rotation of the array about the vertical axis. Data of the analysis demonstrates significant modification of the internal surface of the channel after its electron irradiation. It is made clear that, in the case of the long-term irradiation of polysulfone tubes by charged-particle beams, a dark coating is formed on the surface layer of the channel which can have a significant impact on the transmission capacity and controllability of the channels in the context of the control of charged particles. Comparison of the elemental composition of the surface of the fiber before and after the grazing interaction of a beam of electrons with an energy of 10 keV with it reveals an increase in the carbon concentration in the irradiated fiber of 50 wt %, and an oxygen content increase of 40 wt %. The sulphur amount does not change, and remains at the level of about 10 wt % both in the irradiated and in nonirradiated sample.

**Keywords:** surface modification, polysulfone fiber, dielectric channel, spectral analysis, electron beam, characteristic radiation, electron microscopy, energy-dispersive analysis, carbon-containing layer, concentration of substance

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## INTRODUCTION

The possibility of controlling charged-particle beams by dielectric channels was for the first time demonstrated in [1] and received the name "guiding". To this date, in this field of research, the question of the properties of dielectric surfaces which are used for controlling charged-particle beams [2–4] has not been resolved, and also the most effective control structures and configurations are being sought for [5–19]. However, works are lacking, in which the occurrence of modification of the surface of used channels as a result of irradiation of the dielectric channel by charged particles was systematically investigated. The question concerning modification of the surface of dielectrics after implementation of the guiding is important because any alteration of the surface properties of the channels can have an impact on their controlability.

The investigation carried out in this work is a continuation of earlier work [20], in which the transmission of fast electrons (with an energy of 10 keV) through an array formed from 36 polysulfone hollow fibers with an internal diameter of 200–220 μm of each channel, with a wall thickness of 60 μm, and 7 mm long, is studied. To prevent charging of the channel inputs, the front face of the array is coated by a thin (less than 100 nm) silver layer. The controlling properties of this array in relation to a beam of fast electrons are demonstrated. The present work is concerned with investigation of the internal surface of polysulfone fibers of the array after performing the indicated experiment.

## EXPERIMENTAL

Figure 1 presents schematics of the array formed from polysulfone fibers and the result of investigation of the guiding of electrons with an energy of 10 keV [20]. In this work, the experimental results of investigating the transmission of electrons with an energy of 10 keV through the dielectric channels which are poly(a)  $\frac{1}{2}$  10  $\frac{1}{2}$  (b) (c)

0.4

0.8 0.6 0.4 0.2 0 1 2 3 4 5 6 Proportion of transmitted electrons Angle of incidence, deg 0.3 0.1  $\begin{array}{l}\n\leq\n\leq 0.2\n\end{array}$ 0 50 100 150 200 250 300 350 Time, min 3 mm

**Fig. 1.** Results of examination of the guiding for 10-keV electrons, obtained in [8]: (a) schematics of an array formed of polysulfone fibers (on the left) and view to scale of the end face (on the right); (b) comparison of the proportion of electrons of the incident beam, transmitted through the dielectric (experimental data, circles) and metallic channel (calculation, squares) without loss of energy; (c) time dependence of the quantity of electrons transmitted through an array of channels, without loss of energy *I*.

sulfone hollow fibers collected into a single array were presented. The array of channels was tilted relative to the axis of the incident beam, and the current of the transmitted beam was measured at the output, and the proportion of electrons which lost less than 10% of their initial energy was also estimated. These criteria are necessary for ascertaining the capabilities of using channels of the investigated dielectric material as beam-optics components. It should be noted that the quantity of the electrons transmitted through the channel (*I*) without a loss of energy had the character-

1.0



**Fig. 2.** Image of the coating formed on the inner wall of the polysulfone fiber after irradiation. Regions of measurement of the composition, located at a depth of 2 (*1*); 4 (*2*); 5 (*3*); 6 (*4*) μm, are dotted.

istic time dependence demonstrated in Fig. 1c. It is evident from Fig. 1c that the propagation of electrons started "instantaneously" (counting started at the moment of beam switch on) and decreased with time until complete "closure" of the channel.

In this work, the results of spectral analysis of the surface of a polysulfone fiber taken individually from an array composed of 30 hollow fibers with an internal diameter of the channel of  $160 \pm 60$  µm, after its irradiation by an electron beam with an energy of 10 keV, are presented. The investigations were carried out by dark-field scanning transmission electron microscopy, energy-dispersive analysis, transmission electron microscopy and diffraction analysis by the microscopes Tecnai Osiris and Tecnai G<sup>2</sup>30ST at an accelerating voltage of 15 kV. To investigate the samples of polysulfone fibers, it was necessary to obtain a thin cut which was performed by an ultramicrotome (Leica EM UC7). A cut across the surface of the fiber was examined by an FEI Scios focused-ion-beam scanning electron microscope.

The analysis was carried out on a cross section of the polysulfone tube (Fig. 2). The results demonstrated the presence of a coating which has a large number of pores on the internal surface of the channel, and also a spot of the coating the thickness of which is 400 nm was found closer to the inner surface of the fiber. Analysis of the composition of the coating was carried out on various depths from 400 nm to 6 μm (Fig. 3) from the surface of the channel.

## RESULTS AND DISCUSSION

The data of the relationship between the thickness of the layer of the polysulfone sample and concentration of the substance in the given layer (for the nonir-



**Fig. 3.** Plot of the dependence of the oxygen concentration (circles), carbon (squares), sulfur (triangles), nitrogen (asterisks) in the coating layer on the depth of location of the region of measurement of the composition in the irradiated sample.

radiated fiber) are collected in Table 1. The predominance of oxygen (56 wt %) and carbon (30 wt %) over the examined thickness of the coating in comparison to sulfur and nitrogen (less than 10 wt %) was discovered in the layer composition. Table 2 collects the data of the relationship between the thickness of the layer of the polysulfone sample and concentration of the substance in the layer for the irradiated fiber. Figure 4 presents the dependence of concentration of the sub-



**Fig. 4.** Cross-sectional image of the polysulfone fiber. Regions of measurement of the composition located at depths of 60 (*1*); 5 (*2*); 3 (*3*); 0.5 (*4*) μm are dotted.

stance in the layer on the layer depth in the fiber (for the irradiated sample).

While collating the results of analysis of the composition of the irradiated fiber with the nonirradiated one (Fig. 5), the difference between the carbon concentrations in the surface layer (thickness is about 400 nm) and in the deeper layer (60 μm in thickness) was found (Fig. 6). The analysis was carried out at an acceleration voltage of 20 kV.

We note that the carbon content has decreased by 50% and the oxygen content has increased by 40% in every layer of the fiber after irradiation. The sulfur

**Table 1.** Data of the relationship between the thickness of the layer and concentration of the substance in this layer (for the nonirradiated fiber)

Layer thickness, µm	Carbon concentration, %	Oxygen concentration, %	Sulfur concentration, %
0.5	74.3	10.7	12.3
3.0	83.2	10.8	5.0
5.0	85.1	9.5	4.0
60.0	83.8	11.7	4.4

**Table 2.** Data of the relationship between the thickness of the layer and concentration of substance in this layer (for the irradiated fiber)





**Fig. 5.** Plot of the concentration of oxygen (circles), carbon (squares), sulfur (triangles) in the layer of the nonirradiated fiber as a function of depth of location of the region of measurement of the composition.



**Fig. 6.** Cross-sectional images of the channel of a polysulfone fiber.

amount does not change and is equal to about 10 wt % both for the irradiated and for the nonirradiated samples.

It should be noted that the transmission of electrons which started "instantaneously" after irradiation by an electron beam, decreased with time until complete "closure" of the channel. Perhaps, "closure" of the channel demonstrated in the experiment [20] and presented in Fig. 1c was affected by the formation of the coating on the internal surface of the polysulfone fiber.

### **CONCLUSIONS**

In this work, the results of spectral analysis of the surface of the individual polysulfone fiber after its irradiation by an electron beam with an energy of 10 keV, are presented. The comparative analysis of the substance composition before irradiation and after it at a depth from 400 nm to 6 μm was carried out.

It was made clear that, in the case of the long-term irradiation of polysulfone tubes by charged-particle beams, modification of the surface layer (formation of coating) takes place that can have a significant impact on the transmission capacity and controllability of the channels in the field of control of charged particles. Because the effect of guiding is present only for a while (about 2 h) [20] for each angle of incidence of the fiber as related to the axis of the incident beam, it can be assumed that some of the electrons are facilitate not only the formation of the charge on the internal surface of the channels, but also the occurrence of more complex processes in the polymer substance.

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#### CONFLICT OF INTEREST

We declare that we have no conflicts of interest

### REFERENCES

- 1. N. Stolterfoht, J. H. Bremer, V. Hoffmann, D. Fink, R. Hellhammer, A. Petrov, and B. Sulik, Phys. Rev. Lett. **88**, 133201 (2002). https://www.doi.org/10.1103/PhysRevLett.88.133201
- 2. T. Ikeda, M. Ikekame, Y. Hikima, M. Mori,
- S. Kawamura, T. Minowa, and W.-G. Jin, Nucl. Instrum. Methods Phys. Res., Sect. B **470**, 42 (2020). https://www.doi.org/10.1088/1742-6596/1412/24/242007
- 3. A. Milosavljevic, G. Vikor, Z. Pesic, P. Kolarz, D. Sevic, B. Marinkovic, S. Matefi-Tempfli, M. Matefi-Tempfli, and L. Piraux, Phys. Rev. A **75**, 030901 (2007).

https://www.doi.org/10.1103/PhysRevA.75.03090

- 4. W. Wang, D. Qi, D. Yu, M. Zhang, F. Ruan, J. Chen, and X. Cai, J. Phys.: Conf. Ser. **163**, 012093 (2009). https://www.doi.org/10.1088/1742-6596/163/1/012093
- 5. G. A. B. Gál, I. Rajta, S. Z. Szilasi, Z. Juhász, S. Biri, C. Cserháti, A. Csik, and B. Sulik, Nucl. Instrum. Methods Phys. Res., Sect. B **269**, 2322 (2011). https://www.doi.org/10.1016/j.nimb.2011.02.024
- 6. E. Gruber, G. Kowarik, F. Ladinig, J. P. Waclawek, D. Schrempf, and F. Aumayr, Phys. Rev. A **86**, 062901 (2012). https://www.doi.org/10.1103/PhysRevA.86.062901
- 7. S. J. Wickramarachchi, B. S. Dassanayake,
- D. Keerthisinghe, T. Ikeda, and J. A. Tanis, Phys. Scr. **156**, 014057 (2013). https://www.doi.org/10.1103/PhysRevA.94.022701
- 8. Ch. Lemell, J. Burgdorfer, and F. Aumayr, Prog. Surf. Sci. **88**, 237 (2013).
	- https://www.doi.org/10.1016/j.progsurf.2013.06.001
- 9. B. S. Dassanayake and R. J. Bereczky, Phys. Rev. A **83**, 012707 (2011). https://www.doi.org/10.1103/PhysRevA.83.012707
- 10. B. S. Dassanayake, Phys. Rev. A **81**, 020701 (2010). https://www.doi.org/10.1103/PhysRevA.81.020701
- 11. K. A. Vokhmyanina, A. S. Kubankin, I. A. Kishin, R. M. Nazhmudinov, Yu. S. Kubankin, A. V. Sotnikov, V. S. Sotnikova, and D. A. Kolesnikov, J. Nano- Electron Phys. **10**, 06036 (2018). https://www.doi.org/10.21272/jnep.10(6).06036
- 12. S. Das, B. S. Dassanayake, M. Winkworth, J. L. Baran, N. Stolterfoht, and J. A. Tanis, Phys. Rev. A. **76**, 042716 (2007). https://www.doi.org/10.1103/PhysRevA.76.042716
- 13. V. P. Petukhov and M. V. Petukhov, J. Surf. Invest.: X-ray, Synchrotron Neutron Tech. **11**, 1056 (2017).
- https://www.doi.org/10.1134/S1027451017050330 14. H.-D. Nguyen, J.-P. Wulfkuhler, J. Heisig, and M. Tajmar, Sci. Rep. **11**, 8345 (2021).

https://www.doi.org/10.1038/s41598-021-87156-4

15. K. A. Vokhmyanina, G. P. Pokhil, P. N. Zhukova, E. Irribarra, A. S. Kubankin, V. S. Levina, R. M. Nazhmudinov, A. N. Oleinik, and I. A. Kishin, Nucl. Instrum. Methods Phys. Res., Sect. B **355**, 307 (2015).

https://www.doi.org/10.1016/j.nimb.2015.02.068

- 16. K. A. Vokhmyanina, P. N. Zhukova, A. S. Kubankin, I. A. Kishchin, A. S. Klyuev, R. M. Nazhmudinov, A. N. Oleinik, and G. P. Pokhil, J. Surf. Invest.: X-ray, Synchrotron Neutron Tech. **9**, 286 (2015). https://www.doi.org/10.1134/S1027451015020196
- 17. K. A. Vokhmyanina, A. S. Kubankin, L. V. Myshelovka, H. Zhang, A. A. Kaplii, V. S. Sotnikova, and M. A. Zhukova, J. Instrum. **15**, C04003 (2020). https://www.doi.org/10.1088/1748-0221/15/04/C04003
- 18. Z. Juhász, B. Sulik, S. Biri, K. Tıkési, R. Bereczky J., R. Rácz, Á. Kövér, J. Pálinkás, and N. Stolterfoht, J. Phys.: Conf. Ser. **388**, 132007 (2012). https://www.doi.org/10.1088/1742-6596/388/13/132007
- 19. K. A. Vokhmyanina, A. S. Kubankin, I. A. Kishin, R. M. Nazhmudinov, Yu. S. Kubankin, A. V. Sotnikov, V. S. Sotnikova, and D. A. Kolesnikov, J. Nano-Electron. Phys. **10**, 06036 (2018). https://www.doi.org/10.21272/jnep.10(6).06036
- 20. K. A. Vokhmyanina, L. V. Myshelovka, D. A. Kolesnikov, V. S. Sotnikovaa, A. A. Kaplii, A. S. Kubankin, P. N. Zhukova, and V. Yu. Ionidi, Tech. Phys. Lett. **47**, 31 (2021).

https://www.doi.org/10.1134/S1063785021080289

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